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Supporting Information

Experimental Section

All glasswares were flame-dried under a stream of argon and experiments were run under an argon atmosphere. ^1H NMR and ^{13}C NMR spectra were recorded at 300 MHz and 75 MHz respectively. Chemical shifts are reported in ppm relative to internal tetramethylsilane. Uncorrected melting points were recorded on a Kofler apparatus. Flash column chromatography were performed on Merck Kieselgel 60 (230-400 mesh). THF and ether were distilled from sodium benzophenone ketyl. Dichloromethane, 1,2-dichloroethane (DCE), toluene and DMF were distilled from CaH_2. Other reagents were obtained from commercial sources and used as received.

Preparation of compound 1

1,1-Dibromo-3-phenylbut-1-ene (13). To a solution of CBr_4 (94.9 g, 286.0 mmol) in dry CH_2Cl_2 (400 mL) at 0 °C, was added PPh_3 (150 g, 572 mmol). After 15 min, 2-phenylpropionaldehyde (19.2 g, 143.1 mmol) was added slowly. After 1 h at rt, the reaction mixture was diluted with pentane (1 L), the precipitated solids were filtered off through Celite and thoroughly washed with pentane. The filtrate was concentrated under reduced pressure and the crude product was taken up in pentane. The solids were filtered off again and the filtrate concentrated under reduced pressure. The crude material was purified by flash chromatography (petroleum ether) to give 35 g (85%) of **13** as a colorless oil; IR 3010, 1600, 1490, 810, 760, 700 cm^{-1}; ^1H NMR δ 7.37-7.24 (m, 5H), 6.53 (d, J = 9.6 Hz, 1H), 3.78 (dq, J = 9.6 and 7.0 Hz, 1H), 1.41 (d, J = 7.0 Hz, 3H); ^{13}C NMR δ 142.9 (s), 142.7 (d), 128.6 (d), 126.8 (d), 126.7 (d), 88.4 (s), 43.3 (d), 20.0 (q); EI MS m/z (relative intensity) 292 (M^{+}+4, 7), 290 (M^{+}+2, 13), 288 (M^{+}, 7), 211 (28), 209 (28), 195 (28), 193 (27), 130 (62), 129 (100), 128 (26), 115 (47), 105 (14), 77 (13), 51 (10).

4-Phenylpent-2-yn-1-ol (14). To a solution of **13** (18.1 g, 62.4 mmol) in dry THF (150 mL) at -100 °C, was added n-BuLi (50.1 mL, 2.5M in hexanes, 125.2 mmol) in 2 h. After the addition was complete, dry paraformaldehyde (3.75 g, 124.89 mmol of monomeric HCHO) was added in one portion and the cooling bath was removed immediately. The reaction mixture was allowed to warm to rt (a slighlty exothermic reaction started causing an increase of temperature to 30 °C). After stirring overnight at rt, the reaction mixture was poured into a saturated aqueous NH_4Cl solution and was extracted with AcOEt. The combined extracts were washed with brine, dried over $MgSO_4$, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/AcOEt: 90/10). 9.5 g (95%) of **14** was obtained as a pale yellow liquid; IR 3340 (br), 2240, 1600, 1490, 1450, 1140, 1000, 760, 700 cm^{-1}; ^{1}H NMR δ 7.40-7.20 (m, 5H), 4.33 (d, J = 2.2 Hz, 2H), 3.81 (qt, J = 7.3 and 2.2 Hz, 1H), 1.68 (s, 1H, OH), 1.51 (d, J = 7.3 Hz, 3H); ^{13}C NMR δ 142.8 (s), 128.4 (d), 126.7 (d), 126.6 (d), 88.8 (s), 80.1 (s), 51.2 (t), 31.7 (d), 24.0 (q); EI MS *m/z* (relative intensity) 160 (M^+, 65), 145 (22), 142 (79), 141 (28), 129 (58), 128 (62), 127 (43), 117 (61), 116 (25), 115 (100), 91 (40), 77 (23), 51 (23). Anal. Calcd for $C_{11}H_{12}O$: C, 82.46; H, 7.55. Found: C, 82.39; H, 7.61.

(*Z*)-4-Phenylpent-2-en-1-ol (1). A solution of CuBr (11.0 g, 76.7 mmol) and LiBr (12 g, 138 mmol) in dry THF (20 mL), was added slowly to a refluxing activated zinc suspension [prepared from zinc dust (39.5 g, 604.1 mmol), 1,2-dibromoethane (4.85 mL, 56.28 mmol) in isopropanol (90 mL); reflux 10 min]. After 20 min, a solution of **14** (8.2 g, 51.2 mmol) in isopropanol (25 mL) was added slowly. After 2 h reflux, the reaction mixture was cooled to rt, diluted with ether (300 mL) and filtered through Celite. The filtrate was treated with a saturated aqueous NH_4Cl solution, and was extracted with AcOEt. The combined extracts were dried over $MgSO_4$ and concentrated under reduced pressure to give an oil, which was purified by flash chromatography (cyclohexane/AcOEt gradient: 80/20 to 70/30). 6.6 g (79%) of **1** was obtained as a clear yellow liquid; *Rf*: 0.34 (C_6H_6/AcOEt: 90/10); IR 3440 (br), 1665, 1600, 1490, 1450, 1010, 700 cm^{-1}; ^{1}H NMR δ 7.35-7.14 (m, 5H), 5.70-5.53 (m, 2H), 4.26 (ddd, J = 12.6, 6.5 and 1.2 Hz, 1H), 4.17 (ddd, J = 12.6, 6.1, 0.8 Hz, 1H), 3.76 (dq, J = 9.1 and 7.0 Hz, 1H), 1.81 (s, 1H, OH), 1.34 (d, J = 7.0 Hz, 3H); ^{13}C NMR δ 145.7 (s), 137.6 (d), 128.4 (d), 127.2 (d), 126.7 (d), 126.0 (d), 58.6 (t), 37.4 (d), 22.2 (q); EI MS *m/z* (relative intensity) 144 (M^+-H_2O, 76), 129 (100), 115 (23), 105 (26), 91 (44). Anal. Calcd for $C_{11}H_{14}O$: C, 81.44; H, 8.70. Found: C, 81.33; H, 8.78.

Et_2Zn/ICH_2Cl promoted cyclopropanation of 1.

To a solution of Et_2Zn (62 mL, 1M in hexanes, 62 mmol, 2 equiv.) in dry DCE (500 mL) at -23 °C, was added dropwise ICH_2Cl (9 mL, 124 mmol, 4 equiv.). After 20 min at -23 °C, a solution of **1** (5 g, 31 mmol) in dry DCE (60 mL) was added dropwise. After 1 h at -23 °C, the reaction mixture was poured into a saturated aqueous NH_4Cl solution (150 mL) and extracted with ether. The combined extracts were dried over $MgSO_4$ and concentrated under reduced pressure. After purification by flash chromatography (cyclohexane/AcOEt gradient: 95/5 to 90/10), 0.98 g (18%) of **2b** was obtained as a colorless oil and 3.47 g (64%) of **2a** as a crystalline white solid.

$Sm(Hg)/ICH_2Cl$ promoted cyclopropanation of 1.

Dry $HgCl_2$ (0.67 g, 2.47 mmol, 0.80 equiv.), dry THF (80 mL) and **1** (0.50 g, 3.08 mmol) were added to samarium powder (2.27 g, 15.10 mmol, 5.00 equiv.) . To the vigorously stirred mixture at -50 °C, was added dropwise ICH_2Cl (1.10 mL, 15.10 mmol, 5.00 equiv.). The reaction mixture was then allowed to warm to rt in 1 h. After 1 h stirring at rt, the reaction mixture was poured into a cold saturated aqueous K_2CO_3 solution (50 mL) overlaid with ether (50 mL). After 10 min stirring, the resulting mixture was filtered through Celite and the precipitate thoroughly washed with ether. The aqueous layer was extracted with ether and the combined extracts were washed with brine, dried over Na_2SO_4, concentrated under reduced pressure to give a residue which was purified through a short column of silica gel (cyclohexane/AcOEt: 95/5) to give 0.403 g (74%) of a diasteroisomeric mixture of **2a** and **2b**, in a 30/70 ratio.

(1*S,2*S**)-2-(1(*R**)-1-phenylethyl)cyclopropanemethanol (2a).** Crystallization from *n*-pentane, white solid, mp 63-64 °C; *Rf*: 0.30 (C_6H_6/AcOEt: 90/10); IR 3300 (br), 3050, 3020, 1490, 1450, 1015, 745, 700 cm^{-1}; ^1H NMR δ 7.34-7.18 (m, 5H), 3.77 (m, 2H), 2.30 (dq, J = 10.5 and 6.9 Hz 1H), 1.64 (s, 1H, OH), 1.39 (d, J = 6.9 Hz, 3H), 1.36-1.24 (m, 1H), 1.21-1.12 (m, 1H), 0.70 (m, 1H), 0.07 (m, 1H); ^{13}C NMR δ 147.3 (s), 128.3 (d), 126.8 (d), 125.9 (d), 62.8 (t), 39.3 (d), 24.2 (d), 22.8 (q), 19.3 (d), 9.2 (t); EI MS *m/z* (relative intensity) 158 (M^+-H_2O, 2), 143 (16), 132 (33), 118 (62), 117 (100), 105 (73), 91 (46), 77 (14). Anal. Calcd for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.68; H, 9.18.

(1*R,2*R**)-2-(1(*R**)-1-phenylethyl)cyclopropanemethanol (2b).** *Rf*: 0.38 (C_6H_6/AcOEt: 90/10); IR 3350 (br), 3050, 3020, 1450, 1030, 700 cm^{-1}; ^1H NMR δ 7.34-7.17 (5H, m), 3.66 (dd, J = 11.8 and 5.7 Hz, 1H), 3.33 (dd, J = 11.8 and 8.5 Hz, 1H), 2.29 (dq, J = 10.1 and 6.9 Hz, 1H), 1.36 (d, J = 6.9 Hz, 3H), 1.13-1.26 (m, 2H), 0.85 (m, 1H), 0.82 (s, 1H, OH), 0.12 (m, 1H); ^{13}C NMR δ 147.4 (s), 128.7 (d), 126.4 (d), 126.3 (d), 63.1 (t), 39.9 (d), 23.8 (q), 23.3 (d), 19.0 (d), 9.5 (t); EI MS *m/z* (relative intensity) 158 (M^+-H_2O, 30), 143 (40), 129 (25), 128 (20), 118 (71), 117 (100), 115 (36), 105 (65), 106 (30), 105 (65), 103 (21), 91 (56), 79 (15), 78 (16), 77 (28), 51 (13). Anal. Calcd for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.64; H, 9.22.

(*Z*)-4-Phenyl-1-*tert*-butyldimethylsilyloxypent-2-ene (3). To a solution of **1** (0.30 g, 1.85 mmol) in dry DMF (1 mL) were added imidazole (0.28 g, 4.11 mmol) and TBDMSCl (0.31 g, 2.05 mmol). After 15 min at rt, the reaction was quenched with an aqueous NH_4Cl solution. The reaction mixture was extracted with petroleum ether/CH_2Cl_2: 90/10, and the combined extracts were dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash chromatography (petroleum ether/AcOEt: 100/0 to 95/5) to give 0.49 g (96%) of **3** as a colorless oil; IR 3020, 1655, 1600, 1250, 1080, 830, 775, 720 cm^{-1}; ^1H NMR δ 7.35-7.17 (m, 5H), 5.61-5.48 (m, 2H), 4.23 (dd, J = 12.9 and 4.0 Hz, 1H), 4.35 (dd, J = 12.9 and 3.3 Hz, 1H), 3.76-3.70 (m, 1H), 1.31 (d, J = 7.0 Hz, 3H), 0.09 (s, 9H), 0.06 (s, 6H); ^{13}C NMR δ 145.9 (s), 135.6 (d), 128.6 (d), 128.5 (d), 126.8 (d), 126.6 (d), 59.5 (t), 37.6 (d), 26.0 (q), 22.2 (q), 18.35 (s), -5.0 (q); EI MS *m/z* (relative intensity) 219 (M^+-C_4H_9, 100), 143 (47), 129 (31), 115 (20), 105 (71), 75 (75), 73 (19).

