Asymmetric Synthesis of Cyclic Hydroxy Ketones Derived from Enol
Ethers via the Sharpless Asymmetric Dihydroxylation. A Study in the
Correlation of the Enol Ether Chain Length and Enantioselectivity.

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Experimental.

General All NMR spectra were recorded in CDCl₃ at 400 MHz for ¹H and 100 MHz for ¹³C. The abbreviation om stands for overlapping multiplets. Enantiomeric excess data was obtained from reaction mixtures after work up but before purification. The ee traces were recorded by chiral supercritical fluid chromatography (SFC): AD column, isocratic method 10% MeOH/CO₂ @ 1.5 mL/min, 200 bar, 35 °C, and Chiral GC: Cyclosil B column (Length=30 m; I.D.=0.25 mm; Film=0.25 μm); Flow Rate = 6 mL/min, start temperature 35 °C; then 2.5 °C/min to 110 °C; 10° C/min to 170 °C; 15 °C/min to 230 °C. GCMS: EI ionization. Column (DB-5ms, length = 30 m, id = 0.25 mm, film = 0.25 μm). Enol ethers **2a-c**, **6a**, **10a**, and **12a** were prepared according to the literature procedure (ref. # 2). MPA esters **14a**, **14b**, **15a**, **15b**, **16a**, and **16b** were prepared according to the literature procedure (ref. # 11). All esters were purified by preparative plate chromatography with 100% dichloromethane.

4-Methoxy-2H-chromene (2a). Distillation at 145 °C and 27 torr, gave **2a** as a colorless oil, in 88% yield, and 99 area% pure by HPLC. ¹H NMR (CDCl₃, 400 MHz) δ 3.73 (s, 3H), 4.75 (t, *J* = 3.8, 1H), 4.90 (d, *J* = 3.7, 2H), 6.90, (dd, *J* = 8.1, 0.8, 1H), 6.98, (m, 1H), 7.22 (m, 1H), 7.51 (dd, *J* = 7.6, 1.6, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 54.7, 65.8, 89.4, 115.7, 120.6, 121.0, 122.2, 129.8, 150.5, 155.5; GCMS *m/z* 161, 131, 91, 65.

4-Ethoxy-2H-chromene (2b). Distillation at 143 °C and 15 torr gave **2b** as a colorless oil, in 70% yield, and 93 area% pure by HPLC. ¹H NMR (CDCl₃, 400 MHz) δ 1.45 (t, *J* = 7.0, 3H), 3.91 (q, *J* = 7.0 2H), 4.72 (t, *J* = 3.8, 1H), 4.87 (d, *J* = 3.8, 2H), 6.86, (dd, *J* = 8.1, 1.1, 1H), 6.95 (td, *J* = 7.5, 1.2, 1H), 7.19 (td, *J* = 7.8, 1.6, 1H), 7.51 (dd, *J* = 7.6, 1.7, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 14.5, 62.9, 65.8, 89.7, 115.7, 120.8, 121.0, 122.3, 129.7, 149.6, 155.5; GCMS *m/z* 176, 147, 121, 91, 65.

4-Propoxy-2H-chromene (2c). Distillation at 146 °C and 11 torr gave **2c** as a colorless oil ¹H NMR (CDCl₃, 400 MHz) δ 1.12 (t, *J* = 7.4, 3H), 1.87 (m, 2H), 3.82 (t, *J* = 6.4, 2H), 4.73 (t, *J* = 3.8, 1H), 4.89 (d, *J* = 3.9, 2H), 6.88 (d, *J* = 8.1, 1H), 6.97 (t, *J* = 7.6, 1H), 7.21 (td, *J* = 7.6, 1.6, 1H), 7.54 (dd, *J* = 7.6, 1.5, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 10.8, 22.4, 65.8, 68.8, 89.6, 115.6, 120.8, 120.9, 122.2, 129.6, 149.7, 155.5; GCMS *m/z* 190, 147, 121, 91, 65.

4-Butoxy-2H-chromene (2d). Distillation at 140 °C and 4.5 torr gave **2d** as a colorless oil. ¹H NMR (CDCl₃, 400 MHz) δ 1.00 (t, *J* = 7.4, 3H), 1.54 (m, 2H), 1.79 (m, 2H), 3.85 (t, *J* = 6.4, 2H), 4.73 (t, *J* = 3.8, 1H), 4.86 (d, *J* = 3.8, 2H), 6.82 (d, *J* = 8.0, 1H), 6.92 (t, *J* = 7.6, 1H), 7.17 (td, *J* = 7.6, 1.6, 1H), 7.46, (dd, *J* = 7.6, 1.6, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 13.9, 19.5, 31.1, 65.8, 67.0, 89.6, 115.6, 120.8, 120.9, 122.3, 129.6, 149.7, 155.5; GCMS *m/z* 204, 176, 147, 121, 91, 65.

