Supporting Information

Chelation-Controlled Reduction: Stereoselective Formation of *syn*-1,3-Diol and Synthesis of Compactin and Mevinolin Lactone

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Experimental Section

Anhydrous solvents and reagents were obtained as follows: tetrahydrofuran and diethyl ether, distillation from sodium and benzophenone; methylene chloride, distillation from CaH_2 ; diisopropylethylamine and triethylamine, distillation from CaH_2 . All other solvents were HPLC grade. Analytical HPLC analysis was performed on a reverse phase column (4.6 mm x 25 cm) with 60% $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ as the solvent, flow rate 1.0 mL/min, λ 185 nm). Column chromatography was performed with 240-400 mesh silica gel under low pressure of 5-10 psi. Thin-layer chromatography (TLC) was carried out with silica gel plates.

Yields for nitro aldol reaction and subsequent dehydration reaction as well as ^1H -NMR and ^{13}C -NMR data for the major nitroalkene derivative.

4a: Nitro aldol reaction of **1a** (635.7 mg, 4.88 mmol) and **2a** (954 mg, 4.88 mmol) provided the corresponding aldol product (1.59g, 97%) as a mixture. Subsequent dehydration of the aldol product under specified conditions afforded **4a** (1.32g, 91%). ^1H -NMR (300 MHz, CDCl_3) δ 1.37 (s, 3H), 1.44 (s, 3H), 2.89 (dd, 1H, $J=5.1, 8.7\text{Hz}$), 2.96 (dt, 1H, $J=15.0, 4.2\text{Hz}$), 3.52-3.66 (m, 3H), 3.98 (dd, 1H, $J=6.6, 8.7\text{Hz}$), 4.49 (s, 2H), 4.78-4.83 (m, 1H), 7.12 (d, 1H, $J=8.7\text{Hz}$), 7.25-7.34 (m, 5H); ^{13}C -NMR (75 MHz, CDCl_3) δ 25.6, 26.5, 27.8, 67.0, 68.9, 71.9, 73.3, 110.6, 127.8, 127.9, 128.4, 135.4, 137.5, 150.0.

4b: Nitro aldol reaction of **1a** (48 mg, 0.37 mmol) and **2b** (101 mg, 0.37 mmol) provided the corresponding aldol product (132 mg, 89%) as a mixture. Subsequent dehydration of the aldol product under specified conditions afforded **4b** (115.5 mg, 70%). ^1H -NMR (500 MHz, CDCl_3) δ 1.40 (s, 3H), 1.47 (s, 3H), 2.96 (ddd, 1H, $J=5.24, 8.35, 14.8\text{Hz}$), 3.05 (td, 1H, $J=5.13, 14.8\text{Hz}$), 3.57-3.61 (m, 2H), 3.65-3.70 (m, 1H), 3.91 (dd, 1H, $J=6.4, 8.6\text{Hz}$), 4.84-4.89 (m, 1H), 5.32 (s, 1H), 7.14 (d, 1H, $J=8.6\text{Hz}$), 7.26-7.36 (m, 10H); ^{13}C -NMR (125 MHz, CDCl_3) δ

26.0, 27.0, 28.3, 66.4, 69.1, 72.2, 84.6, 110.9, 127.3, 128.1, 128.8, 135.3, 141.9, 150.7.

4e: Nitro aldol reaction of **1b** (115.3 mg, 0.46 mmol) and **2c** (120.6 mg, 0.46 mmol) provided the corresponding aldol product (228 mg, 97%) as a mixture. Subsequent dehydration of the aldol product under specified conditions afforded **4e** (202 mg, 92%). ¹H-NMR (300 MHz, CDCl₃) δ 1.01-1.04 (m, 21H), 1.44 (s, 3H), 1.45 (s, 3H), 2.85 (t, 2H, J=6.3Hz), 3.64 (d, 2H, J=4.5Hz), 3.76-3.83 (m, 2H), 4.00-4.06 (m, 1H), 4.58 (ABq, 2H, J=12.3, Δv =19.8Hz), 4.72 (t, 1H, 1H, J=8.4Hz), 7.06 (d, 1H, J=8.4Hz), 7.28-7.34 (m, 5H); ¹³C-NMR (75 MHz, CDCl₃) δ 11.7, 17.8, 26.7, 30.3, 60.9, 68.5, 73.6, 73.9, 79.6, 110.5, 127.6, 127.7, 128.3, 133.4, 137.4, 151.5.