Cyclopropanation of 3. Compound **3** (0.20 g, 0.72 mmol) was cyclopropanated by using Et_2Zn (2.90 mL, 1M in hexanes, 2.90 mmol) and ICH_2Cl (0.42 mL, 5.77 mmol) in DCE (10 mL) at -23 °C for 2 h. The crude product was dissolved in dry THF (2 mL) and treated with *n*-Bu_4NF (1.10 mL, 1M in THF, 1.10 mmol). After 3 h between 0 °C and rt, the reaction mixture was quenched with a saturated aqueous NH_4Cl solution and extracted with ether. The combined extracts were washed with brine, dried over $MgSO_4$, concentrated under reduced pressure and the crude product was purified by filtration through silica gel (petroleum ether/AcOEt: 70/30) to give 0.058 g (45%) of a diastereoisomeric mixture of **2a** and **2b** in a 80/20 ratio.

Et_2Zn/ICH_2Cl promoted cyclopropanation of 4. Compound **4** (0.20 g, 1.23 mmol) was cyclopropanated by using Et_2Zn (4.90 mL, 1M in hexanes, 4.90 mmol), ICH_2Cl (0.72 mL, 9.84 mmol) in DCE (12 mL). The crude material was purified by filtration through silica gel (cyclohexane/AcOEt: 70/30) to give 0.204 g (94%) of a diastereoisomeric mixture of **7a** and **7b** in a 75/25 ratio.

Sm(Hg)/ICH_2Cl promoted cyclopropanation of 4. Compound **4** (0.20 g, 1.23 mmol) was cyclopropanated by using Sm (0.93 g, 6.18 mmol), ICH_2Cl (0.45 mL, 6.18 mmol), $HgCl_2$ (0.27 g, 0.99 mmol) in THF (16 mL). The crude material was purified by filtration through silica gel (cyclohexane/AcOEt: 85/15) to give 0.205 g (95%) of a diastereoisomeric mixture of **7a** and **7b** in a 50/50 ratio.

Preparation of authentic samples of 7a and 7b

(1*S,2*S**)-2-(1(*R**)-1-phenylethyl)cyclopropanecarbaldehyde (15a).** To a stirred suspension of **2a** (0.15 g, 0.85 mmol), 4Å molecular sieves (0.70 g) in dry CH_2Cl_2 (10 mL) was added PCC (0.37 g, 1.70 mmol). After 2 h at rt, the reaction mixture was filtered through silica gel (CH_2Cl_2) to give 0.136 g (92%) of **15a** as a colorless oil; IR 3020, 1710, 1600, 1490, 980, 950, 750, 700 cm^{-1}; ^1H NMR δ 9.59 (d, J = 4.8 Hz, 1H), 7.35-7.19 (m, 5H), 2.72-2.62 (m, 1H), 2.10-2.00 (m, 1H), 1.74-1.62 (m, 1H), 1.35-1.31 (m, 1H), 1.27 (d, J = 6.6 Hz, 3H), 1.20-1.13 (m, 1H); ^{13}C NMR δ 201.0 (d), 145.6 (s), 128.4 (d), 126.7 (d), 126.3 (d), 37.9 (d), 32.8 (d), 28.3 (d), 22.0 (q), 14.9 (t); EI MS *m/z* (relative intensity) 174 (M^+, 2), 156 (23), 145 (66), 143 (30), 130 (25), 129 (20), 128 (21), 118 (100), 117 (82), 115 (34), 106 (34), 105 (48), 103 (21), 91 (64), 77 (24), 69 (33). Anal. Calcd for $C_{12}H_{14}O$: C, 82.72; H, 8.10. Found: C, 82.72; H, 8.10.

(1*R,2*R**)-2-(1(*R**)-1-phenylethyl)cyclopropanecarbaldehyde (15b).** Compound **2b** (0.50 g, 2.84 mmol) was oxidized with PCC (1.22 g, 5.68 mmol), 4Å molecular sieves (2.5 g) in dry CH_2Cl_2 (30 mL) at rt for 4 h. The reaction mixture was filtered through silica gel (CH_2Cl_2) to give 0.44 g (89%) of **15b** as a colorless oil; IR 3020, 1700, 1600, 1490, 1170, 980, 950, 760, 700 cm^{-1}; ^1H NMR δ 9.37 (d, J = 4.5 Hz, 1H), 7.28-7.12 (m, 5H), 2.72-2.63 (m, 2H), 1.93 (m, 1H), 1.74 (m, 1H), 1.40 (d, J = 7.0 Hz, 3H), 1.26-1.36 (m, 1H); ^{13}C NMR δ 201.0 (d), 146.0 (s), 128.4 (d), 126.5 (d), 126.2 (d), 38.2 (d), 32.1 (d), 28.3 (d), 22.7 (q), 14.7 (t); EI MS m/z (relative intensity) 174 (M^+, 2), 145 (54), 143 (27), 131 (20), 130 (26), 118 (100), 117 (64), 115 (29), 105 (42), 91 (52), 77 (21). Anal. Calcd for $C_{12}H_{14}O$: C, 82.72; H, 8.10. Found: C, 82.64; H, 8.13.

(1*R,2*S**)-2-(1(*R**)-1-phenylethyl)cyclopropanecarbaldehyde (16a).** To a solution of **15a** (0.30 g, 1.72 mmol) in MeOH (20 mL) was added MeONa (0.93 g, 17.21 mmol). After 6 h reflux, the reaction mixture was quenched by addition of a saturated aqueous NH_4Cl solution and extracted with ether. The combined extracts were dried over $MgSO_4$, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/AcOEt: 90/10, 85/15) to give 0.23 g (77%) of **16a** as a colorless oil; IR 3020, 1710, 1600, 1490, 1450, 860, 750, 700 cm^{-1}; ^{1}H NMR δ 9.05 (d, J = 5.5 Hz, 1H), 7.34-7.19 (m, 5H), 2.27 (m, 1H), 1.80-1.68 (m, 2H), 1.35 (d, J = 7.0 Hz, 3H), 1.26 (m, 1H), 1.00 (m, 1H); ^{13}C NMR δ 200.5 (d), 144.9 (s), 128.4 (d), 126.8 (d), 126.3 (d), 42.6 (d), 30.3 (d), 29.3 (d), 21.1 (q), 14.1 (t). Anal. Calcd for $C_{12}H_{14}O$: C, 82.72; H, 8.10. Found: C, 82.72; H, 8.09.

(1*S,2*R**)-2-(1(*R**)-1-phenylethyl)cyclopropanecarbaldehyde (16b).** Compound **15b** (0.30 g, 1.72 mmol) was epimerized with MeONa (0.93 g, 17.21 mmol) in refluxing MeOH (20 mL) for 6 h. Flash chromatography (cyclohexane/AcOEt: 90/10, 85/15) gave 0.24 g (80%) of **16b** as a colorless oil; IR 3020, 1700, 1600, 1490, 1450, 1400, 1020, 860, 750, 700 cm^{-1}; ^{1}H NMR δ 9.01 (d, J = 4.9 Hz, 1H), 7.30-7.16 (m, 5H), 2.31 (m, 1H), 1.76-1.68 (m, 2H), 1.37 (m, 1H), 1.33 (d, J = 7.0 Hz, 3H), 1.00 (m, 1H); ^{13}C NMR δ 200.5 (d), 144.9 (s), 128.4 (d), 126.8 (d), 126.2 (d), 42.3 (d), 29.4 (d), 29.3 (d), 21.2 (q), 14.5 (t); EI MS m/z (relative intensity) 174 (M^+, 0.4), 129 (12), 128 (11), 118 (44), 117 (59), 115 (21), 106 (100), 105 (29), 91 (38), 77 (15), 69 (28). Anal. Calcd for $C_{12}H_{14}O$: C, 82.72; H, 8.10. Found: C, 82.83; H, 8.16.

(1*R,2*S**)-2-(1(*R**)-1-phenylethyl)cyclopropanemethanol (7a).** $NaBH_4$ (0.06 g, 1.59 mmol) was added to a solution of **16a** (0.21 g, 1.23 mmol) in MeOH (5 mL). After 20 min at rt, the reaction mixture was quenched with a 1M aqueous hydrochloric acid solution and extracted with AcOEt. The combined extracts were evaporated and the residue taken up in CH_2Cl_2, dried over

$MgSO_4$, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/AcOEt gradient: 95/5 to 80/20) to give 0.15 g (70%) of **7a** as a colorless oil; IR 3340 (br), 3050, 3020, 1600, 1490, 1450, 1050, 750, 700 cm^{-1}; ^1H NMR δ 7.26-7.10 (m, 5H), 3.42 (d, J = 7.0 Hz, 2H), 2.05 (m, 1H), 1.57 (s, 1H, OH), 1.28 (d, J = 7.0 Hz, 3H), 0.94 (m, 1H), 0.80 (m, 1H), 0.41-0.29 (m, 2H); ^{13}C NMR δ 146.6 (s), 128.3 (d), 126.9 (d), 125.9 (d), 66.9 (t), 43.3 (d), 24.6 (d), 21.5 (q), 21.3 (d), 9.7 (t); EI MS *m/z* (relative intensity) 158 (M^+-H_2O, 5), 143 (18), 132 (47), 118 (84), 117 (100), 115 (26), 106 (60), 105 (82), 104 (23), 103 (19), 91 (41), 78 (10), 77 (15). Anal. Calcd for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.70; H, 9.20.

(1*S,2*R**)-2-(1(*R**)-1-phenylethyl)cyclopropanemethanol (7b).** Compound **16b** (0.21 g, 1.23 mmol) was reduced with $NaBH_4$ (0.06 g, 1.59 mmol) in MeOH (5 mL). Purification by flash chromatography (cyclohexane/AcOEt gradient: 95/5 to 80/20) gave 0.17 g (79%) of **7b** as a colorless oil; IR 3340 (br), 3050, 1600, 1490, 1450, 1050, 750, 700 cm^{-1}; ^1H NMR δ 7.22-7.05 (m, 5H), 3.20 (d, J = 7.0 Hz, 2H), 2.05-1.96 (m, 1H), 1.64 (s, 1H, OH), 1.22 (d, J = 7.3 Hz, 3H), 0.85-0.70 (m, 2H), 0.43-0.34 (m, 2H); ^{13}C NMR δ 146.8 (s), 128.2 (d), 126.7 (d), 125.9 (d), 66.4 (t), 43.2 (d), 24.6 (d), 21.1 (q), 20.5 (d), 9.9 (t); EI MS *m/z* (relative intensity) 158 (M^+-H_2O, 17), 143 (15), 118 (90), 117 (100), 115 (16), 106 (52), 105 (79), 91 (29). Anal. Calcd for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.72; H, 9.18.

Preparation of compound 5

3-Cyclohexyl-1,1-dibromobut-1-ene (17). To a solution of CBr_4 (9.75 g, 29.40 mmol) in dry CH_2Cl_2 (50 mL) at 0 °C, was added PPh_3 (15.4 g, 58.7 mmol). After 15 min, 2-cyclohexylpropionaldehyde (2.0 g, 14.7 mmol) was added slowly. After 1 h at rt, the reaction mixture was diluted with pentane (100 mL), the precipitate was filtered off through Celite and thoroughly washed with pentane. The filtrate was concentrated under reduced pressure and the crude product was taken up in pentane. The solids were filtered off again and the filtrate concentrated under reduced pressure. The crude material was purified by flash chromatography (pentane) to give 2.95 g (68%) of **17** as a colorless oil; IR 1615, 1445, 800, 770 cm^{-1}; ^1H NMR δ 6.22 (d, J = 9.9 Hz, 1H), 2.31-2.23 (m, 1H), 1.75-1.66 (m, 4H), 1.26-1.08 (m, 4H), 1.02-0.70 (m, 3H), 0.96 (d, J = 7.0 Hz, 3H); ^{13}C NMR δ 143.4 (d), 87.2 (s), 43.8 (d), 42.7 (d), 30.4 (t), 30.3 (t), 26.5 (t), 26.4 (t), 26.3 (t), 16.3 (q); EI MS *m/z* (relative intensity) 298 (M^{+}+4, 1), 296 (M^{+}+2, 2), 294 (M^{+}, 1), 135 (11), 110 (18), 83 (100), 82 (29), 67 (10), 55 (67).