4-Pentoxy-2H-chromene (2e). Distillation at 155 °C and 10 torr gave **2e** as a colorless oil in 50% yield and 99 area% pure by HPLC. ^1H NMR (CDCl_3 , 400 MHz) δ 0.95 (t, $J = 7.2$, 3H), 1.47 (m, 4H), 1.80 (m, 2H), 3.83 (t, $J = 6.4$, 2H), 4.71 (t, $J = 3.8$, 1H), 4.85 (d, $J = 3.8$, 2H), 6.81 (dd, $J = 8.0$, 1.0, 1H), 6.91 (td, $J = 7.4$, 1.1, 1H), 7.16 (td, $J = 7.7$, 1.7, 1H), 7.45 (dd, $J = 7.6$, 1.6, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 14.1, 22.6, 28.5, 28.7, 65.8, 67.3, 89.6, 115.6, 120.8, 120.9, 122.3, 129.6, 149.7, 155.5; GCMS m/z 218, 190, 147, 121, 91, 55.

4-Decyloxy-2H-chromene (2f). Distillation at 1160 °C and 1 torr gave **2f** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 0.92 (m, 3H), 1.31 (m, 12H), 1.48 (m, 2H), 1.81 (m, 2H), 3.84 (t, $J = 6.4$, 2H), 4.72 (t, $J = 3.6$, 1H), 4.86 (d, $J = 3.8$, 2H), 6.83 (d, $J = 8.0$, 1H), 6.93 (td, $J = 7.4$, 1.0, 1H), 7.17 (td, $J = 7.6$, 1.6, 1H), 7.47 (dd, $J = 7.4$, 1.6, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 14.1, 22.7, 26.2, 28.9, 29.3, 29.4, 29.6 (2C), 31.9, 65.7, 67.2, 89.4, 115.5, 120.7, 120.9, 122.2, 129.5, 149.6, 155.3.

4-Isobutoxy-2H-chromene (2h). Distillation at 115 °C and 1 torr gave **2h** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 1.05 (d, $J = 6.8$, 6H), 1.82 (m, 1H), 3.61 (d, $J = 6.4$, 2H), 4.71 (t, $J = 3.8$, 1H), 4.85 (d, $J = 4.0$, 2H), 6.81 (d, $J = 8.0$, 1H), 6.92 (t, $J = 7.4$, 1H), 7.16 (t, $J = 7.4$, 1H), 7.47 (d, $J = 8.0$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 19.3, 28.1, 65.7, 73.5, 89.4, 115.6, 120.8, 120.9, 122.1, 129.6, 149.6, 155.3.

4-Isopentoxy-2*H*-chromene (2i). Distillation at 130 °C and 1 torr gave **2i** as a colorless oil. ¹H NMR (CDCl₃, 400 MHz) δ 0.99 (d, *J* = 4.4, 6H), 1.71 (q, *J* = 6.4, 2H), 1.87 (m, 1H), 3.87 (t, *J* = 6.6, 2H), 4.73 (t, *J* = 3.8, 1H), 4.86 (d, *J* = 4.0, 2H), 6.82 (m, 1H), 6.92 (m, 1H), 7.17 (td, *J* = 7.8, 1.6, 1H), 7.45 (dd, *J* = 7.6, 1.6, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 22.6, 25.3, 37.7, 65.6, 65.7, 89.5, 115.6, 120.9, 122.1, 122.2, 129.6, 149.6, 155.4; GCMS *m/z* 218, 147, 121, 91, 55.

4-(2-methoxyethoxy)-2*H*-chromene (2j). Distillation at 140 °C and 3 torr gave **2j** as a colorless oil. ¹H NMR (CDCl₃, 400 MHz) δ 3.47 (s, 3H), 3.77 (m, 2H), 4.00 (m, 2H), 4.74 (t, *J* = 3.8, 1H), 4.85 (d, *J* = 3.8, 2H), 6.81 (dd, *J* = 8.1, 0.8, 1H), 6.91 (td, *J* = 7.5, 1.0, 1H), 7.16 (td, *J* = 7.8, 1.6, 1H), 7.47 (dd, *J* = 7.6, 1.6, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 59.2, 65.6, 66.8, 70.7, 89.9, 115.5, 120.4, 120.9, 122.3, 129.7, 149.6, 155.3; GCMS *m/z* 206, 148, 120, 91, 59.