4f: Nitro aldol reaction of **1a** (48 mg, 0.37 mmol) and **2d** (94 mg, 0.37 mmol) provided the corresponding aldol product (132 mg, 95%) as a mixture. Subsequent dehydration of the aldol product under specified conditions afforded **4f** (101 mg, 80%). ¹H-NMR (400 MHz, CDCl₃) δ 1.05-1.07 (m, 21H), 1.41 (s, 3H), 1.47 (s, 3H), 3.79 (dd, 1H, J=6.8, 8.6Hz), 4.22 (dd, 1H, J=6.5, 8.6Hz), 4.69 (d, 1H, J=13.4Hz), 4.86 (d, 1H, J=13.4Hz), 5.03-5.08 (m, 1H), 7.10 (d, 1H, J=8.2), ¹³C

NMR (100 MHz, CDCl₃) δ 12.2, 18.2, 26.0, 26.9, 57.8, 69.5, 71.6, 111.1, 136.2, 151.8.

4g: Nitro aldol reaction of **1c** (183 mg, 0.95 mmol) and **2c** (249 mg, 0.95 mmol) provided the corresponding aldol product (406 mg, 94%) as a mixture. Subsequent dehydration of the aldol product under specified conditions afforded **4g** (339 mg, 87%). ¹H-NMR (300 MHz, CDCl₃) δ 1.02-1.05 (m, 21H), 1.65 (dd, 1H, J=1.2, 13.5Hz), 2.09 (ddd, 1H, J=4.8, 12.0, 25.5Hz), 2.94 (t, 2H, J=5.7Hz), 3.89 (dt, 2H, J=2.7, 6.0), 4.03 (dt, 1H, J=2.1, 11.7), 4.32 (dd, 1H, J=4.8, 11.4Hz), 4.78-4.84 (m, 1H), 5.57 (s, 1H), 7.18 (d, 1H, J=7.5Hz), 7.34-7.39(m, 3H), 7.46-7.50 (m, 2H); ¹³C-NMR (75 MHz, CDCl₃) δ 11.7, 17.8, 30.2, 30.8, 60.8, 66.3, 73.4, 101.1, 125.9, 128.2, 128.9, 135.3, 137.7, 149.9.

4h: Nitro aldol reaction of **1d** (78 mg, 0.67 mmol) and **2c** (176 mg, 0.67 mmol) provided the corresponding aldol product (236 mg, 90%) as a mixture. Subsequent dehydration of the aldol product under specified conditions afforded **4h** (213 mg, 94%). ¹H-NMR (400 MHz, CDCl₃) δ 1.00-1.05 (m, 21H), 1.32 (d, 3H, J=5.1Hz), 1.50 (dd, 1H, J=0.9, 13.4Hz), 1.90 (m, 1H), 2.89 (t, 2H, J=6.1Hz), 3.76-3.88 (m, 3H), 4.13 (dd, 1H, J=4.4, 11.7Hz), 4.57(ddd, 1H, J=2.6, 7.9, 11.0Hz), 4.74 (q, 1H,

J=5.1Hz), 7.08 (d, 1H, J=7.9Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 11.7, 17.8, 20.9, 30.0, 30.7, 60.7, 65.7, 72.8, 98.7, 135.4, 149.7.

4i: Nitro aldol reaction of **1e** (85 mg, 0.65 mmol) and **2c** (172 mg, 0.65 mmol) provided the corresponding aldol product (231 mg, 94%) as a mixture. Subsequent dehydration of the aldol product under specified conditions afforded **4i** (184 mg, 84%). ^1H NMR (300 MHz, CDCl_3) δ 0.95-1.05 (m, 21H), 1.57(m, 1H), 1.92-2.04 (m, 1H), 2.88 (t, 2H, J=6.0Hz), 3.73 (dt, 1H, J=2.4, 11.7Hz), 3.83 (dt, 2H, J=2.4, 6.0Hz), 4.12 (dd, 1H, J=4.5, 11.7Hz), 4.49-4.56 (m, 1H), 4.72 (d, 1H, J=6.3Hz), 5.05 (d, 1H, J=6.3Hz), 7.07 (d, 1H, J=7.5Hz); ^{13}C -NMR (100 MHz, CDCl_3) δ 11.7, 17.8, 30.7, 30.9, 60.8, 65.8, 72.8, 93.4, 135.2, 149.9.