4-Cyclohexylpent-2-yn-1-ol (18). To a solution of **17** (2.95 g, 9.96 mmol) in dry THF (30 mL) at -78 °C, was added *n*-BuLi (8 mL, 2.5M in hexanes, 20 mmol). After 10 min at -78 °C, dry paraformaldehyde (0.9 g, 30.0 mmol of monomeric HCHO) was added in one portion and the cooling bath was removed immediately. The reaction mixture was allowed to warm to rt. After 1 h, the reaction mixture was poured into a saturated aqueous NH_4Cl solution and extracted with ether. The combined extracts were washed with brine, dried over Na_2CO_3, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/ether: 80/20) to give 1.25 g (75%) of **18** as a pale yellow liquid; IR 3300 (br), 2230, 1450, 1020 cm^{-1}; ^{1}H NMR δ 4.27 (d, J = 2.0 Hz, 2H), 2.32 (m, 1H), 1.82-1.64 (m, 7H), 1.25-1.00 (m, 5H), 1.13 (d, J = 7.0 Hz, 3H); ^{13}C NMR δ 90.0 (s), 79.3 (s), 51.4 (t), 42.6 (d), 31.9 (d), 30.9 (t), 29.5 (t), 26.5 (t), 26.4 (t), 26.3 (t), 18.2 (q); EI MS *m/z* (relative intensity) 148 (M^{+}-H_2O, 6), 135 (22), 119 (15), 91 (17), 83 (34), 81 (22), 79 (17), 66 (100), 55 (70), 53 (14). Anal. Calcd for $C_{11}H_{18}O$: C, 79.46; H, 10.91. Found: C, 79.52; H, 11.01.

(*Z*)-4-Cyclohexylpent-2-en-1-ol (5). A solution of CuBr (0.91 g, 6.34 mmol) and LiBr (0.99 g, 11.40 mmol) in dry THF (6 mL), was added slowly to a refluxing activated zinc suspension [prepared from zinc dust (3.26 g, 50.60 mmol) and 1,2-dibromoethane (0.40 mL, 4.64 mmol) in isopropanol (15 mL); reflux 10 min]. After 20 min, a solution of **18** (0.70 g, 4.21 mmol) in isopropanol (5 mL) was added slowly. After 5 h reflux, the reaction mixture was cooled to rt, diluted with ether (100 mL) and filtered through Celite. The filtrate was treated with a saturated aqueous NH_4Cl solution and was extracted with AcOEt. The combined extracts were dried over $MgSO_4$, concentrated under reduced pressure, to give an oil which was purified by flash chromatography (cyclohexane/AcOEt: 70/30, 60/40). 0.56 g (79%) of **5** was obtained as a clear yellow liquid; IR 3320 (br), 1450, 1030, 1010 cm^{-1}; ^{1}H NMR δ 5.55 (dtd, J = 11.0, 7.0 and 0.7 Hz, 1H), 5.35 (m, 1H), 4.24-4.09 (m, 2H), 2.23 (m, 1H), 1.72-1.61 (m, 5H), 1.60 (s, 1H, OH), 1.25-1.00 (m, 4H), 0.95-0.82 (m, 2H), 0.93 (d, J = 6.6 Hz, 3H); ^{13}C NMR δ 138.0 (d), 127.1 (d), 58.8 (t), 43.1 (d), 37.4 (d), 30.6 (t), 30.3 (t), 26.6 (t), 26.5 (t), 26.4 (t), 18.4 (q); EI MS *m/z* (relative intensity) 168 (M^+, 0.3), 150 (M^+-H_2O, 21), 110 (23), 83 (23), 81 (25), 69 (17), 68 (100), 67 (42), 55 (59), 53 (15). Anal. Calcd for $C_{11}H_{20}O$: C, 78.51; H, 11.98. Found: C, 78.44; H, 11.97.

Et_2Zn/ICH_2Cl promoted cyclopropanation of 5. 5 (0.10 g, 0.59 mmol) was cyclopropanated by using Et_2Zn (2.40 mL, 1M in hexanes, 2.40 mmol), ICH_2Cl (0.35 mL, 4.81 mmol) in DCE (12 mL). The crude material was purified by filtration through silica gel (cyclohexane/AcOEt: 80/20) to give 0.087 g (80%) of a diastereoisomeric mixture of **8a** and **8b** in a 20/80 ratio.

Sm(Hg)/ICH_2Cl promoted cyclopropanation of 5. 5 (0.10 g, 0.59 mmol) was cyclopropanated by using Sm (0.45 g, 2.99 mmol), ICH_2Cl (0.22 mL, 3.02 mmol), $HgCl_2$ (0.13 g, 0.48 mmol) in THF (16 mL). The crude material was purified by filtration through silica gel (cyclohexane/AcOEt: 80/20) to give 0.083 g (76%) of a diastereoisomeric mixture of **8a** and **8b** in a 15/85 ratio.

(1*S,2*S**)-2-((1*S**)-1-cyclohexylethyl)cyclopropanemethanol (8a).** IR 3320 (br), 3050, 1450, 1020 cm^{-1}; ^1H NMR δ 3.75 (dd, J = 11.0 and 7.0 Hz, 1H), 3.49 (dd, J = 11.0 and 7.7 Hz, 1H), 1.74-1.64 (m, 6H), 1.24-0.68 (m, 13H), 0.05 (m, 1H); ^{13}C NMR δ 63.1 (t), 44.1 (d), 38.5 (d), 31.0 (t), 29.6 (t), 26.9 (t), 26.8 (t), 26.7 (t), 21.7 (d), 17.8 (q), 17.3 (d), 10.0 (t); EI MS *m/z* (relative intensity) 164 (M^+-H_2O, 1), 124 (39), 123 (25), 110 (32), 109 (28), 95 (28), 83 (30), 82 (70), 81 (87), 69 (31), 67 (66), 55 (100). Anal. Calcd for $C_{12}H_{22}O$: C, 79.06; H, 12.16. Found: C, 79.00; H, 12.30.

(1*R,2*R**)-2-((1*S**)-1-cyclohexylethyl)cyclopropanemethanol (8b).** IR 3320 (br), 3050, 1450, 1020 cm^{-1}; ^1H NMR δ 3.82 (dd, J = 11.0 and 6.2 Hz, 1H), 3.37 (dd, J = 11.0, 8.5 Hz, 1H), 1.74-1.64 (m, 6H), 1.24-0.68 (m, 13H), -0.08 (m, 1H); ^{13}C NMR δ 63.6 (t), 43.8 (d), 38.1 (d), 31.3 (t), 29.1 (t), 26.9 (t), 26.8 (t), 26.7 (t), 22.1 (d), 20.0 (d), 17.1 (q), 8.4 (t) ; EI MS *m/z* (relative intensity) 164 (M^+-H_2O, 1), 124 (47), 123 (35), 110 (24), 109 (28), 96 (18), 95 (38), 83 (29), 82 (68), 81 (87), 79 (26), 69 (29), 68 (20), 55 (100), 53 (28). Anal. Calcd for $C_{12}H_{22}O$: C, 79.06; H, 12.16. Found: C, 79.00; H, 12.30.

Correlation for the determination of the relative stereochemistry of 8a

(1*S,2*S**)-2-[1(*R**)-1-(cyclohexa-1,4-dien-1-yl)ethyl]cyclopropanemethanol (10a).** Compound **2a** (0.53 g, 3.00 mmol) in dry *t*-BuOH (3.0 g, 40.5 mmol) and dry THF (10 mL) was added slowly to dry liquid ammonia (20 mL). Lithium (0.10 g, 14.41 mmol, 4.80 equiv.), freshly cut into four pieces, was then added to the refluxing reaction mixture, at such a rate that the initially observed blue colour has discharged. Once the addition was complete, solid NH_4Cl (2 g) was added in one portion and the ammonia was allowed to evaporate. Ether was then added followed by the addition of a saturated aqueous NH_4Cl solution. The layers were separated and the aqueous one was extracted with ether. The combined extracts were dried over $MgSO_4$, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/AcOEt: 80/20) to give 0.38 g (72%) of **10a** as a colorless oil; ^{1}H NMR δ 5.73 (m, 2H), 5.47 (s, 1H), 3.65 (dd, J = 7.7 and 7.0 Hz, 2H), 2.69 (m, 4H), 1.81 (s, 1H, OH), 1.60 (m, 1H), 1.2-1.07 (m, 1H), 1.14 (d, J = 7.0 Hz, 3H), 0.95-0.83 (m, 1H), 0.72 (m, 1H), 0.10 (m, 1H); ^{13}C NMR δ 139.2 (s), 124.5 (d), 124.1 (d), 117.2 (d), 62.8 (t), 40.2 (d), 26.6 (t), 26.5 (t), 21.7 (d), 19.8 (q), 18.3 (d), 9.2 (t); EI MS *m/z* (relative intensity) 178 (M^+, 0.5), 160 (M^+-H_2O, 2), 145 (13), 131 (23), 119 (37), 118 (27), 117 (19), 106 (14), 105 (57), 92 (17), 91 (100), 79 (49), 78 (25), 77 (38), 67 (17), 65 (12), 55 (16).

Reduction of 10a with diimide. To a stirred mixture of potassium azodicarboxylate (1.98 g, 5.04 mmol) and **10a** (0.30 g, 1.68 mmol) in EtOH (20 mL), was added dropwise AcOH (1.2 mL, 10.1 mmol). After 24 h at 70 °C, the reaction mixture was filtered, evaporated under reduced pressure and the reduction process was repeated with the crude material. After the second reduction, a crude mixture of **11a** and **8a** was obtained in a 70/30 ratio, observed by ^{1}H NMR and GC/MS analysis.

(1S*,2S*)-2-[1(R*)-(cyclohex-1-en-1-yl)ethyl]cyclopropanemethanol (11a). ^{1}H NMR δ 5.43 (m, 1H), 3.73 (m, 1H), 3.51 (m, 1H), 2.00 (m, 5H), 1.38-1.03 (m, 5H), 1.11 (d, J = 7.0 Hz, 3H), 0.96-0.82 (m, 1H), 0.76-0.67 (m, 1H), 0.02 (m, 1H); EI MS *m/z* (relative intensity) 180 (M^+, 0.6), 162 (M^+-H_2O, 5), 149 (62), 147 (22), 133 (24), 121 (26), 109 (27), 107 (73), 105 (40), 94 (20), 93 (62), 91 (62), 81 (51), 79 (100), 77 (45), 67 (44), 55 (25), 53 (20).

Preparation of compound 6

1,1-Dibromo-4-[4-(methoxybenzyl)oxy]-3-methylbut-1-ene (19). To a solution of CBr_4 (15.0 g, 45.2 mmol) in dry CH_2Cl_2 (100 mL) at 0 °C, was added PPh_3 (22.5 g, 85.8 mmol). After 30 min, 3-[4-(methoxybenzyl)oxy]-2-methylpropionaldehyde (8.9 g, 42.7 mmol) was added slowly. After 1 h at rt, the reaction mixture was diluted with cyclohexane (300 mL), the precipitated solids were filtered off through Celite and thoroughly washed with cyclohexane. The filtrate was concentrated under reduced pressure and the crude product was taken up in cyclohexane. The solids were filtered off again and the filtrate concentrated under reduced pressure. The crude material was purified by flash chromatography (toluene/ether: 90/10) to give 12.3 g (79%) of **19** as a colorless oil; IR 1610, 1585, 1510, 1250, 1090, 1030, 845, 820, 785 cm^{-1}; ^1H NMR δ 7.28-7.25 (m, 2H), 6.92-6.88 (m, 2H), 6.31 (d, J = 8.8 Hz, 1H), 4.48 (d, J = 11.8 Hz, 1H), 4.44 (d, J = 11.8 Hz, 1H), 3.83 (s, 3H), 3.38-3.34 (m, 2H), 2.83-2.74 (m, 1H), 1.06 (d, J = 7.0 Hz, 3H); ^{13}C NMR δ 159.1 (s), 141.1 (d), 130.2 (s), 129.0 (d), 113.7 (d), 88.7 (s), 72.6 (t), 72.5 (t), 55.1 (q), 38.6 (d), 15.8 (q); EI MS m/z (relative intensity) 366 (M^++4, 1), 364 (M^++2, 2), 362 (M^+, 1), 136 (17), 121 (100), 91 (3), 78 (6).