4-Benzylloxy-2*H*-chromene (2k). Using a short path distillation head, two fractions were collected at 120 °C and 2 torr. Fraction 2 contained **2k** and approximately 30 mol% by-product, identified as α-benzylchromanone. ¹H NMR (CDCl₃, 400 MHz) δ 4.85 (t, *J* = 3.8, 1H), 4.90 (d, *J* = 3.8, 2H), 4.98 (s, 2H), 6.89 (d, *J* = 7.5, 1H), 6.97 (t, *J* = 7.6, 1H), 7.22 (td, *J* = 7.7, 1.6, 1H), 7.29 (m, 1H), 7.36-7.50 (om, 4H), 7.57 (dd, *J* = 7.6, 1.6, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 65.8, 69.3, 90.7, 115.7, 121.1, 122.5, 127.4, 128.1, 128.6, 129.9, 136.0, 136.9, 149.5, 155.5; GCMS *m/z* 238, 147, 120, 91, 65.

4-Methoxy-1,2-dihydronaphthalene (6a). Distillation at 110 °C and 12 torr gave **6a** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 2.41 (m, 2H), 2.83 (t, $J = 7.8$, 2H), 3.77 (s, 3H), 5.05 (t, $J = 4.4$, 1H), 7.19-7.30 (om, 3H), 7.61 (d, $J = 7.2$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 21.9, 28.4, 54.5, 95.1, 121.6, 126.2, 127.1, 127.5, 132.0, 137.0, 152.8; GCMS m/z 160, 115, 91, 63.

4-Propoxy-1,2-dihydronaphthalene (6b). Distillation at 125 °C and 5 torr gave **6b** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 1.10 (t, $J = 7.4$, 3H), 1.86 (m, 2H), 2.37 (m, 2H), 2.80 (t, $J = 7.8$, 2H), 3.83 (t, $J = 6.4$, 2H), 5.01 (t, $J = 4.0$, 1H), 7.15-7.27 (om, 3H), 7.56 (d, $J = 7.6$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 10.9, 22.0, 22.6, 28.4, 68.5, 95.6, 121.6, 126.2, 127.0, 127.4, 132.3, 137.0, 152.0; GCMS m/z 188, 146, 115, 91, 63.

4-Pentoxo-1,2-dihydronaphthalene (6c). Distillation at 155 °C and 12 torr gave **6c** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 0.97 (t, $J = 7.1$, 3H), 1.38-1.56 (om, 4H), 1.80 (m, 2H), 2.36 (m, 2H), 2.78 (t, $J = 7.8$, 2H), 3.84 (t, $J = 6.4$, 2H), 4.99 (t, $J = 4.7$, 1H), 7.19 (m, 3H), 7.57 (d, $J = 7.6$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 14.0, 21.9, 22.5, 28.3, 28.5, 28.9, 66.9, 95.5, 121.6, 126.1, 127.0, 127.3, 132.2, 137.0, 152.0; GCMS m/z 216, 188, 146, 118, 91, 55.

4-Isopropoxy-1,2-dihydronaphthalene (6d). Distillation gave a clear colorless oil containing 84 mol% of **6d** at 110 °C and 2 torr. ^1H NMR (CDCl_3 , 400 MHz) δ 1.39

(d, $J = 6.0$, 6H), 2.40 (m, 2H), 2.81 (t, $J = 7.9$, 2H), 4.44 (m, 1H), 5.07 (t, $J = 4.7$, 1H), 7.16-7.28 (om, 3H), 7.61 (dd, $J = 7.6$, 1.4, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 22.0, 22.1, 28.5, 68.6, 97.6, 121.9, 126.2, 127.1, 127.3, 132.7, 137.3, 150.3; GCMS m/z 188, 146, 115, 91, 63.

4-Isobutoxy-1,2-dihydronaphthalene (6e). Distillation at 120 °C and 1 torr gave **2j** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 1.07 (d, $J = 6.7$, 6H), 2.14 (m, 1H), 2.36 (m, 2H), 2.78 (t, $J = 7.8$, 2H), 3.62 (d, $J = 6.4$, 2H), 4.98 (t, $J = 4.5$, 1H), 7.13-7.23 (om, 3H), 7.60 (d, $J = 7.2$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 19.5, 22.0, 28.3, 28.4, 73.3, 95.5, 121.6, 126.2, 127.0, 127.4, 132.3, 137.0, 152.0; GCMS m/z 202, 147, 115, 91, 57.

4-Phenoxy-1,2-dihydronaphthalene (6f). Distillation at 150 °C and 20 torr gave **6f** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 2.43 (m, 2H), 2.90 (t, $J = 8.0$, 2H), 5.41 (t, $J = 4.7$, 1H), 7.06-7.12 (om, 3H), 7.19-7.23 (om, 3H), 7.32-7.37 (om, 2H), 7.46 (m, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 22.0, 28.0, 109.1, 118.2, 121.9, 122.7, 126.4, 127.4, 127.8, 129.5, 131.2, 137.0, 150.9, 157.2.