Yields (2-steps) and Characterization data for Ketones **5a-c** and **5e-i**

5a (0.60 g, 84%) was prepared from **4a** (0.79g, 2.57 mmol). $[\alpha]_{\text{D}}^{23} +2.72$ (c 3.32, CHCl_3); IR (film) 2985, 2934, 2868, 1713, 1454, 1369, 1213, 1098 cm^{-1} ; ^1H -NMR (400 MHz, CDCl_3) δ 1.34, (s, 3H), 1.39 (s, 3H), 2.62 (dd, 1H, J=7.1, 17.0Hz), 2.73 (t, 2H, J=6.2Hz), 2.93 (dd, 1H, J=5.9, 17.0Hz), 3.52 (dd, 1H, J=6.6, 8.3Hz), 3.73 (t, 2H, J=6.3Hz), 4.18 (dd, 1H, J=6.0, 8.3Hz), 4.43-4.48 (m, 1H), 4.50 (s, 2H), 7.29-7.34 (m, 5H); ^{13}C -NMR (100 MHz, CDCl_3) δ 25.4, 26.8, 43.5, 47.5, 65.0, 69.4,

71.5, 73.2, 108.8, 127.7, 128.4, 137.9, 206.9; HRMS (EI) m/z calcd for $C_{16}H_{22}O_4Na$ ($M^+ + Na$) 301.1416, found 301.1412.

5b (50 mg, 83%) was prepared from **4b** (65 mg, 0.17 mmol). $[\alpha]_D^{23} +5.36$ (c 1.12, $CHCl_3$); IR (film) 2984, 2934, 2868, 1713, 1373, 1094, 1073 cm^{-1} ; 1H -NMR (500 MHz, $CDCl_3$) δ 1.37 (s, 3H), 1.42 (s, 3H), 2.66 (dd, 1H, $J=5.3, 17.0Hz$), 2.79 (t, 2H, $J=6.2Hz$), 2.97 (dd, 1H, $J=6.0, 17.0Hz$), 3.54 (dd, 1H, $J=6.7, 8.2Hz$), 3.75 (t, 2H, $J=6.2Hz$), 4.20 (dd, 1H, $J=6.0, 8.2Hz$), 4.47-4.51 (m, 1H), 5.38 (s, 1H), 7.26-7.34 (m, 10H), ^{13}C -NMR (125 MHz, $CDCl_3$) δ 25.9, 27.2, 44.0, 48.0, 64.6, 69.8, 71.9, 84.5, 109.2, 127.3, 127.9, 128.8, 142.3, 207.4; HRMS (EI) m/z calcd for $C_{22}H_{26}O_4Na$ ($M^+ + Na$) 377.1729, found 377.1730.

5c: $[\alpha]_D^{23} +8.10$ (c 0.72, $CHCl_3$); IR (film) 2984, 2932, 1713, 1448, 1071 cm^{-1} ; 1H -NMR (400 MHz, $CDCl_3$) δ 1.33 (s, 3H), 1.37 (s, 3H), 2.60 (dd, 1H, $J=7.3, 17.0$), 2.68 (t, 2H, $J=6.4Hz$), 2.90 (dd, 1H, $J=5.9, 17.0Hz$), 3.36-3.43 (m, 2H), 3.50 (dd, 1H, $J=6.6, 8.2Hz$), 4.17 (dd, 1H, $J=5.9, 8.2Hz$), 4.42-4.48 (m, 1H), 7.23-7.42 (m, 15H); ^{13}C -NMR (100 MHz, $CDCl_3$) δ 25.8, 27.2, 44.2, 47.7, 59.5, 69.8, 71.9, 87.2, 109.1, 127.4, 128.3, 128.9, 144.2, 207.6; HRMS (EI) m/z calcd for $C_{28}H_{30}O_4Na$ ($M^+ + Na$) 453.2042, found 453.2050.

5e (63 mg, 85%) was prepared from **4e** (81 mg, 0.16 mmol). $[\alpha]_D^{23} +7.65$ (*c* 1.75, CHCl₃), IR (film) 2926, 2865, 1718, 1096 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 1.01-1.06 (m, 21H), 1.39 (s, 6H), 2.67 (t, 2H, J=6.4Hz), 2.71 (dd, 1H, J=4.2, 16.5Hz), 2.82 (dd, 1H, J=7.9, 16.5Hz), 3.56 (dd, 1H, J=4.8, 10.1Hz), 3.62 (dd, 1H, J=5.7, 10.1Hz), 3.83-3.88 (m, 1H), 3.96 (t, 2H, J=6.3Hz), 4.27 (dt, 1H, J=4.2, 7.8Hz), 4.56 (ABq, 2H, J=12.1, $\Delta\nu$ =14.6Hz), 7.32-7.34 (m, 5H); ¹³C-NMR (100 MHz, CDCl₃) δ 1.8, 17.9, 26.9, 27.1, 46.6, 47.1, 59.0, 70.3, 73.5, 74.2, 79.6, 109.2, 127.6, 128.3, 137.8, 207.2; HRMS (EI) *m/z* calc'd for C₂₄H₄₄O₅NaSi (M⁺+Na) 487.2856, found 487.2832.