5-[4-(methoxybenzyl)oxy]-4-methyl-pent-2-yn-1-ol (20). To a solution of **19** (12.3 g, 33.8 mmol) in dry THF (100 mL) at -78 °C, was added n-BuLi (30 mL, 2.5M in hexanes, 75 mmol). After 10 min at -78 °C, dry paraformaldehyde (3 g, 100 mmol of monomeric HCHO) was added in one portion and the cooling bath was removed immediately. After stirring overnight at rt, the reaction mixture was poured into a saturated aqueous NH_4Cl solution and extracted with ether. The combined extracts were washed with brine, dried over $MgSO_4$, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/AcOEt: 90/10) to give 6.4 g (82%) of **20** as colorless oil; IR 3400 (br), 2240, 1610, 1585, 1510, 1250, 1080, 1030, 820 cm^{-1}; ^1H NMR δ 7.29-7.25 (m, 2H), 6.91-6.86 (m, 2H), 4.52 (d, J= 12.0 Hz, 1H), 4.47 (d, J= 12.0 Hz, 1H), 4.26 (m, 2H), 3.82 (s, 3H), 3.48 (dd, J = 9.0 and 6.4 Hz, 1H), 3.34 (dd, J = 9.0 and 7.1 Hz, 1H), 2.75 (m, 1H), 1.65 (s, 1H, OH), 1.19 (d, J = 7.0 Hz, 3H); ^{13}C NMR δ 158.9 (s), 129.8 (s), 129.2 (d), 113.5 (d), 87.3 (s), 79.4 (s), 73.3 (t), 72.3 (t), 54.9 (q), 50.6 (t), 26.5 (d), 17.4

(q); EI MS *m/z* (relative intensity) 234 (M^+, 1), 203 (21), 122 (9), 121 (100). Anal. Calcd for $C_{14}H_{18}O_3$: C, 71.77; H, 7.74. Found: C, 71.54; H, 7.86.

(*Z*)-5-[4-(methoxybenzyl)oxy]-4-methylpent-2-en-1-ol (6). A solution of CuBr (4.6 g, 32.1 mmol) and LiBr (5.0 g, 57.6 mmol) in dry THF (25 mL), was added slowly to a refluxing activated zinc suspension [prepared from zinc dust (16.8 g, 256.9 mmol) and 1,2-dibromoethane (2.0 mL, 23.2 mmol) in isopropanol (80 mL); reflux 10 min]. After 20 min, a solution of **20** (5.0 g, 21.3 mmol) in isopropanol (20 mL) was added slowly. After 2 h reflux and stirring overnight at rt, the reaction mixture was diluted with ether (400 mL) and filtered through Celite. The filtrate was then treated with a saturated aqueous NH_4Cl solution, the organic phase was separated and the aqueous layer was extrated with AcOEt. The combined extracts were washed with brine, dried over Na_2CO_3, concentrated under reduced pressure to give an oil which was purified by flash chromatography (cyclohexane/AcOEt: 80/20, 70/30) to give 4.1 g (82%) of **6** as a colorless liquid; IR 3400 (br), 3000, 1610, 1585, 1510, 1250, 1080, 1030, 820, 755 cm^{-1}; ^1H NMR δ 7.27-7.22 (m, 2H), 6.91-6.86 (m, 2H), 5.82 (dddd, J = 11.0, 7.9, 6.6 and 1.1 Hz, 1H), 5.35 (m, 1H), 4.45 (s, 2H), 4.20 (ddd, J = 12.1, 7.9 and 1.1 Hz, 1H), 3.93 (dd, J = 12.1 and 6.6 Hz, 1H), 3.81 (s, 3H), 3.38 (dd, J = 8.8 and 4.8 Hz, 1H), 3.13 (t, J = 8.8 Hz, 1H), 2.93-2.83 (m, 1H), 2.15 (s, 1H, OH), 0.96 (d, J = 6.6 Hz, 3H); ^{13}C NMR δ 159.1 (s), 136.7 (d), 129.6 (s), 129.3 (d, 2 C), 113.7 (d), 74.1 (t), 72.8 (t), 57.7 (t), 55.1 (q), 32.3 (d), 17.2 (q); EI MS *m/z* (relative intensity) 236 (M^+, 2), 150 (10), 122 (10), 121 (100). Anal Calcd for $C_{14}H_{20}O_3$: C, 71.16; H, 8.53. Found: C, 70.99; H, 8.63.

Et_2Zn/ICH_2Cl promoted cyclopropanation of 6. 6 (0.40 g, 1.69 mmol) was cyclopropanated by using Et_2Zn (6.8 mL, 1M in hexanes, 6.80 mmol), ICH_2Cl (1.0 mL, 13.7 mmol) in DCE (20 mL). The crude material was filtered through silica gel (cyclohexane/AcOEt: 70/30) to give 0.34 g (81%) of a pure diastereoisomeric mixture of **9a** and **9b** in a 70/30 ratio. Purification by flash chromatography (toluene/ether: 80/20) gave 0.084 g of **9b** (20%) and 0.20 g (47%) of **9a** as colorless oils.

Sm(Hg)/ICH_2Cl promoted cyclopropanation of 6. 6 (0.40 g, 1.69 mmol) was cyclopropanated by using Sm (1.30 g, 8.64 mmol), ICH_2Cl (0.62 mL, 8.51 mmol), $HgCl_2$ (0.37 g, 1.36 mmol) in

THF (50 mL). Purification by flash chromatography (toluene/ether: 80/20) gave 0.23 g (54 %) of **9b** and 0.15 g (36%) of **9a** as colorless oils.

(1*S,2*R**)-2-{2-[4-methoxybenzyl)oxy]-1(*R**)-1-methylethyl}cyclopropanemethanol (9a).** IR 3440 (br), 3050, 1610, 1585, 1510, 845, 820, 735 cm^{-1}; ^1H NMR δ 7.29-7.24 (m, 2H), 6.90-6.85 (m, 2H), 4.48 (d, J = 11.6 Hz, 1H), 4.43 (d, J = 11.6 Hz, 1H), 3.81 (s, 3H), 3.68 (dd, J = 11.4 and 7.3 Hz, 1H), 3.58 (dd, J = 11.4 and 7.3 Hz, 1H), 3.51 (dd, J = 9.0 and 4.6 Hz, 1H), 3.30 (dd, J = 9.0 and 7.5 Hz, 1H), 1.43 (s, 1H, OH), 1.43-1.25 (m, 1H), 1.20-1.08 (m, 1H), 1.08 (d, J = 6.6 Hz, 3H), 0.75-0.65 (m, 2H), 0.10-0.06 (m, 1H); ^{13}C NMR δ 158.9 (s), 130.7 (s), 128.9 (d), 113.6 (d), 75.8 (t), 72.6 (t), 62.7 (t), 55.1 (q), 33.9 (d), 20.3 (d), 18.2 (d), 17.4 (q), 8.5 (t); EI MS *m/z* (relative intensity) 250 (M^+, 2), 137 (7), 121 (100), 78 (7), 77 (7); Anal. Calcd. for $C_{15}H_{22}O_3$: C, 71.97; H, 8.86. Found: C, 71.80; H, 8.93.

(1*R,2*S**)-2-{2-[4-methoxybenzyl)oxy]-1(*R**)-1-methylethyl}cyclopropane methanol (9b).** IR 3420 (br), 3050, 1610, 1585, 1510, 845, 820, 740 cm^{-1}; ^1H NMR δ 7.31-7.28 (m, 2H), 6.91-6.87 (m, 2H), 4.52 (d, J = 12.0 Hz, 1H), 4.48 (d, J = 12.0 Hz, 1H), 3.90 (dd, J = 11.8 and 4.6 Hz, 1H), 3.80 (s, 3H), 3.48 (dd, J = 8.8 and 4.0 Hz, 1H), 3.22 (dd, J = 10.3 and 8.8 Hz, 1H), 3.09 (t, J = 11.8 Hz, 1H), 1.48-1.44 (m, 1H), 1.30-1.26 (m, 1H), 0.95 (d, J = 7.0 Hz, 3H), 0.65-0.53 (m, 2H), -0.16- -0.21(m, 1H); ^{13}C NMR δ 159.3 (s), 129.6 (d), 129.1 (s), 113.7 (d), 76.2 (t), 73.0 (t), 62.5 (t), 55.0 (q), 32.2 (d), 21.6 (d), 19.4 (d), 17.7 (q), 5.7 (t); EI MS *m/z* (relative intensity) 250 (M^+, 2), 137 (19), 121 (100), 78 (8), 77 (8); Anal. Calcd. for $C_{15}H_{22}O_3$:C, 71.97; H, 8.86. Found: C, 71.78; H, 8.95.

Correlation for the determination of the relative stereochemistry of 9a and 9b

2(*R)-2-[(1*R**,2*S**)-2-(acetoxymethyl)cyclopropyl]propanoic acid (12a).**

Preparation from 2a. To a solution of **2a** (0.20 g, 1.13 mmol) in dry CH_2Cl_2 (10 mL) containing DMAP (0.0075 g, 0.0614 mmol) was added Ac_2O (0.35 mL, 3.71 mmol). After 1 h at rt, MeOH (2 mL) was added dropwise, followed by the addition of a saturated aqueous $NaHCO_3$ solution. The organic phase was separated and the aqueous layer was extracted with ether. The combined extracts were dried over $MgSO_4$, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/AcOEt: 9/1) to give 0.24 g (98%) of the corresponding acetate, which was dissolved in a mixture of CCl_4 (3.8 mL), MeCN (3.8 mL), H_2O (5.6 mL). $RuCl_3xH_2O$ (0.0043 g, 0.0207 mmol) and $NaIO_4$ (2.9 g, 13.7 mmol) were added to the vigorously stirred reaction mixture. After 16 h at rt, CH_2Cl_2 (10 mL) and water (10 mL) were added. The organic layer was separated and the aqueous one was extracted with CH_2Cl_2. The combined extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure to give a crude material which was taken up in ether. After 24 h stirring, the mixture was filtered through Celite, and the crude carboxylic acid was purified by flash chromatography (cyclohexane/AcOEt: 70/30) to give 0.09 g (52%) of **12a** as a waxy solid.

Preparation from 9a. To a solution of **9a** (0.12 g, 0.45 mmol) in dry CH_2Cl_2 (5 mL) containing a catalytic amount of DMAP, was added Ac_2O (0.40 mL, 4.24 mmol). After 1 h at rt, the reaction mixture was quenched with MeOH (1 mL). After addition of ether and water, the organic phase was separated and the aqueous layer was extracted with ether. The combined extracts were neutralized with saturated aqueous $NaHCO_3$ solution, dried over $MgSO_4$, concentrated under reduced pressure and the crude acetate was dissolved in a mixture of CH_2Cl_2 (5 mL) and H_2O (0.5 mL). DDQ (0.11 g, 0.48 mmol) was then added in one portion at 0 °C and after 1 h at rt, the reaction mixture was diluted with water and extracted with AcOEt. The combined extracts were washed with brine, dried over $MgSO_4$, concentrated under reduced pressure and the crude material was purified by rapid filtration on silica gel (CH_2Cl_2/ether: 100/0, 70/30) to give an alcohol which was dissolved in dry DMF (3 mL). PDC (1.00 g, 2.66 mmol) was added in one portion and after 24 h at rt, water was added. The resulting mixture was extracted with AcOEt, the combined extracts were washed with brine, dried over Na_2SO_4, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/AcOEt: 70/30 to 60/40) to give 0.047 g (54%) of **12a** as a waxy solid; mp 36-38 °C; IR 3500-2500 (br), 1735, 1710 cm^{-1}; ^1H NMR δ 8.40 (br s, 1H, OH), 4.30 (dd, J = 11.9 and 6.4 Hz, 1H), 3.87 (dd, J= 11.9 and 9.0 Hz, 1H), 2.08 (s, 3H), 2.07-1.99 (m, 1H), 1.33-1.25 (m, 1H), 1.32 (d, J = 6.9 Hz, 3H), 1.15-1.09 (m, 1H), 0.91-0.83 (m, 1H), 0.34 (dd, J = 11.2 and 5.6 Hz, 1H); ^{13}C NMR δ 182.1 (s), 171.1 (s), 64.5 (t), 39.4 (d), 21.0 (q), 20.1 (d), 17.4 (q), 14.3 (d), 8.9 (t). Anal. Calcd. for $C_9H_{14}O_4$: C, 58.05; H 7.58. Found: C, 57.89; H, 7.72.