9-Methoxy-6,7-dihydro-5H-benzo[*a*]cycloheptene (8a). Distillation at 125 °C and 14 torr gave **8a** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 1.90 (q, $J = 7.1$, 2H), 2.08 (m, 2H), 2.65 (t, $J = 6.9$, 2H), 3.73 (s, 3H), 5.16 (t, $J = 7.1$, 1H), 7.20-7.30 (om,

3H), 7.50 (d, $J = 7.2$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 22.8, 32.6, 34.2, 55.1, 97.8, 126.0, 126.7, 127.9, 128.8, 137.1, 141.0, 155.6; GCMS m/z 174, 142, 115, 91, 63.

9-Propoxy-6,7-dihydro-5H-benzo[*a*]cycloheptene (8b). Distillation at 110 °C and 2 torr gave **8b** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 1.06 (t, $J = 7.4$, 3H), 1.76-1.92 (om, 4H), 2.07 (m, 2H), 2.65 (t, $J = 6.8$, 2H), 3.80 (t, $J = 6.5$, 2H), 5.15 (t, $J = 7.0$, 1H), 7.19-7.28 (om, 3H), 7.52 (d, $J = 7.3$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 10.8, 22.6, 22.9, 32.5, 34.2, 69.1, 98.7, 125.9, 126.7, 127.7, 128.7, 137.2, 141.1, 154.8.

9-Pentoxo-6,7-dihydro-5H-benzo[*a*]cycloheptene (8c). Distillation at 130 °C and 2 torr gave **8c** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 1.00 (t, $J = 7.2$, 3H), 1.40-1.55 (om, 4H), 1.82 (m, 2H), 1.91 (m, 2H), 2.10 (m, 2H), 2.69 (t, $J = 6.9$, 2H), 3.86 (t, $J = 6.5$, 2H), 5.18 (t, $J = 7.1$, 1H), 7.22-7.33 (om, 3H), 7.56 (dd, $J = 7.6$, 1.4, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 14.1, 22.6, 22.9, 28.5, 29.0, 32.5, 34.3, 67.6, 98.7, 125.9, 126.7, 127.7, 128.7, 137.3, 141.1, 154.9; GCMS m/z 230, 201, 160, 131, 91, 55.

9-Isopropoxy-6,7-dihydro-5H-benzo[*a*]cycloheptene (8d). Distillation gave a clear colorless oil containing **8d** (85 mol%) and benzosuberone (15 mol%). at 103 °C and 2 torr. ^1H NMR (CDCl_3 , 400 MHz) δ 1.31 (d, $J = 6.0$, 6H), 1.87 (q, $J = 7.2$, 2H), 2.08 (m, 2H), 2.66 (t, $J = 6.9$, 2H), 4.29 (m, 1H), 5.29 (t, $J = 7.1$, 1H), 7.19-7.30 (om, 3H), 7.49 (d,

$J = 8.3$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 22.0, 22.9, 32.6, 34.4, 69.1, 102.9, 126.0, 126.8, 127.7, 128.8, 137.3, 141.5, 153.0; GCMS m/z 202, 173, 145, 115, 91, 65.

1-Methoxycyclohexene (10a). Distillation gave a clear colorless oil at 139 °C atmospheric pressure. ^1H NMR (CDCl_3 , 400 MHz) δ 1.50 (m, 2H), 1.62 (m, 2H), 1.98-2.03 (om, 4H), 3.43 (s, 3H), 4.55 (t, $J = 3.4$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 22.8, 22.9, 23.4, 27.7, 53.4, 92.8, 155.4; GCMS m/z 112, 84, 54.

1-Pentoxycyclohexene (10b). Distillation gave a clear colorless oil containing 81 mol% of **10b** at 110 °C and 2 torr. ^1H NMR (CDCl_3 , 400 MHz) δ 0.91 (m, 3H), 1.34-1.38 (om, 4H), 1.54 (m, 2H), 1.62-1.70 (om, 4H), 2.02-2.07 (om, 4H), 3.61 (t, $J = 6.6$, 2H), 4.59 (t, $J = 2.8$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 14.0, 22.6, 22.9, 23.0, 23.6, 28.0, 28.5, 29.0, 66.2, 93.5, 154.7; GCMS 168, 125, 98, 70.

1-Methoxycycloheptene (12a). Distillation at 160 °C atmospheric pressure, gave **12a** as a colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 1.44-1.54 (om, 4H), 1.68 (m, 2H), 2.04 (m, 2H), 2.23 (m, 2H), 3.40 (s, 3H), 4.68 (t, $J = 6.7$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 25.3, 25.6, 28.2, 32.1, 33.7, 54.0, 96.2, 162.0; GCMS m/z 126, 97, 72, 50.