5f (42 mg, 63%) was obtained from **4f** (73 mg, 0.20 mmol). $[\alpha]_D^{23} +12.5$ (*c* 0.67, CHCl₃); IR (film) 2943, 2867, 1719, 1463, 1113, 1060 cm⁻¹; ¹H- NMR (400 MHz, CDCl₃) δ 1.03-1.09 (m, 21H), 1.35 (s, 3H), 1.40 (s, 3H), 2.73 (dd, 1H, J=7.3, 17.5Hz), 3.09 (dd, 1H, J=6.0, 17.5Hz), 3.54 (t, 1H, J=6.8Hz), 4.20 (dd, 1H, J=6.0, 8.3 Hz), 4.23 (s, 2H), 4.48-4.51 (m, 1H); ¹³C-NMR (75 MHz, CDCl₃) δ 11.8, 17.8, 25.4, 26.8, 43.1, 69.5, 69.9, 71.5, 108.8, 209.4; HRMS (EI) *m/z* calc'd for C₁₇H₃₄O₄NaSi (M⁺+Na) 353.2124, found 353.2122.

5g (246 mg, 90%) was obtained from **4g** (291 mg, 0.67 mmol). $[\alpha]_D^{23} +16.1$ (*c* 6.04, CHCl₃); ¹H-NMR (300 MHz, CDCl₃) δ 1.02-1.06 (m, 21H), 1.61-1.66 (m,

1H), 1.80 (ddd, 1H, J=4.8, 11.7, 23.4Hz), 2.60-2.69 (m, 3H), 2.93 (dd, 1H, J=6.6, 16.8Hz), 3.97 (t, 2H, J=6.0Hz), 4.01 (dd, 1H, J=2.7, 12.0Hz), 4.25 (dd, 1H, J=4.8, 11.4Hz), 4.37 (m, 1H), 5.53 (s, 1H), 7.32-7.35 (m, 3H), 7.43-7.47 (m, 2H); ¹³C-NMR (75 MHz, CDCl₃) δ 11.7, 17.8, 31.1, 46.7, 49.5, 58.9, 66.7, 73.1, 101.0, 125.9, 128.1, 128.6, 138.4, 207.6; HRMS (EI) *m/z* calc'd for C₂₃H₃₈O₄NaSi (M⁺+Na) 429.2437, found 429.2419.

5h (47 mg, 91%) was obtained from **4h** (55 mg, 0.15 mmol). [α]_D²³ +4.11 (*c* 1.17, CHCl₃); IR (film) 2941, 2865, 1715, 1463, 1374, 1122, 1100 cm⁻¹; ¹H-NMR (300 MHz, CDCl₃) δ 1.02-1.06 (m, 21H), 1.27 (d, 3H, J=5.1Hz), 1.48-1.66 (m, 2H), 2.54 (dd, 1H, J=5.7, 16.8Hz), 2.65 (t, 2H, J=6.6Hz), 2.81 (dd, 1H, J=6.9, 16.8Hz), 3.76 (dt, 1H, J=2.7, 11.7Hz), 3.96 (t, 2H, J=6.6Hz), 4.03-4.15 (m, 2H), 4.69 (q, 1H, J=5.1Hz); ¹³C-NMR (75 MHz, CDCl₃) δ 11.8, 17.9, 21.1, 30.9, 46.8, 49.6, 59.0, 66.3, 72.4, 99.0, 207.6; HRMS (EI) *m/z* calc'd for C₁₈H₃₆O₄NaSi (M⁺+Na) 367.2281, found 367.2273.

5i (85 mg, 84%) was obtained from **4i** (101 mg, 0.28 mmol). [α]_D²³ +0.64 (*c* 2.33, CHCl₃); IR (film) 2942, 2865, 1714, 1463, 1108, 1036 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 0.98-1.06 (m, 21H), 1.54 (d, 1H, J=12.6Hz), 1.74 (ddd, 1H, J=4.9, 12.8, 24.4Hz), 2.53 (dd, 1H, J=5.6, 16.6Hz), 2.64 (dt, 2H, J=1.6, 6.1Hz), 2.81(dd, 1H,

J=6.9, 16.6Hz), 3.72 (dt, 1H, J=2.54, 11.9Hz), 3.95 (t, 2H, J=6.1Hz), 4.04-4.11 (m, 2H), 4.70 (d, 1H, J=6.4Hz), 4.99 (d, 1H, J=6.4Hz); ^{13}C -NMR (100 MHz, CDCl_3) δ 11.8, 17.9, 31.7, 46.7, 49.5, 59.0, 66.4, 72.4, 93.8, 207.5; HRMS (EI) m/z calcd for $\text{C}_{17}\text{H}_{34}\text{O}_4\text{NaSi}$ ($\text{M}^+ + \text{Na}$) 353.2124, found 353.2108.