2(*R)-2-[(1*S**,2*R**)-2-(acetoxymethyl)cyclopropyl]propanoic acid (12b).** To a solution of **9b** (0.15 g, 0.60 mmol) in dry CH_2Cl_2 (5 mL) containing a catalytic amount of DMAP, was added Ac_2O (0.40 mL, 4.24 mmol). After 1 h at rt, the reaction mixture was quenched with MeOH (1 mL). After addition of ether and water, the organic phase was separated and the aqueous layer was extracted with ether. The combined extracts were neutralized with saturated aqueous $NaHCO_3$ solution, dried over $MgSO_4$, concentrated under reduced pressure and the crude acetate was dissolved in a mixture of CH_2Cl_2 (5 mL) and H_2O (0.5 mL). DDQ (0.15 g, 0.66 mmol) was then added in one portion at 0 °C and after 1 h at rt, the reaction mixture was diluted with water and extracted with AcOEt. The combined extracts were washed with brine, dried over $MgSO_4$, concentrated under reduced pressure and the crude material was purified by rapid filtration through silica gel (CH_2Cl_2/ether: 100/0, 70/30) to give an alcohol which was dissolved in dry DMF (3 mL). PDC (1.00 g, 2.66 mmol) was added in one portion and after 24 h at rt, water was added. The resulting mixture was extracted with AcOEt, the combined extracts were washed with brine, dried over Na_2SO_4, concentrated under reduced pressure and the crude material was purified by flash chromatography (cyclohexane/AcOEt: 60/40) to give 0.064 g (56%) of **12b** as a white solid; mp 63-64 °C; IR 3500-2500 (br), 1740, 1710 cm^{-1}; ^1H NMR δ 4.27 (dd, J = 12.0 and 7.1 Hz, 1H), 3.94 (dd, J = 12.0 and 8.7 Hz, 1H), 2.07-1.96 (m, 1H), 2.02 (s, 3H), 1.35-1.25 (m, 1H), 1.33 (d, J = 7.0 Hz, 3H), 1.23-1.12 (m, 1H), 0.92- 0.85 (td, J = 8.4 and 5.2 Hz, 1H), 0.14 (dd, J = 11.0 and 5.5 Hz, 1H); ^{13}C NMR δ 182.4 (s), 171.7 (s), 65.2 (t), 39.4 (d), 20.9 (q), 19.7 (d), 18.0 (q), 15.1 (d), 9.0 (t). Anal. Calcd. for $C_9H_{14}O_4$: C, 58.05; H, 7.58. Found: C, 58.02; H, 7.61.

Crystal Data

A selected crystal was set up on an automatic diffractometer. Unit cell dimensions with estimated standard deviations were obtained from least-squares refinements of the setting angles of 25 well centered reflections. Two standard reflections were monitored periodically; they showed no change during data collection. Crystallographic data and other information are summarized in Table I. Corrections were made for Lorentz and polarisation effects. An extinction correction was necessary.

Computations were performed by using the PC version of CRYSTALS.[1] Atomic form factors for neutral O, C and H were taken from ref. 2. Real and imaginary parts of anomalous dispersion were taken into account. The structure was solved by direct methods using the SHELXS86[3] program and succesive fourrier maps. All hydrogens atoms have been located on difference maps, their position were not refined and they were given an overall thermal parameter.

Oxygen atom was anisotropically refined, other atoms were isotropically refined. Full matrix least-squares refinements were carried out by minimising the function

$$\sum w(F_0 - |F_C|)^2$$

where Fo and F_C are the observed and calculated structure factors. Models reached convergence with

$$R = \sum(|F_O - |F_C||) / \sum wF_O \quad \text{and} \quad Rw = \left[\sum w(F_O - |F_C|^2) / \sum (F_O)^2\right]^{1/2}$$

having value listed in table I.

In the last stages of the refinement, each reflection was assigned a weight:unity. Criteria for a satisfactory complete analysis were the ratio of rms shift to standard deviations being less than 0.2 and no significant features in the difference map. There are hydrogens bonds between molecules building infinite chains parallel to axis a.

Atomic coordinates are given in table II. Interatomic distances and bond angles in Table III. Supplementary material available: Table of atomic parameters for hydrogen atoms, observed and calculated strucure factors. The figure represents a view of the molecule using CAMERON.[4]

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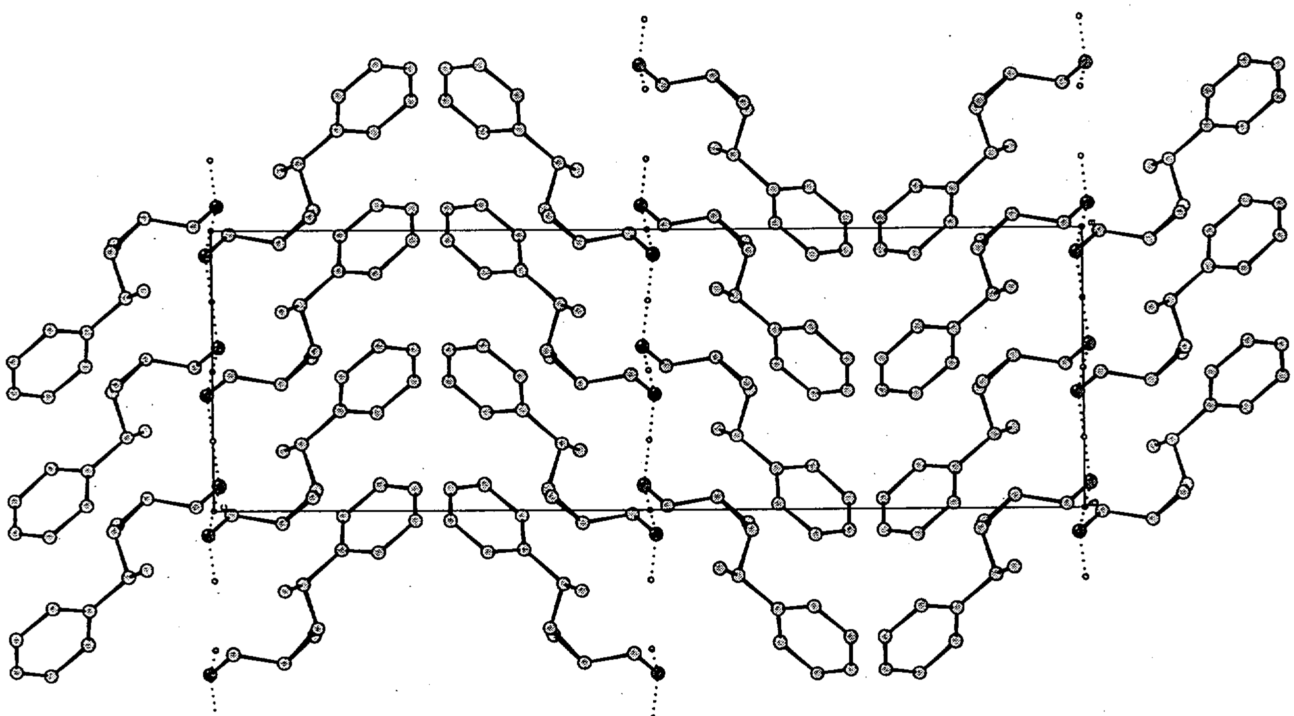
1. D.J. Watkin, J.R. Carruthers and P.W. Betteridge. CRYSTALS, An advanced Crystallographic Computer Program, Chemical Crystallography Laboratory, Oxford (1989).
2. International Tables for X-ray Crystallography, Vol. IV, Kynoch Press, Birmingham, England, 1974.
3. G.M. Sheldrick. SHELXS86, Program for Crystal Structure Solution, University of Göttingen. Göttingen.
4. L.J. Pearce and D.J. Watkin. Chemical Crystallography Laboratory, Oxford

NUMBER OF REFLECTIONS WITH I > nSIGMA(I)

CONDITIONS	<1S	<2S	<3S	<4S	<5S	<6S	<7S	<8S	<9S	<10S	>10S	N	AVINT	AVSIG
00L : L = 2N+1 *	3	2	0	0	0	1	0	0	0	0	3	9.	1266.3	162.7
0K0 : K = 2N+1 *	0	0	0	0	0	0	0	0	0	0	0	0.	0.0	0.0
H00 : H = 2N+1 *	0	0	0	0	0	0	0	0	0	0	0	0.	0.0	0.0
0KL : K = 2N+1 *	0	0	0	0	0	0	0	0	0	0	0	0.	0.0	0.0
0KL : L = 2N+1 *	41	16	0	2	2	1	3	0	2	3	13	83.	598.0	130.3
0KL : K+L = 2N+1 *	41	16	0	2	2	1	3	0	2	3	13	83.	598.0	130.3
H0L : H = 2N+1 *	0	0	0	0	0	0	0	0	0	0	0	0.	0.0	0.0
H0L : L = 2N+1 *	86	26	2	2	1	3	5	4	5	13	39	186.	2413.8	143.2
H0L : H+L = 2N+1 *	86	26	2	2	1	3	5	4	5	13	39	186.	2413.8	143.2
HK0 : H = 2N+1 *	94	40	2	7	3	7	5	11	16	7	80	272.	3438.8	148.8
HK0 : K = 2N+1 *	94	40	2	7	3	7	5	11	16	7	80	272.	3438.8	148.8
HK0 : H+K = 2N+1 *	0	0	0	0	0	0	0	0	0	0	0	0.	0.0	0.0
ALL : H+K = 2N+1 *	0	0	0	0	0	0	0	0	0	0	0	0.	0.0	0.0
ALL : H+L = 2N+1 *	1850	661	41	57	35	56	50	75	109	112	533	3579.	969.8	125.3
ALL : K+L = 2N+1 *	1850	661	41	57	35	56	50	75	109	112	533	3579.	969.8	125.3
ALL : H+K+L = 2N+1 *	1799	665	32	41	36	44	58	71	94	115	496	3451.	903.3	125.3
ALL : -H+K+L = 3N+1 *	2486	887	51	59	60	74	79	106	125	142	709	4778.	1068.2	126.9
ALL : H-K+L = 3N+1 *	2453	923	51	60	51	61	68	97	137	170	714	4785.	1047.0	126.5
HHL : L = 2N+1 *	58	25	0	1	1	3	1	2	1	3	25	120.	1381.7	137.4
H-HL: L = 2N+1 *	66	30	1	0	0	2	3	3	4	6	39	154.	4474.2	164.1
00L : L = 3N+1 *	6	2	0	0	0	0	0	0	0	0	4	12.	1574.0	174.7
00L : L = 4N+1 *	5	2	0	0	0	1	0	0	0	1	4	13.	1713.6	168.2
00L : L = 6N+1 *	6	3	0	0	0	1	0	0	0	0	5	15.	1579.0	172.0

NUMBER OF REFLECTIONS WITH I > nSIGMA(I)

THETA	<1S	<2S	<3S	<4S	<5S	<6S	<7S	<8S	<9S	<10S	>10S	N
0.0 - 2.0 :	0	0	0	0	0	0	0	0	0	0	9	9
2.0 - 4.0 :	7	4	0	0	0	0	1	1	1	1	23	38
4.0 - 6.0 :	16	4	0	1	0	1	0	4	2	3	59	90
6.0 - 8.0 :	22	12	0	1	1	1	0	1	7	7	114	166
8.0 - 10.0 :	43	23	0	0	4	1	2	5	11	14	148	251
10.0 - 12.0 :	90	34	1	4	5	5	6	11	16	27	181	380
12.0 - 14.0 :	129	63	2	5	7	8	11	23	33	36	184	501
14.0 - 16.0 :	242	121	8	3	6	13	21	19	34	40	156	663
16.0 - 18.0 :	338	160	13	14	17	16	26	28	38	38	120	808
18.0 - 20.0 :	519	254	10	17	15	18	23	23	35	31	45	990
20.0 - 22.0 :	751	245	19	21	13	25	18	23	20	26	25	1186
22.0 - 24.0 :	991	295	15	23	14	16	5	14	4	7	3	1387
24.0 - 26.0 :	535	152	7	3	0	2	0	0	0	0	0	699
TOTAL :	3683	1367	75	92	82	106	113	152	201	230	1067	7168