1-Pentoxycycloheptene (12b). Distillation gave a clear colorless oil containing 90 mol% of **12b** at 110 °C and 2 torr. ^1H NMR (CDCl_3 , 400 MHz) δ 0.91 (m, 3H), 1.33-1.38 (om, 4H), 1.48-1.57 (om, 4H), 1.63-1.72 (om, 4H), 2.05 (m, 2H), 2.26 (m, 2H), 3.52

(t, $J = 6.6$, 2H), 4.69 (t, $J = 6.8$, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 13.9, 22.4, 25.4, 25.6, 28.2, 28.3, 28.8, 32.2, 33.8, 66.6, 96.6, 161.4; GCMS m/z 182, 139, 112, 83, 55.

(*R*)- α -Hydroxytetralone (7) from 6c. $[\alpha]_{\text{D}}^{20} +28.24$ (CHCl_3); SFC retention time, 7.2 min for *R* enantiomer, 6.8 min for *S* enantiomer.

(*R*)- α -Hydroxybenzosuberone (9) from 8c. ^1H NMR (CDCl_3 , 400 MHz) δ 1.64-1.84 (om, 2H), 2.16 (m, 1H), 2.37 (m, 1H), 2.89-3.06 (om, 2H), 4.54 (dd, $J = 11.4$, 5.4, 1H), 7.25 (d, $J = 7.6$, 1H), 7.34 (t, $J = 7.67$, 1H), 4.6 (td, $J = 7.6$, 1.2, 1H) 7.92 (dd, $J = 7.6$, 1.2, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 23.6, 32.3, 34.7, 76.0, 126.9, 129.8, 130.6, 133.1, 135.1, 143.1, 204.7; GCMS m/z 176, 147, 120, 91, 65; SFC retention time, 9.5 min for *R* enantiomer, 7.1 min for *S* enantiomer.

(*S*)- α -Hydroxycyclohexanone (11) from 10a. $[\alpha]_{\text{D}}^{20} -20.0$ (CH_3Cl); Chiral GC retention time, 26.9 min for *S* enantiomer, 25.6 min for *R* enantiomer.

(*S*)- α -Hydroxycycloheptanone (13) from 12a. $[\alpha]_{\text{D}}^{20} -143.5$ (CH_3Cl); ^1H NMR (CDCl_3 , 400 MHz) δ 1.29 (m, 1H), 1.49-1.67 (om, 3H), 1.70-1.86 (om, 3H), 1.99 (m, 1H), 2.46 (ddd, $J = 11.2$, 7.7, 3.6, 1H), 2.68 (m, 1H), 4.29 (dd, $J = 9.2$, 3.6, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 23.5, 26.6, 29.6, 33.8, 40.1, 77.1, 213.8; GCMS m/z 128, 110, 99, 81, 67, 57, 50; Chiral GC retention time, 32.0 min for *S* enantiomer, 31.2 min for *R* enantiomer.

(6R)-5-oxo-6,7,8,9-tetrahydro-5H-benzo[a][7]annulen-6-yl (2R)-

(methyloxy)(phenyl)ethanoate (14b). ¹H NMR (CDCl₃, 400 MHz) δ 1.86 (m, 1H), 2.04 (m, 1H), 2.09 (m, 1H), 2.22 (m, 1H), 2.93 (m, 2H), 3.48 (s, 3H), 4.84 (s, 1H), 5.40 (dd, *J* = 9.1, 5.3, 1H), 7.12 (d, *J* = 7.6, 1H), 7.26 (t, *J* = 7.6, 1H), 7.34 (om, 3H), 7.36 (td, *J* = 7.6, 1.5, 1H), 7.40 (m, 2H), 7.65 (d, *J* = 7.6, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 23.5, 29.2, 34.0, 57.6, 78.7, 82.6, 126.7, 127.2, 128.5, 128.6, 129.2, 129.8, 131.9, 135.9, 136.5, 141.2, 170.1, 199.2.

(3R)-4-oxo-3,4-dihydro-2H-chromen-3-yl (2S)-(methyloxy)(phenyl)ethanoate

(15a). ¹H NMR (CDCl₃, 400 MHz) δ 3.52 (s, 3H), 4.27 (dd, *J* = 11.6, 10.8, 1H), 4.40 (dd, *J* = 10.8, 5.6, 1H), 4.96 (s, 1H), 5.73 (dd, *J* = 11.6, 5.6, 1H), 6.97 (d, *J* = 8.4, 1H), 7.07 (m, 1H), 7.39 (m, 3H), 7.50 (m, 3H), 7.90 (dd, *J* = 8.0, 1.6, 1H), ¹³C NMR (CDCl₃, 100 MHz) δ 57.6, 67.9, 69.8, 82.2, 117.8, 119.7, 122.1, 127.3, 127.6, 128.7, 129.0, 135.8, 136.5, 161.3, 169.5, 187.4.