Characterization data for the major syn-1,3-diol **6a-c** and **6e-i**

6a: IR (film) 3467, 2924, 2866, 1369, 1092 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.36 (s, 3H), 1.42 (s, 3H), 1.68-1.81 (m, 4H), 3.56 (dt, 1H, J=3.3, 8.1 Hz), 3.63-3.73 (m, 2H), 4.00-4.05 (m, 1H), 4.10 (ddd, 1H, J=1.8, 6.0, 8.1Hz), 4.24-4.38 (m, 1H), 4.52 (s, 2H), 7.30-7.34 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ 25.7, 26.8, 36.8, 40.5, 68.1, 69.1, 69.6, 73.2, 75.0, 109.1, 127.6, 128.3, 128.5, 138.9; MS (EI) m/z 91.1, 225.2, 303.2 ($\text{M}^+ + \text{Na}$).

6b: IR (film) 3512, 2926, 2869, 1091, 1074 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.36 (s, 3H), 1.41 (s, 3H), 1.65-1.87 (m, 4H), 3.42 (br, 1H), 3.55 (t, 1H, J=7.6 Hz), 3.59-3.68 (m, 2H), 4.00-4.05 (m, 1H), 4.08 (dd, 1H, J=5.9, 8.1 Hz), 4.24-4.31 (m, 1H); 5.35 (s, 1H), 7.31-7.32 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 25.7, 26.9, 36.9, 40.5, 67.0, 69.2, 69.6, 75.0, 84.1, 109.1, 126.8, 126.9, 127.4, 127.5, 128.4, 128.4, 141.9, 142.0; MS (EI) m/z 167.3, 379.3 ($\text{M}^+ + \text{Na}$).

6c: IR (film) 3447, 2931, 2881, 1447, 1067 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.35 (s, 3H), 1.40 (s, 3H), 1.59-1.74 (m, 3H), 1.81-1.88 (m, 1H), 3.18-3.22 (m, 1H), 3.32-3.36 (m, 2H), 3.52 (t, 1H, $J=7.6\text{Hz}$), 3.96-3.99 (m, 1H), 4.07(dd, 1H, $J=5.9, 8.0\text{ Hz}$), 4.22-4.27 (m, 1H), 7.23-7.43 (m, 15H); ^{13}C NMR (125 MHz, C_6D_6) δ 26.1, 27.2, 38.1, 41.1, 61.8, 69.0, 70.0, 75.7, 87.1, 109.3, 127.4, 128.4, 129.3, 145.0; MS (EI) m/z 243.3, 455.2 ($\text{M}^+\text{+Na}$).

6e: IR (film) 3452, 2941, 2866, 1462, 1379, 1249, 1095 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.03-1.09 (m, 21H), 1.40 (s, 3H), 1.41 (s, 3H), 1.64-1.78 (m, 4H), 3.59 (d, 2H, $J=4.9\text{ Hz}$), 3.64 (br, 1H), 3.83-3.92 (m, 3H), 3.94-4.03 (m, 2H), 4.59 (ABq, 2H, $J=12.2\text{ Hz}$, $\Delta\nu=15.2\text{Hz}$), 7.28-7.33 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ 12.2, 18.3, 27.4, 27.6, 39.4, 40.8, 62.4, 69.7, 70.7, 73.9, 77.4, 80.7, 109.5, 128.1, 128.8, 138.8; MS (EI) m/z 489.3 ($\text{M}^+\text{+Na}$).

6f: IR (film) 3482, 2924, 2866, 1462, 1378, 1116, 1066 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.04-1.10 (m, 21H), 1.36 (s, 3H), 1.42 (s, 3H), 1.70-1.82 (m, 2H), 3.58 (t, 1H, $J=7.8\text{ Hz}$), 3.64 (d, 2H, $J=5.7\text{ Hz}$), 3.79-3.83 (m, 1H), 4.11 (dd, 1H, $J=5.9, 7.8\text{ Hz}$), 4.27-4.32 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 12.4, 18.3, 26.0, 27.3, 37.4, 68.0, 69.9, 71.1, 75.0, 109.4; MS (EI) m/z 268.1, 355.2 ($\text{M}^+\text{+Na}$).