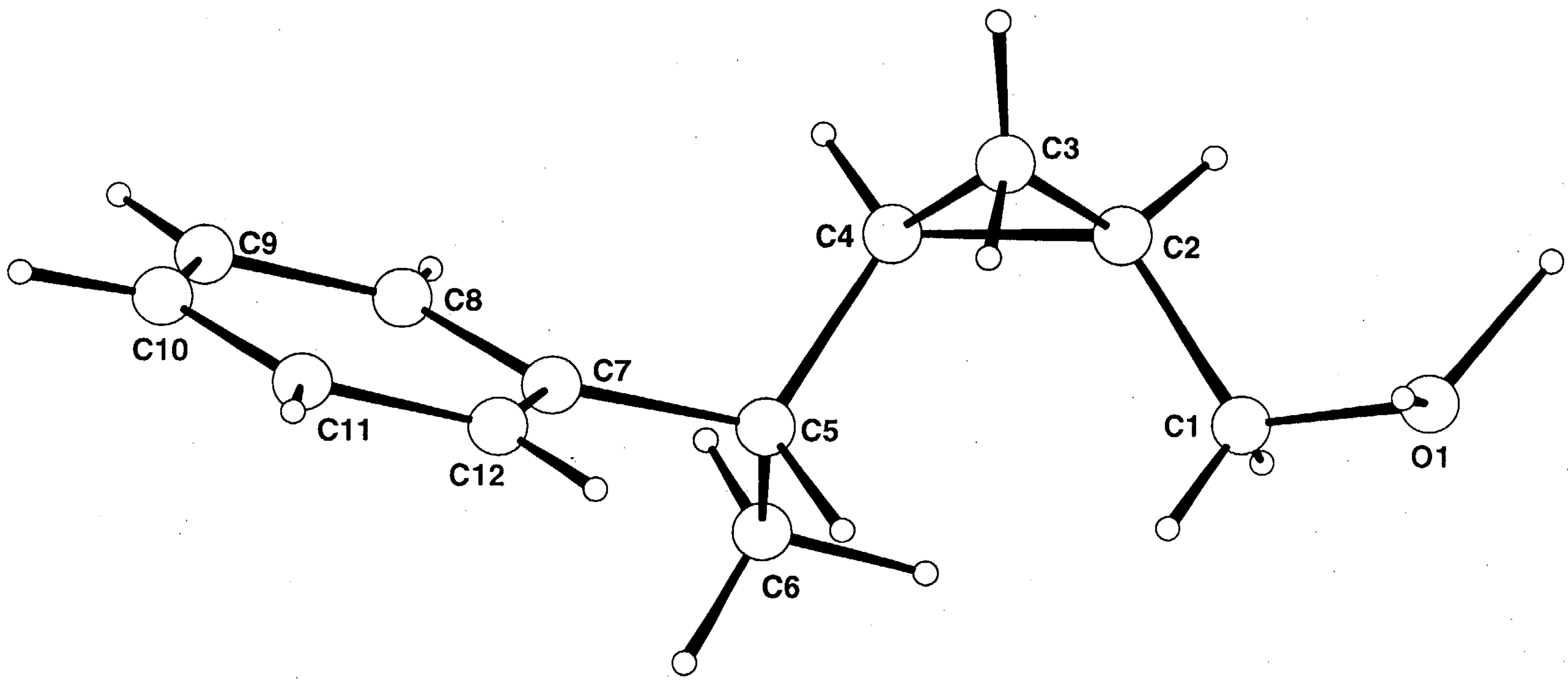

C3
C4
C2
C9
C8
C10
C7
C5
C1
O1
C11
C12
C6

TABLE I

CRYSTAL DATA: $C_{12}H_{16}O$	
Formula	$C_{12}H_{16}O$
Fw =	176.25
Crystal System	Orthorhombic
a (Å) =	8.119(2)
b (Å) =	10.356(2)
c (Å) =	25.363(7)
V ($Å^3$) =	2132(1)
Z =	8
Space group	P n a b
Crystal shape	parallelepiped
Crystal colour	colourless
Crystal dimensions (mm)	0.2,0.4,0.5
Linear absorption coefficient μ (cm^{-1})	0.63
Density ρ(g cm^3)	1.09
Diffractometer	MACH3
Radiation	MoKα (λ = 0.71069 Å)
Scan type	ω /2θ
Scan range (°)	0.8 + 0.345 tgθ
θ Limits (°)	1 - 25
Temperature of measurement	Room temperature
Octants collected	h:0, 9; k: 0, 12; l:0, 30
Nb of data collected	2194
Nb of unique data collected	1880
Nb of unique data used for refinement	709$(Fo)^2 > 3\sigma(Fo)^2$
Decay of standards reflections %	none
$R = \Sigma \lvert Fo - \lvert Fc \rvert\rvert / \Sigma Fo$	0.072
$Rw = [\Sigma w(Fo - \lvert Fc \rvert)^2 / \Sigma w Fo^2]^{1/2}$	0.063
Secondary extinction coefficient	135
Nb of variables	60
$\Delta\rho$min (e/ $Å^3$)	-0.21
$\Delta\rho$max (e/ $Å^3$)	0.29

Table II: Fractional atomic coordinates for :

Atom	x/a	y/b	z/c	U(iso)	U(eqv)
O(1)	0.0868(5)	0.3901(4)	0.5068(2)		0.0729
C(1)	0.0148(9)	0.2835(6)	0.4795(2)	0.069(2)	
C(2)	0.0462(8)	0.2849(6)	0.4219(2)	0.058(2)	
C(3)	-0.0485(8)	0.3724(7)	0.3869(3)	0.071(2)	
C(4)	-0.0736(8)	0.2299(6)	0.3825(2)	0.057(2)	
C(5)	-0.2368(7)	0.1717(6)	0.3991(2)	0.052(2)	
C(6)	-0.2116(9)	0.0341(7)	0.4203(3)	0.076(2)	
C(7)	-0.3589(7)	0.1750(5)	0.3540(2)	0.049(2)	
C(8)	-0.3506(8)	0.0880(6)	0.3123(2)	0.065(2)	
C(9)	-0.4635(9)	0.0958(7)	0.2700(3)	0.078(2)	
C(10)	-0.5779(9)	0.1891(7)	0.2699(3)	0.082(2)	
C(11)	-0.5910(9)	0.2743(7)	0.3096(3)	0.085(2)	
C(12)	-0.4806(9)	0.2674(6)	0.3521(3)	0.069(2)	

Table III: Interatomic distances (Å)and bond angles (°):

O(1) - C(1)	1.429(7)	C(1) - C(2)	1.482(8)		
C(2) - C(3)	1.484(8)	C(2) - C(4)	1.507(8)		
C(3) - C(4)	1.494(8)	C(4) - C(5)	1.516(8)		
C(5) - C(6)	1.537(8)	C(5) - C(7)	1.513(7)		
C(7) - C(8)	1.392(7)	C(7) - C(12)	1.376(8)		
C(8) - C(9)	1.414(8)	C(9) - C(10)	1.341(9)		
C(10) - C(11)	1.343(9)	C(11) - C(12)	1.402(9)		

O(1) - C(1) - C(2)	113.6(6)	C(1) - C(2) - C(3)	120.4(6)
C(1) - C(2) - C(4)	122.6(6)	C(3) - C(2) - C(4)	60.0(4)
C(2) - C(3) - C(4)	60.8(4)	C(2) - C(4) - C(3)	59.3(4)
C(2) - C(4) - C(5)	122.0(5)	C(3) - C(4) - C(5)	119.4(5)
C(4) - C(5) - C(6)	110.4(5)	C(4) - C(5) - C(7)	110.7(5)
C(6) - C(5) - C(7)	111.9(5)	C(5) - C(7) - C(8)	121.9(6)
C(5) - C(7) - C(12)	120.9(6)	C(8) - C(7) - C(12)	117.2(6)
C(7) - C(8) - C(9)	120.6(6)	C(8) - C(9) - C(10)	119.4(7)
C(9) - C(10) - C(11)	121.9(7)	C(10) - C(11) - C(12)	119.4(7)
C(7) - C(12) - C(11)	121.5(7)		

Table: Hydrogen atoms interatomic distances (Å) and bond angles (°) for $C_{12}H_{16}O$

O(1)	- H(1)	1.339(4)	O(1)	- H(2)	1.350(4)
C(1)	- H(11)	1.089(7)	C(1)	- H(12)	0.959(7)
C(2)	- H(21)	0.976(6)	C(3)	- H(31)	0.963(7)
C(3)	- H(32)	0.955(7)	C(4)	- H(41)	1.017(6)
C(5)	- H(51)	1.068(6)	C(6)	- H(61)	1.158(7)
C(6)	- H(62)	0.897(7)	C(6)	- H(63)	1.006(7)
C(8)	- H(81)	0.974(6)	C(9)	- H(91)	1.021(7)
C(10)	- H(101)	0.992(7)	C(11)	- H(111)	0.967(8)
C(12)	- H(121)	0.879(7)			

C(1)	- O(1)	- H(1)	113.0(4)	C(1)	- O(1)	- H(2)	112.1(4)
H(1)	- O(1)	- H(2)	116.6(3)				
O(1)	- C(1)	- H(11)	112.6(5)	C(2)	- C(1)	- H(11)	102.4(5)
O(1)	- C(1)	- H(12)	105.0(6)	C(2)	- C(1)	- H(12)	111.1(6)
H(11)	- C(1)	- H(12)	112.4(6)	C(1)	- C(2)	- H(21)	98.3(5)
C(3)	- C(2)	- H(21)	128.7(6)	C(4)	- C(2)	- H(21)	126.3(6)
C(2)	- C(3)	- H(31)	117.4(6)	C(4)	- C(3)	- H(31)	111.7(6)
C(2)	- C(3)	- H(32)	119.8(6)	C(4)	- C(3)	- H(32)	115.5(6)
H(31)	- C(3)	- H(32)	117.9(7)	C(2)	- C(4)	- H(41)	117.7(5)
C(3)	- C(4)	- H(41)	121.9(6)	C(5)	- C(4)	- H(41)	109.2(5)
C(4)	- C(5)	- H(51)	108.0(5)	C(6)	- C(5)	- H(51)	105.2(5)
C(7)	- C(5)	- H(51)	110.5(5)	C(5)	- C(6)	- H(61)	111.4(5)
C(5)	- C(6)	- H(62)	104.5(6)	H(61)	- C(6)	- H(62)	111.3(7)
C(5)	- C(6)	- H(63)	108.4(6)	H(61)	- C(6)	- H(63)	106.3(6)
H(62)	- C(6)	- H(63)	115.0(7)	C(7)	- C(8)	- H(81)	120.6(6)
C(9)	- C(8)	- H(81)	118.8(6)	C(8)	- C(9)	- H(91)	107.4(6)
C(10)	- C(9)	- H(91)	133.2(7)	C(9)	- C(10)	- H(101)	117.9(7)
C(11)	- C(10)	- H(101)	120.1(8)	C(10)	- C(11)	- H(111)	129.6(8)
C(12)	- C(11)	- H(111)	110.9(7)	C(7)	- C(12)	- H(121)	113.7(7)
C(11)	- C(12)	- H(121)	124.7(8)				

Table : Hydrogen atoms fractional atomic coordinates for $C_{12}H_{16}O$

H(1)	0.2500	0.3981	0.5000	0.112(6)
H(2)	0.0000	0.5000	0.5000	0.112(6)
H(11)	0.0671	0.1912	0.4913	0.112(6)
H(12)	-0.1009	0.2891	0.4869	0.112(6)
H(21)	0.1647	0.2696	0.4229	0.112(6)
H(31)	-0.1434	0.4147	0.4017	0.112(6)
H(32)	0.0046	0.4124	0.3576	0.112(6)
H(41)	-0.0267	0.1780	0.3519	0.112(6)
H(51)	-0.2822	0.2260	0.4318	0.112(6)
H(61)	-0.1254	0.0334	0.4567	0.112(6)
H(62)	-0.1671	-0.0092	0.3932	0.112(6)
H(63)	-0.3210	0.0002	0.4328	0.112(6)
H(81)	-0.2690	0.0190	0.3123	0.112(6)
H(91)	-0.4371	0.0219	0.2447	0.112(6)
H(101)	-0.6596	0.1896	0.2409	0.112(6)
H(111)	-0.6634	0.3479	0.3133	0.112(6)
H(121)	-0.4850	0.3166	0.3803	0.112(6)