(3S)-4-oxo-3,4-dihydro-2H-chromen-3-yl (2S)-(methyloxy)(phenyl)ethanoate

(15b). ¹H NMR (CDCl₃, 400 MHz) δ 3.52 (s, 3H), 4.43 (dd, *J* = 12.0, 11.6, 1H), 4.53 (dd, *J* = 11.2, 5.6, 1H), 5.94 (s, 1H), 5.69 (dd, *J* = 11.6, 5.6, 1H), 6.98 (d, *J* = 8.0, 1H), 7.04 (m, 1H), 7.39 (m, 3H), 7.50 (m, 3H), 7.84 (dd, *J* = 8.0, 1.6, 1H), ¹³C NMR (CDCl₃, 100 MHz) δ 57.6, 68.1, 69.9, 82.5, 117.8, 119.7, 122.1, 127.3, 127.6, 128.7, 128.9, 135.6, 136.5, 161.2, 169.6, 187.

(2*R*)-1-oxo-1,2,3,4-tetrahydro-2-naphthalenyl (2*S*)-

(methyloxy)(phenyl)ethanoate (16a). ¹H NMR (CDCl₃, 400 MHz) δ 2.14-2.26 (om, 2H), 3.01 (dt, *J* = 16.9, 4.0, 1H), 3.15 (m, 1H), 3.56 (s, 3H), 4.99 (s, 1H), 5.59 (m, 1H), 7.25 (br d, *J* = 7.6, 1H), 7.37 (om, 4H), 7.51 (om, 3H), 8.04 (dd, *J* = 7.6, 1.2, 1H), ¹³C NMR (CDCl₃, 100 MHz) δ 27.7, 28.8, 57.6, 75.1, 82.3, 127.0, 127.3, 127.9, 128.6, 128.6, 128.7, 131.4, 134.0, 136.2, 143.0, 170.0, 192.3.

(2*S*)-1-oxo-1,2,3,4-tetrahydro-2-naphthalenyl (2*S*)-

(methyloxy)(phenyl)ethanoate (16b). ¹H NMR (CDCl₃, 400 MHz) δ 2.29-2.41 (om, 2H), 3.07 (dt, *J*=16.9, 3.6, 1H), 3.19 (m, 1H), 3.57 (s, 3H), 4.97 (s, 1H), 5.58 (m, 1H), 7.25 (br d, *J* = 7.6, 1H), 7.32 (br t, *J* = 7.6, 1H), 7.37 (m, 1H), 7.41 (m, 2H), 7.50 (td, *J* = 7.6, 1.2, 1H), 7.56 (m, 2H), 8.0 (dd, *J* = 7.6, 1.2, 1H), ¹³C NMR (CDCl₃, 100 MHz) δ 27.9, 29.0, 57.7, 75.1, 82.8, 127.0, 127.3, 127.8, 128.55, 128.6, 128.7, 131.5, 133.9, 136.1, 142.9, 170.2, 192.0.

44307-41-3 fraction 4:CDCl₃
nmr400b h-1

2a

Current Data Parameters
NAME 44307-41-3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010822
Time 16.04
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 16
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700208 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

307 react on (CDCl₃)
nmr400b h decoupled)

Current Data Parameters
NAME 44307-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010622
Time 16.07
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 101
DS 4
SWH 24315.769 Hz
FIDRES 0.401547 Hz
AQ 1.2452240 sec
RG 8192
DN 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.628112 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.628112 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D MRS plot parameters
CX 40.00 cm
FLP 240.000 ppm
F1 24131.38 Hz
F2D -10.000 ppm
F2 -1005.47 Hz
PRNCH 6.25800 ppm/cm
HSCN 528.42114 Hz/cm

29

44307-42-1 fraction 1:CDCl3
nmr400b h-1

Current Data Parameters
NAME 44307-42-1
EXTNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110823
Time 14.42
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 12
DS 2
SWH 6576.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 28.5
CW 76.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10010100 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 199.8724552 MHz

F2 - Processing parameters
SI 16384
SF 199.8700208 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1F 9.750 ppm
F1 1818.73 Hz
F2F -0.250 ppm
F2 -59.97 Hz
FREQM 0.25000 ppm/cm
RGCM 99.96751 Hz/cm

2b

44307-42-2 fraction 2; CDC13
nmr400b c-13 (h-1 decoupled)

Current Data Parameters
NAME 44307-42-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010823
Time 14.55
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpgc
TD 65536
SOLVENT CDC13
NS 265
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24131.38 Hz
F2P -10.000 ppm
F2 -1005.47 Hz
PFMCH 6.25000 ppm/cm
HZCH 628.42114 Hz/cm

2b

4430/-43-4 IRAC1001 3/LA
nmr400b h-1

Current Data Parameters
NAME 44307-43-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010827
Time 16.10
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 16
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700208 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -39.97 Hz
PCMCN 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