6g: IR (film) 3504, 2942, 2865, 1105 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.05-1.10 (m, 21H), 1.63 (dd, 1H, $J=1.3, 13.3$), 1.67-1.71 (m, 2H), 1.75-1.97 (m, 3H), 3.75 (br, 1H), 3.87-3.92 (m, 1H), 3.95 (dd, 1H, $J=5.1, 10.2$ Hz), 4.00 (dt, 1H, $J=2.4, 11.9$ Hz), 4.08-4.18 (m, 2H), 4.27 (dd, 1H, $J=5.1, 11.5$ Hz), 5.54 (s, 1H), 7.31-7.36 (m, 3H), 7.46-7.47 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 12.2, 18.3, 31.6, 39.4, 43.5, 62.9, 67.4, 69.6, 76.3, 101.5, 126.4, 128.6, 129.1, 138.9; MS (EI) m/z 153.3, 409.0 ($\text{M}^+\text{+H}$).

6h: IR (film) 3498, 2942, 2865, 1100 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.04-1.09 (m, 21H), 1.30 (d, 3H, $J=5.2$ Hz), 1.50-1.59 (m, 2H), 1.64-1.85 (m, 4H), 3.77 (dt, 2H, $J=2.5, 12.1$ Hz), 3.87-3.95 (m, 3H), 4.03-4.06 (m, 1H), 4.09 (ddd, 1H, $J=1.0, 5.1, 11.6$ Hz); 4.72 (q, 1H, $J=5.2$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 11.7, 17.9, 21.2, 31.0, 39.2, 43.1, 62.2, 66.4, 69.2, 75.5, 98.8; MS (EI) m/z 153.3, 347.1 ($\text{M}^+\text{+H}$).

6i: IR (film) 3503, 2944, 2924, 2866, 1099 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.05-1.10 (m, 21H), 1.53-1.88 (m, 6H), 3.73 (dt, 2H, $J=2.5, 11.9$ Hz), 3.84-3.98 (m, 3H), 4.00-4.06 (m, 1H), 4.10 (dd, 1H, $J=4.9, 11.4$ Hz), 4.72 (d, 1H, $J=6.2$ Hz), 5.05 (d, 1H, $J=6.2$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 11.7, 17.9, 31.9, 38.9, 43.1, 62.5, 66.6, 69.1, 75.4, 93.7; MS (EI) m/z 153.3, 333.2 ($\text{M}^+\text{+H}$).

H1 standard parameters, CDCl3
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-30008 "varian"
PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 49.1 degrees
Acq. time 2.105 sec
Width 3799.4 Hz
8 repetitions
OBSERVE H1, 299.8959758 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 0 min, 44 sec

3 standard parameters, CDCl₃

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

Mercury-300BB "varian"

PULSE SEQUENCE

Relax. delay 2.000 sec

Pulse 26.2 degrees

Acq. time 0.462 sec

Width 17006.8 Hz

352 repetitions

OBSERVE G13, 75.4089220 MHz

DECOUPLE H1, 299.8977540 MHz

Power 58 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 3.0 Hz

FI size 32768

Total time 1 hr, 26 min, 26 sec

¹H standard parameters, CDC13

Pulse Sequence: s2pu1
Solvent: CDC13
Ambient temperature
Mercury-300BB "varian"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 26.5 degrees
Acq. time 2.276 sec
Width 3539.7 Hz
8 Repetitions
OBSERVE H1 299.9291353 MHZ
DATA PROCESSING
Line broadening 0.3 Hz
FT/size 16384
Total time 0 min, 36 sec

2c

3 standard parameters, C0C13

Pulse Sequence: s2pul
Solvent: C0C13
Ambient temperature
Mercury-30088 "varian"
PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 36.7 degrees
Acq. time 0.482 sec
Width 17006.8 Hz
32 repetitions
OBSERVE C13, 75.4172461 MHz
DECOUPLE H1, 299.9307943 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 16384
Total time 8 min, 45 sec

17.792
11.707

30.291

59.500

72.440

77.368

200 180 160 140 120 100 80 60 40 20 0 ppm

Carbon-13 standard parameters. 880 probe

Proton standard parameters, BB0 probe

Current Data Parameters
 NAME bnketone
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020605
 Time 19.20
 INSTRUM spect
 PROBHD 5 mm BB0 BB-1
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 5009.615 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 203.2
 DW 83.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 5.00 usec
 SF01 500.1327507 MHz
 NUC1 1H
 PL1 3.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300130 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 10.100 ppm
 F1 5051.31 Hz
 F2P -0.100 ppm
 F2 -50.01 Hz
 PPMCM 0.51000 ppm/cm
 HZCM 255.06631 Hz/cm

Hui Lei sample in CDCl₃, 6-6-02, 880 probe.