Table : Anisotropic thermal parameters for :

Atom	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	0.069(3)	0.075(3)	0.085(3)	-0.019(3)	-0.017(3)	0.008(3)

Table : Fo/Fc for $C_{12}H_{16}O$ page 1

```
 H /FO/ /FC/ PHI

** K=  0  L=  0 **

 2 1467 1452   0
 4   94  120   0
 6  168  182 180

** K=  2  L=  0 **

 0 1659 1661 180
 1 1213 1206   0
 2 1166 1144 180
 3  179  196   0
 4  135  128 180
 6   82   96   0

** K=  4  L=  0 **

 0  197  183 360
 2  110  132   0
 3  100  107   0
 4  153  193   0

** K=  6  L=  0 **

 0  250  238 180
 1  201  188   0
 2  424  426 180
 5  151  111   0

** K=  8  L=  0 **

 2  181  178   0
 3  310  301 180
 5   80   67 180

** K= 10  L=  0 **

 0   91  111   0
 2   76   79   0
 3   71   86   0

** K=  0  L=  1 **

 2  188  192   0
 4  233  228   0
 6   63   66   0
 8  211  212 180

** K=  1  L=  1 **

 1  429  449 180
 2  905  908   0
 3  368  351   0
 4   91   66 180

** K=  2  L=  1 **

 1  888  914   0
 2  424  435 180
 3   61   18 180
 4  258  250 180
 5   56   41 180
 7   82   79   0
 8   87   77   0

** K=  3  L=  1 **

 0  612  611 180
 1   90   72 180
 2  464  459 180
 3  162  143   0
 4   97   93 180
 7   96  108 180
```

```
 H /FO/ /FC/ PHI

 8   54   64   0

** K=  4  L=  1 **

 1  157  131   0
 2  280  260   0
 3  116  107   0

** K=  5  L=  1 **

 0  315  305 180
 2  247  254   0
 4  167  148   0
 6  153  165   0

** K=  6  L=  1 **

 1  160  152 180
 3   92   58 180
 6   97   91   0

** K=  7  L=  1 **

 0  150  146 180
 1  227  231 180
 2   65   74 180
 4   98  105 180

** K=  8  L=  1 **

 2   93   66   0

** K=  9  L=  1 **

 0  306  290   0
 2   59   70   0
 5   64   63 180

** K= 10  L=  1 **

 1  104  100 180
 3   59   50   0

** K= 12  L=  1 **

 2   59   46 180

** K=  0  L=  2 **

 0  168  199   0
 2   85   78 180
 4  274  255   0

** K=  1  L=  2 **

 1   65   78   0
 2  301  308   0
 3  306  304   0
 4   92  101   0
 5   49   41   0
 6  113  105 180
 7  190  208 180
 8   86   85   0

** K=  2  L=  2 **

 0   52   36   0
 1  197  204   0
 2  269  256 180
 3  137  145   0
 4  273  264 180
 5  194  180   0
 6  116  116 180
```

```
 H /FO/ /FC/ PHI

 7   65   87 180

** K=  3  L=  2 **

 2  284  287 180
 3   82   92   0
 4  160  136   0
 5   95   97   0
 6  116   96   0
 9   67   43   0

** K=  4  L=  2 **

 0   78   94 360
 1   90  101 180
 3  121  116 180
 4  180  169   0
 5  121  124 180
 6  160  167   0
 7   76   77 180

** K=  5  L=  2 **

 2  275  257 180
 3  185  166 180
 4  173  171 180
 5   64   60 180
 6  166  178 180

** K=  6  L=  2 **

 3   53   44 180
 6   48   47   0

** K=  7  L=  2 **

 1   41   41 180
 2   83   86   0
 4  170  170   0
 5  132  108   0
 6   95   90   0

** K=  8  L=  2 **

 0   90  110   0
 2   44   45   0
 3   68   69 180
 5   48   30 180
 6   51   29 180

** K=  9  L=  2 **

 1   76   64   0
 2   72   59 180

** K= 12  L=  2 **

 0   79   68 180

** K=  0  L=  3 **

 2  154  151   0
 4  123  114 180
 6  121  146   0
 8   76   59 180

** K=  1  L=  3 **

 0  988  982   0
 1  648  673   0
 3  426  422   0
 4  227  209 180
 5  127  128   0
```

```
 H /FO/ /FC/ PHI

 7  102   84   0

** K=  2  L=  3 **

 1  182  192 180
 2  639  645   0
 3   77   86   0
 4   78   86   0
 5   40   35 180
 6  127  113   0
 7   80   66   0

** K=  3  L=  3 **

 0  572  591 180
 1  420  434   0
 3  314  310   0
 4  114  122   0
 5   79   68 180

** K=  4  L=  3 **

 1  198  209 180
 2  188  192 180
 4  201  216 180
 5  109   94 180
 6   91  102 180

** K=  5  L=  3 **

 0  142  132 180
 1  131  130 180
 4  129  139   0

** K=  6  L=  3 **

 2  127  147 180
 3   79   75   0
 4   96   88 180
 7   80   62   0

** K=  7  L=  3 **

 0   83  105 180
 1   54   49 180
 3  170  173 180
 5   89   91 180

** K=  8  L=  3 **

 2  108  108   0
 4   87  112   0

** K=  9  L=  3 **

 1   62   62 180
 5   68   63   0

** K= 11  L=  3 **

 0  113  107   0

** K=  0  L=  4 **

 0  472  492 180
 2  499  495   0
 4  234  216 180
 6   71   55 180

** K=  1  L=  4 **

 1 1523 1521   0
 2  504  478 180
```

```
 H /FO/ /FC/ PHI

 3   68   73   0
 4  209  213   0

** K=  2  L=  4 **

 0  652  626   0
 1  167  187 180
 2  229  250   0
 3  472  469   0
 5  146  177   0
 6   73   63 180
 7   81   81   0

** K=  3  L=  4 **

 1  390  408 180
 2  127  133   0
 3  371  380 180
 5   89   85   0

** K=  4  L=  4 **

 0  402  380 180
 1  551  543   0
 2  208  198 180
 3  310  310   0
 4   73   72   0
 5   70   77   0
 6   74   96   0

** K=  5  L=  4 **

 1  296  296   0
 2  104   98 180
 4   94   83 180
 6   80   67 180

** K=  6  L=  4 **

 0  226  211 180
 1  282  292 180
 2  150  152   0
 3  261  275 180
 5  111  132 180
 8   57   54 180

** K=  7  L=  4 **

 1   62   62 180
 2  152  164   0
 3   95   94 180
 4   99   96   0

** K=  8  L=  4 **

 1  123  120 180
 2  127  138 180

** K= 10  L=  4 **

 0  120  141   0
 1   76   77   0

** K=  0  L=  5 **

 2  275  274   0
 4  348  326 180
 6  178  167 180

** K=  1  L=  5 **

 0  196  221   0
 1  813  835   0
```

Table : Fo/Fc for $C_{12}H_{16}O$

H	/FO/	/FC/	PHI
2	393	400	180
3	416	424	0
4	49	42	360
5	336	315	0
6	146	148	180
7	122	120	180
** K= 2 L= 5 **			
1	258	251	0
3	425	398	0
5	54	51	0
6	83	74	0
7	52	54	180
** K= 3 L= 5 **			
0	114	105	180
1	88	66	180
2	200	217	0
3	90	79	180
5	74	74	0
6	99	88	0
8	69	70	180
** K= 4 L= 5 **			
1	380	388	180
2	172	160	180
3	156	157	180
5	62	44	180
7	76	78	0
** K= 5 L= 5 **			
0	403	379	0
1	91	99	180
2	91	92	180
4	97	106	0
** K= 6 L= 5 **			
1	336	341	0
2	187	180	0
3	178	198	0
** K= 7 L= 5 **			
1	97	110	0
2	126	110	180
3	239	227	180
** K= 8 L= 5 **			
1	220	238	180
2	113	113	180
3	58	80	180
** K= 9 L= 5 **			
2	94	106	0
3	46	46	0
4	65	84	180
** K= 0 L= 6 **			
0	75	51	180
2	449	462	0
4	459	425	180
6	143	151	0
8	104	99	0
** K= 1 L= 6 **			
1	502	487	180
2	115	108	0
3	109	88	0
5	279	244	0
7	215	208	0
** K= 2 L= 6 **			
0	291	280	180
1	279	288	0
2	120	108	180
3	567	548	0
4	79	97	180
5	198	184	180
6	143	145	180
** K= 3 L= 6 **			
1	405	429	180
2	90	83	0
3	223	210	180
5	202	201	180
7	114	118	180
8	65	61	0
** K= 4 L= 6 **			
0	209	219	180
1	43	23	0
2	220	214	0
3	42	53	180
4	246	238	0
5	63	69	0
6	71	71	0
** K= 5 L= 6 **			
1	55	58	0
2	98	108	180
3	185	172	0
5	138	121	0
6	60	38	0
** K= 6 L= 6 **			
0	273	266	180
1	181	186	180
3	65	55	180
5	127	112	180
** K= 7 L= 6 **			
1	198	181	0
2	109	129	180
4	82	65	180
5	68	56	180
** K= 8 L= 6 **			
1	117	113	180
3	88	76	0
4	53	51	0
7	63	57	0
** K= 9 L= 6 **			
2	103	108	0
** K= 0 L= 7 **			
2	425	443	180
4	77	85	0
6	91	88	180
** K= 1 L= 7 **			
0	989	1012	0
1	189	190	180
2	103	86	0
3	474	439	180
4	106	109	0
** K= 2 L= 7 **			
1	428	428	180
3	299	295	180
4	212	192	0
5	98	102	0
6	72	80	0
7	88	75	0
** K= 3 L= 7 **			
0	90	106	360
1	576	591	0
2	270	280	180
3	518	511	0
4	109	111	180
5	178	174	0
** K= 4 L= 7 **			
2	58	58	0
3	146	157	180
** K= 5 L= 7 **			
0	379	373	180
1	105	103	0
2	196	186	180
3	126	121	180
4	173	191	0
5	96	120	180
** K= 6 L= 7 **			
1	166	163	0
3	118	102	0
5	68	52	0
** K= 7 L= 7 **			
3	164	187	180
5	75	68	0
** K= 8 L= 7 **			
1	42	22	180
** K= 9 L= 7 **			
0	93	91	0
2	54	54	0
6	65	48	0
** K= 0 L= 8 **			
0	841	867	0
2	844	859	0
4	88	101	0
6	118	138	180
** K= 1 L= 8 **			
1	248	243	180
2	172	173	0
3	169	155	0
4	139	131	180
8	67	57	180
** K= 2 L= 8 **			
0	466	454	180
1	103	86	180
2	91	96	0
4	88	63	180
** K= 3 L= 8 **			
1	131	141	0
2	287	265	180
3	132	141	0
** K= 4 L= 8 **			
0	443	418	180
1	136	117	180
2	74	59	180
3	233	239	0
5	136	132	0
6	107	112	0
** K= 5 L= 8 **			
2	255	240	0
3	79	72	180
** K= 6 L= 8 **			
0	55	51	360
1	138	128	180
2	143	133	180
5	49	43	180
** K= 7 L= 8 **			
3	122	125	0
4	53	42	180
** K= 8 L= 8 **			
0	91	75	0
2	85	90	0
** K= 9 L= 8 **			
1	81	92	180
** K= 0 L= 9 **			
4	156	150	0
** K= 1 L= 9 **			
0	309	316	0
1	73	76	180
3	98	92	0
4	173	169	180
5	187	187	180
** K= 2 L= 9 **			
1	82	64	360
5	55	57	0
6	99	114	180
8	58	42	0
** K= 3 L= 9 **			
0	148	152	0
1	197	205	0
2	146	144	180
3	96	114	0
4	111	116	0
6	97	91	180
7	66	94	180
** K= 4 L= 9 **			
1	131	118	180
2	118	124	180
3	190	230	0
5	84	86	0
7	85	87	180
** K= 5 L= 9 **			
0	445	396	180
2	65	72	180
3	192	178	180
4	91	81	0
6	163	148	0
** K= 6 L= 9 **			
2	121	136	180
3	113	115	180
4	126	116	0
** K= 7 L= 9 **			
0	166	165	0
2	172	160	0
3	107	114	0
6	64	43	180
** K= 8 L= 9 **			
1	102	84	0
3	86	86	180
** K= 10 L= 9 **			
1	50	52	180
** K= 0 L= 10 **			
0	754	769	0
2	143	145	180
4	47	32	180
6	214	247	180
** K= 1 L= 10 **			
2	76	67	0
4	121	98	0
6	143	135	0
** K= 2 L= 10 **			
1	313	317	0
4	61	44	180
5	117	95	0
6	128	138	0
** K= 3 L= 10 **			
1	196	197	180
3	108	109	180
** K= 4 L= 10 **			
0	230	240	180