44307-43 chromanone propyl ether
ether; months later; CDC13
nmr400b c-13

Current Data Parameters
NAME 44307-43
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020628
Time 10.52
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDC13
NS 138
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
FIP 240.000 ppm
F1 24131.38 Hz
F2P -10.000 ppm
F2 -1005.47 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.42120 Hz/cm

2c

44307-61-12 1D, 1H, 13C, 2D
nmr400b h-1

Current Data Parameters
NAME 44307-61-12
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011017
Time 8.53
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 101.6
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700208 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCH 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

72454-64-4 Butyl enol chromanone rx
44307 61 fr5;CDCl3
nmr400b c-13

Current Data Parameters
NAME 72454-64-4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Data_ 20020724
Time 11.43
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 135
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24131.38 Hz
F2P -10.000 ppm
F2 -1005.47 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.42120 Hz/cm

2d

72454-64-3 pentyl enol chromanone rx
44307-115; CDC13
nmr400b h-1

Current Data Parameters
NAME 72454-64-3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020724
Time 11.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 16
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F1 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

nmr400a h-1

Current Data Parameters
 NAME 44180-95-12 F1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011029
 Time 11.47
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 21
 DS 2
 SWH 6578.947 Hz
 FIDRES 0.200774 Hz
 AQ 2.4904180 sec
 RG 57
 DW 76.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 -3.00 dB
 SFO1 400.2522014 MHz

F2 - Processing parameters
 SI 16384
 SF 400.2500593 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 40.00 cm
 F1P 8.000 ppm
 F1 3202.00 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCN 0.20000 ppm/cm
 HZCM 80.05001 Hz/cm

nmr400a c-13

Current Data Parameters
 NAME 44180-95-bf13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011207
 Time 11.31
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgdc
 TD 65536
 SOLVENT CDCl3
 NS 202
 DS 0
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.6250012 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 93.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.2520012 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6429588 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 F1P 200.000 ppm
 F1 20128.59 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 FPMCM 5.00000 ppm/cm
 HZCM 503.21478 Hz/cm

44307-87-7 fraction 4;CDCl3
nmr400b h-1

44307-87-7 fraction 3;CDCl3
nmr400b c-13 (h-1 decoupled)

nmr400a h-1

Current Data Parameters
NAME 44180-83-ff2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020801
Time 16.02
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 19
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 64
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SP 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 8.000 ppm
F1 3202.00 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCK 0.20000 ppm/cm
HZCK 40.05001 Hz/cm

nmr400a h-1 decoupled c-13

Current Data Parameters
 NAME 44180-83-f2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020801
 Time 16.06
 INSTRUM spect
 PROBED 5 mm QNP 1H
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 155
 DS 0
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.6539737 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 93.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.2520012 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6429588 MHz
 MW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 F1P 240.000 ppm
 F1 24154.31 Hz
 F2P -10.000 ppm
 F2 -1006.43 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 629.01849 Hz/cm

nmr400a h-1

Current Data Parameters
NAME 44180-96-f2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011105
Time 16.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 143.7
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 Hz
F1P 8.000 Hz
P1 3200.000 Hz
P2P 0.000 Hz
P2 0.00 Hz
PPMCM 0.000000 Hz/cm
H2CM 0.050000 Hz/cm

nmr400a c-13

2j

nmr400a h-1

Current Data Parameters
NAME 44180-108-f4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011113
Time 15.42
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 16
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.000 ppm
F1 3602.25 Hz
F2P 1.000 ppm
F2 400.25 Hz
PPMCM 0.20000 ppm/cm
HZCM 80.05001 Hz/cm

2j

nmr400a c-13

Current Data Parameters
NAME 44180-106-f4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011113
Time 15.55
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 485
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6251327 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6251327 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 160.000 ppm
F1 16102.87 Hz
F2P 40.000 ppm
F2 4025.72 Hz
PPHMC 3.00000 ppm/cm
HZCM 301.92889 Hz/cm

133.7
130.6
129.7
129.6
129.2
127.9
122.3
121.7
120.9
120.4
119.9
116.9
115.5
89.9
77.4
77.0
76.7
72.2
70.7
67.4
66.8
65.6
62.2
59.2
59.1
59.0

nmr400b h-1

2K

Current Data Parameters
NAME 44307-82-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011108
Time 15.48
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 57
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700208 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

44307-82-4 fraction 2;CDCl₃
nmr400b c-13 (h-1 decoupled)