Proton standard parameters, 880 probe

Current Data Parameters
NAME 6-B3ketone
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010111
Time 10.53
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 64
OW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 5.00 usec
SF01 500.1327507 MHz
NUC1 1H
PL1 3.00 dB

F2 - Processing parameters
SI 16384
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 10.000 ppm
F1 5001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/
HZCM 250.06500 Hz/c

Carbon-13 standard parameters, 880 probe

Current Data Parameters
NAME 6-B3ketone
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010111
Time 10.58
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 309
DS 2
SWH 30303.031 Hz
FIDRES 0.924775 Hz
AQ 0.5407220 sec
RG 13004
DM 16.500 usec
DE 6.00 usec
TE 300.0 K
d11 0.03000000 sec
d12 0.00020000 sec
PL13 22.00 dB
D1 2.00000000 sec
CPOPRG2 waltz16
PCPD2 80.00 usec
SF02 500.1320000 MHz
NUC2 1H
PL2 3.00 dB
PL12 22.00 dB
P1 3.80 usec
SF01 125.7715724 MHz
NUC1 13C
PL1 3.00 dB

F2 - Processing parameters
SI 32768
SF 125.7577390 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 27666.70 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1383.33508 Hz/cm

Proton standard parameters, 880 probe

Current Data Parameters
 NAME nmr
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameter
 Date_ 20010214
 Time 17.00
 INSTRUM spect
 PROBD 5 mm BBO BB-1
 PULPROG zg
 TO 16384
 SOLVENT CDCl₃
 NS 8
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.292314 Hz
 AQ 1.7105396 s
 RG 228.1
 DW 104.400 µs
 DE 6.00 µs
 TE 300.0 K
 D1 1.00000000 s
 P1 5.50 µs
 SF01 400.1342000 MHz
 NUC1 ¹H
 PL1 -3.00 dB
 F2 - Processing parameters
 SI 8192
 SF 400.1320090 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 cm
 F1P 10.100 pF
 F1 4041.33 Hz
 F2P -0.100 pF
 F2 -40.01 Hz
 PPMCM 0.51000 pF
 HZCM 204.06735 Hz

Carbon-13 standard parameters, BBO probe

C13 standard parameters, CDCl3

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 Mercury-30088 "varian"
 PULSE SEQUENCE
 Relax. delay 2.000 sec
 Pulse 26.2 degrees
 Acq. time 0.482 sec
 Width 17000.0 Hz
 300 repetitions
 OBSERVE C13, 75.4171082 MHz
 DECOUPLE H1, 299.9903081 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 3.0 Hz
 FI size 32768
 Total time 13 min, 4 sec

Proton standard parameters, BBI probe

Current Data Parameters
NAME ketone6-55
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20001219
Time 11:55
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 16
DW 104.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SF01 400.1342000 MHz
NUC1 1H
PL1 0.00 dB

F2 - Processing parameters
SI 8192
SF 400.1320090 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.32 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHMC 0.50000 ppm/cm
HZCM 200.06601 Hz/cm

Carbon-13 standard parameters, BBI probe

Current Data Parameters
 NAME ketone6-55
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20001219
 Time 12.03
 INSTRUM spect
 PROBHD 5 mm BBI 1H-B
 PULPROG zgpg
 TO 32768
 SOLVENT CDCl3
 NS 414
 DS 2
 SMH 24154.590 Hz
 FIDRES 0.737140 Hz
 AQ 0.6783478 sec
 RG 9195.2
 DW 20.700 usec
 DE 29.57 usec
 TE 300.0 K
 d11 0.03000000 sec
 d12 0.0002000 sec
 PL13 21.00 dB
 D1 2.00000000 sec
 CPDPRG2 waltz16
 PCPD2 80.00 usec
 SF02 400.1342220 MHz
 NUC2 1H
 PL2 0.00 dB
 PL12 21.00 dB
 P1 4.50 usec
 SF01 100.6242960 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters

SF 32768
 SF 100.6132740 MHz
 EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.92 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.74597 Hz/cm

C13 standard parameters, CDC13

Pulse Sequence: s2pul

Solvent: CDC13

Ambient temperature

Mercury-300BB "varian"

PULSE SEQUENCE

Relax. delay 2.000 sec

Pulse 26.2 degrees

Acq. time 0.482 sec

Width 17006.8 Hz

30000 repetitions

OBSERVE C13, 75.4144711 MHz

DECOUPLE H1, 299.9198709 MHz

Power 58 db

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 3.0 Hz

FT size 32768

Total time 21 hr, 34 min, 55 sec

¹H standard parameters, CDCl₃

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

Mercury-300088 "varian"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 28.9 degrees