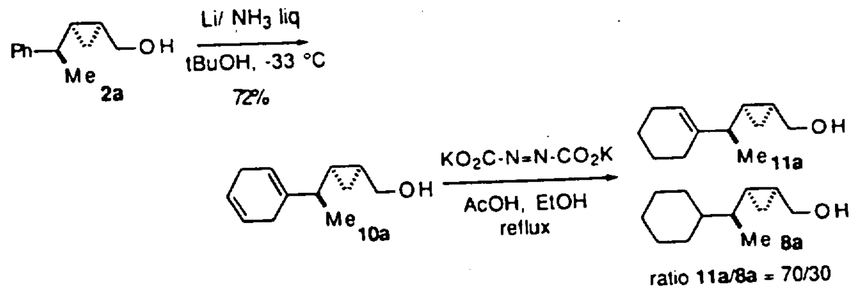
Table : Fo/Fc for $C_{12}H_{16}O$

H	/FO/	/FC/	PHI
2	63	68	180
3	76	78	0
5	215	209	180
** K= 5 L= 10 **			
1	98	107	0
2	104	111	180
4	206	219	180
** K= 6 L= 10 **			
0	131	129	180
2	67	52	180
4	251	261	0
5	106	103	0
** K= 7 L= 10 **			
1	72	82	180
2	111	106	0
3	93	76	0
** K= 8 L= 10 **			
0	88	77	0
3	145	172	180
4	104	102	180
** K= 10 L= 10 **			
0	53	35	0
3	66	81	0
** K= 0 L= 11 **			
2	526	551	0
4	366	384	0
** K= 1 L= 11 **			
0	201	212	0
1	108	99	0
2	458	431	0
3	451	425	0
4	119	111	180
** K= 2 L= 11 **			
1	54	58	180
2	349	333	180
3	65	92	0
4	108	85	180
5	100	101	0
** K= 3 L= 11 **			
0	466	469	180
1	126	139	180
2	165	181	180
4	156	170	0
5	67	65	0
** K= 4 L= 11 **			
1	116	126	0
2	43	29	360
3	176	208	180
5	56	52	180
7	56	46	180
** K= 5 L= 11 **			
4	156	152	180
5	126	133	0
** K= 6 L= 11 **			
1	72	72	180
4	118	121	180
5	77	84	0
** K= 7 L= 11 **			
0	102	92	180
1	87	109	180
5	129	166	180
** K= 8 L= 11 **			
2	67	62	0
4	96	91	0
5	61	50	180
** K= 9 L= 11 **			
0	84	98	0
** K= 0 L= 12 **			
0	466	470	0
2	84	103	180
4	66	59	0
** K= 1 L= 12 **			
1	525	491	0
2	187	182	0
3	233	229	0
4	66	59	0
5	141	131	0
** K= 2 L= 12 **			
0	104	130	0
1	219	218	0
2	84	80	180
3	160	130	0
5	114	111	0
** K= 3 L= 12 **			
1	232	214	180
2	67	64	180
4	213	211	180
7	49	52	180
** K= 4 L= 12 **			
4	138	130	0
6	79	77	0
** K= 5 L= 12 **			
2	96	102	180
3	158	181	0
4	115	116	0
** K= 6 L= 12 **			
3	107	133	180
** K= 7 L= 12 **			
3	148	167	180
** K= 9 L= 12 **			
2	86	80	0
** K= 0 L= 13 **			
2	185	195	0
** K= 1 L= 13 **			
0	107	86	180
1	160	148	0
2	71	45	180
3	256	228	0
4	133	132	180
5	193	181	0
** K= 2 L= 13 **			
3	92	85	0
4	55	51	0
** K= 3 L= 13 **			
0	74	59	360
1	91	87	0
2	144	134	0
3	73	82	0
4	108	126	180
5	121	131	180
** K= 4 L= 13 **			
1	50	53	180
2	95	82	180
3	190	174	180
4	86	78	180
5	57	37	180
** K= 5 L= 13 **			
0	84	75	180
2	129	162	0
4	199	219	0
5	109	97	180
** K= 6 L= 13 **			
1	82	119	0
2	80	71	0
3	89	93	0
** K= 7 L= 13 **			
0	141	151	180
1	107	114	180
2	91	79	180
** K= 8 L= 13 **			
1	54	78	180
5	56	40	180
** K= 9 L= 13 **			
0	98	120	0
** K= 0 L= 14 **			
0	191	199	0
2	182	159	0
** K= 1 L= 14 **			
1	94	84	180
2	55	59	180
4	118	101	0
5	148	106	0
** K= 2 L= 14 **			
0	63	38	180
1	71	69	0
2	149	154	180
3	240	229	0
6	109	115	180
** K= 3 L= 14 **			
1	199	184	180
3	203	194	180
4	77	78	0
** K= 4 L= 14 **			
0	92	85	0
1	68	56	0
4	141	158	0
5	136	139	0
** K= 5 L= 14 **			
1	109	111	0
2	75	88	180
4	117	128	180
6	97	86	180
** K= 6 L= 14 **			
0	47	9	180
3	126	107	180
4	138	142	180
6	58	48	0
** K= 0 L= 15 **			
2	219	222	180
4	255	277	180
8	83	72	180
** K= 1 L= 15 **			
0	201	190	0
1	114	129	0
2	126	113	0
5	130	123	0
6	78	79	180
** K= 2 L= 15 **			
2	365	356	0
4	108	115	0
6	102	84	0
** K= 3 L= 15 **			
0	241	248	180
3	133	124	0
4	86	69	180
6	109	104	0
** K= 4 L= 15 **			
1	246	270	180
2	131	152	180
3	92	101	0
4	133	141	180
** K= 5 L= 15 **			
1	71	66	180
2	61	60	180
3	96	92	0
** K= 6 L= 15 **			
1	221	242	0
3	80	74	0
4	80	110	0
5	84	69	180
6	67	78	0
** K= 7 L= 15 **			
2	68	70	180
3	116	136	180
** K= 8 L= 15 **			
1	77	70	180
** K= 0 L= 16 **			
0	117	127	0
4	200	236	180
** K= 1 L= 16 **			
2	47	31	0
3	152	135	0
6	87	94	180
** K= 2 L= 16 **			
0	103	107	0
3	135	148	0
5	71	62	180
6	68	69	0
** K= 3 L= 16 **			
1	63	54	180
2	136	138	180
3	48	55	180
** K= 4 L= 16 **			
0	73	43	180
1	121	123	0
** K= 5 L= 16 **			
1	52	81	0
** K= 6 L= 16 **			
0	176	192	180
2	82	71	0
4	84	85	0
6	51	48	0
** K= 7 L= 16 **			
1	79	63	0
** K= 8 L= 16 **			
1	90	109	180
3	55	51	180
** K= 9 L= 16 **			

Table : Fo/Fc for $C_{12}H_{16}O$

H	/FO/	/FC/	PHI
1	76	80	180
** K= 0 L= 17 **			
2	59	64	180
** K= 1 L= 17 **			
0	82	91	0
1	84	77	0
2	75	72	180
5	98	101	180
** K= 2 L= 17 **			
3	82	71	180
5	76	61	0
6	105	98	0
** K= 3 L= 17 **			
2	87	84	0
4	104	87	0
** K= 4 L= 17 **			
3	71	71	180
4	93	81	180
5	82	84	180
** K= 5 L= 17 **			
0	95	111	180
1	66	58	180
2	134	154	180
3	68	65	180
** K= 6 L= 17 **			
1	104	111	0
3	105	96	0
** K= 7 L= 17 **			
0	189	184	0
1	75	95	0
2	83	110	0
** K= 9 L= 17 **			
0	74	101	180
** K= 0 L= 18 **			
6	136	133	180
** K= 1 L= 18 **			
1	98	107	0
2	86	64	0
3	99	96	0
** K= 2 L= 18 **			
0	89	88	0
2	168	177	0
6	57	52	0
** K= 3 L= 18 **			
1	283	263	180
3	96	87	180
** K= 4 L= 18 **			
0	216	223	180
2	109	118	180
3	134	153	0
4	98	87	180
** K= 5 L= 18 **			
1	114	90	0
2	123	152	180
3	84	75	0
** K= 9 L= 18 **			
1	45	29	0
** K= 0 L= 19 **			
2	203	220	0
4	110	101	0
** K= 1 L= 19 **			
0	190	185	0
1	78	93	180
5	134	133	180
** K= 2 L= 19 **			
2	129	90	180
3	134	129	0
5	71	62	0
** K= 3 L= 19 **			
0	124	128	0
1	156	173	0
2	90	89	180
3	119	126	0
4	84	86	0
** K= 5 L= 19 **			
0	251	234	180
2	80	78	180
3	74	75	180
** K= 7 L= 19 **			
2	75	69	0
** K= 0 L= 20 **			
4	50	67	180
** K= 1 L= 20 **			
1	221	211	0
** K= 2 L= 20 **			
1	112	107	0
2	137	142	0
** K= 3 L= 20 **			
4	98	68	180
** K= 4 L= 20 **			
0	115	117	180
1	120	113	180
3	68	72	0
** K= 6 L= 20 **			
0	80	75	0
** K= 7 L= 20 **			
2	68	63	0
** K= 0 L= 21 **			
4	99	94	0
6	65	44	0
** K= 1 L= 21 **			
0	102	97	180
3	96	87	0
** K= 2 L= 21 **			
1	106	87	180
** K= 3 L= 21 **			
0	177	170	0
** K= 4 L= 21 **			
2	70	60	180
** K= 6 L= 21 **			
2	64	57	180
** K= 7 L= 21 **			
0	55	17	180
** K= 8 L= 21 **			
1	57	63	0
** K= 0 L= 22 **			
0	59	36	0
4	72	80	180
** K= 1 L= 22 **			
1	227	235	180
2	106	115	0
** K= 2 L= 22 **			
0	113	97	0
2	82	75	0
** K= 3 L= 22 **			
2	84	83	180
** K= 4 L= 22 **			
0	94	98	180
1	44	39	0
** K= 6 L= 22 **			
3	68	70	180
** K= 8 L= 22 **			
0	66	50	180
** K= 3 L= 23 **			
0	62	35	180
** K= 0 L= 24 **			
0	238	228	180
** K= 1 L= 24 **			
1	78	84	180
** K= 2 L= 24 **			
0	124	131	0
1	80	70	180
** K= 4 L= 24 **			
3	55	57	180
** K= 2 L= 25 **			
1	151	121	180
** K= 3 L= 25 **			
2	60	66	0
** K= 4 L= 25 **			
2	62	52	180
** K= 6 L= 25 **			
1	66	45	0
** K= 0 L= 26 **			
0	206	212	0
2	82	103	0
** K= 2 L= 26 **			
2	60	77	180
** K= 0 L= 30 **			
0	80	80	180

The relative stereochemistry of **8a** was assigned by comparaison with an authentic sample prepared from **2a** by performing a Birch reduction followed by reduction of the 1,4-diene:



The relative stereochemistry of **9a** and **9b** was assigned by converting them to the corresponding diastereoisomeric carboxylic acids **12a** and **12b** and comparison with the material obtained from **2a** by oxidation of the aromatic ring after acetylation.