Acq. time 2.276 sec

Width 3599.7 Hz

8 repetitions

OBSERVE H1, 299.9291353 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 16384

Total time 0 min, 36 sec

Standard parameters, CDC13

Sequence: s2pul
 Solvent: CDC13
 Ambient temperature
 Mercury-30088 "varian"
 PULSE SEQUENCE
 Relax. delay 2.000 sec
 Pulse 36.7 degrees
 Acq. time 0.482 sec
 Width 17006.8 Hz
 512 repetitions
 OBSERVE C13, 75.4172461 MHz
 DECOUPLE H1, 299.9307943 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FT size 16384
 Total time 1 hr, 26 min, 22 sec

H1 standard paramters, CDC13

Pulse Sequence: s2pul

Solvent: CDC13

Ambient temperature

Mercury-300BB "varian"

PULSE SEQUENCE

Relax. delay 2.000 sec

Pulse 49.1 degrees

Acq. time 2.105 sec

Width 3799.4 Hz

8 repetitions

OBSERVE H1, 299.9181253 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FI size 16384

Total time 0 min, 44 sec

Standard parameters, CDC13

File Sequence: s2pul
 Solvent: CDC13
 Ambient temperature
 Mercury-30088 "varian"
 PULSE SEQUENCE
 Relax. delay 2.000 sec
 Pulse 26.2 degrees
 Acq. time 0.482 sec
 Width 17006.8 Hz
 436 repetitions
 OBSERVE C13, 75.4144711 MHz
 DECOUPLE H1, 299.3198709 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 3.0 Hz
 FT size 32768
 Total time 14 hr, 23 min, 19 sec

Carbon-13 standard parameters, 880 probe

Current Data Parameters	
NAME	B-16E
EXPNO	2
PROCNO	1
F2 - Acquisition Parameters	
Date_	20020115
Time	14.17
INSTRUM	spect
PROBHD	5 mm BBO BB-1
PULPROG	zgpg
TD	32768
SOLVENT	cdcl3
NS	1188
DS	2
SWH	24154.590
FIDRES	0.737140
AQ	0.6783476
RG	4096
DW	20.700000
DE	29.57
TE	300.0
d11	0.03000000
d12	0.00002000
PL13	19.00
D1	2.00000000
CPDPRG2	waltz16
PCPD2	75.00
SF02	400.1342220
NUC2	¹³ C
PL2	120.00
PL12	16.00
P1	3.00
SF01	100.6242960
NUC1	¹ H
PL1	6.00
F2 - Processing parameters	
SI	32768
SF	100.6132740
WDW	EM
SSB	0
LB	3.00
GB	0
PC	1.40
1D NMR plot parameters	
CX	20.00
F1P	220.000000
F1	22134.92
F2P	0.000
F2	0.00
PPMCM	11.00000
HZCM	1106.74597

H1 standard parameters, CDC13

Pulse Sequence: s2pul

Solvent: CDC13

Ambient temperature

Mercury-300BB "varian"

PULSE SEQUENCE

Relax. delay 2.000 sec

Pulse 49.1 degrees

Acq. time 2.105 sec

Width 3799.4 Hz

32 repetitions

OBSERVE H1, 299.8959758 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 16384

Total time 2 min, 27 sec

wdd

Proton standard parameters, BB0 probe

Current Data Parameters
 NAME diphenylme
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020513
 Time 19.08
 INSTRUM spect
 PROBHD 5 mm BB0 BB-1
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.292314 Hz
 AQ 1.7105396 sec
 RG 203.2
 DW 104.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 5.50 usec
 SF01 400.1342000 MHz
 NUC1 1H
 PL1 -3.00 dB

F2 - Processing parameters
 SI 8192
 SF 400.1320090 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.100 ppm
 F1 4041.33 Hz
 F2P -0.100 ppm
 F2 -40.01 Hz
 PPMCM 0.51000 ppm/cm
 HZCM 204.06735 Hz/cm

Carbon-13 standard parameters, BBO probe

Proton standard parameters, BBO probe

Current Data Parameters
 NAME trred
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020518
 Time 16.11
 INSTRUM spect
 PROBHD 5 mm BBO BB-J
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 6009.615 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 406.4
 DW 83.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 5.00 usec
 SF01 500.1327507 MHz
 NUC1 1H
 PL1 3.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300130 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.50000 ppm/c
 HZCM 250.06500 Hz/cm