Current Data Parameters
NAME 44307-82-4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011108
Time 15.52
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 184
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DN 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24131.37 Hz
F2P -10.000 ppm
F2 -1005.48 Hz
PFMCM 6.25000 ppm/cm
HZCM 628.42120 Hz/cm

nmr400a h-1

69

nmr400a h-1 decoupled c-13

Current Data Parameters
 NAME 44180-78-f1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020731
 Time 15.09
 INSTRUM spect
 PROBRD 5 mm QNP 1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 95
 DS 0
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.6259737 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 93.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.2520012 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6259588 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 P1P 240.000 ppm
 F1 24154.31 Hz
 F2P -10.000 ppm
 F2 -1006.43 Hz
 PPMCH 6.25000 ppm/cm
 HZCH 629.01849 Hz/cm

eb

nmr400a h-1

nmr400a h-1 decoupled c-13

Current Data Parameters
 NAME 44180-79-ff4
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020731
 Time 16.10
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 71
 DS 0
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 d1 0.10000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.6539737 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 93.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.2520012 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6429588 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 F1P 240.000 ppm
 F1 24154.31 Hz
 F2P -10.000 ppm
 F2 -1006.43 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 629.01849 Hz/cm

6c

nmr400a c-13

Current Data Parameters
 NAME 44180-120-b-f3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011214
 Time 15.58
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 229
 DS 0
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.625137 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 93.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.252012 MHz

F2 - Processing parameters
 SI 32768
 SF 100.625137 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 F1P 220.000 ppm
 F1 22141.45 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 5.50000 ppm/cm
 HZCM 553.53625 Hz/cm

44307-92-0 11/01/01 J/CC/11
nmr400b h-1

Current Data Parameters
NAME 44307-92-8
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011121
Time 10.05
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 35.9
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700208 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

6d

44307-92-8 fraction 3; CDCl₃
nmr400b c-13 (h-1 decoupled)

Current Data Parameters
NAME 44307-92-8
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011121
Time 10.07
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl₃
NS 86
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24131.37 Hz
F2P -10.000 ppm
F2 -1005.48 Hz
PPMCH 6.25000 ppm/cm
HZCH 628.42120 Hz/cm

150.3
137.3
132.7
127.3
127.3
127.1
126.2
121.9

77.5
77.2
76.8
68.6

28.5
22.1
22.0

6d

6e

nmr400a h-

Current Data Parameters
NAME 44180-80-f3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010924
Time 15.06
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 114
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.000 ppm
F1 3602.25 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.22500 ppm/cm
HZCM 90.05627 Hz/cm

6e

Current Data Parameters
NAME 44180-80-ff2
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20011116
Time 11.51
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDC13
NS 107
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6539737 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 160.000 ppm
F1 16102.87 Hz
F2P 0.000 ppm
F2 0.00 Hz
FPMCH 4.00000 ppm/cm
HZCM 402.57184 Hz/cm

137.0
132.3
127.4
127.0
126.2
121.6

95.5

77.4
77.1
76.7
73.3

28.4
28.3

22.0
19.5

ppm 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

nmr400a h-1

Current Data Parameters
NAME 44180-98-f5
EXPNO
PROCNO

F2 - Acquisition Parameters
Date_ 20011030
Time 16.42
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS
SWH 6576.94 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 128
DN 76.000 umsec
DE 5.00 umsec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL =====
NUC1 13
P1 1.50 umsec
PL1 0.00 dB
SFO1 400.251101 MHz

F2 - Processing parameters
SI 32768
SF 400.251101 MHz
WDM
SSB
LB 1.00 Hz
GB
PC

1D NMR plot parameters
CX 4.00 cm
F1P 1.000 Hz
F1 1328.00 Hz
F2P 1.000 Hz
F2 10.00 Hz
PPMCM 0.25000 Hz/cm
HZCM 20.0000 Hz/cm

nmr400a c-13

89

nmr400a h-1

Current Data Parameters
NAME 44180-87-b-f1-
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011130
Time 11.26
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.260774 Hz
AQ 2.4904180 sec
RG 128
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
FIP 9.000 ppm
F1 3602.25 Hz
F2P 1.000 ppm
F2 400.25 Hz
PPMCH 0.20000 ppm/cm
HZCH 80.05001 Hz/cm

Current Data Parameters
NAME 44180-87-bf2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011203
Time 15.43
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 239
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

***** CHANNEL f1
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6259737 MHz

***** CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 160.000 ppm
F1 16102.88 Hz
F2P 12.000 ppm
F2 1207.72 Hz
PPHCH 3.70000 ppm/cm
H2CH 372.37894 Hz/cm

155.6

141.0
137.1

128.8
127.9
126.7
126.0

97.8

77.5
77.2
76.9

55.1

34.2
32.6

22.8

U

8b

nmr400a h-1

Current Data Parameters
 NAME 44180-88-E3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011005
 Time 15.27
 INSTRUM spect
 PROBRD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6578.947 Hz
 FIDRES 0.200774 Hz
 AQ 2.4904180 sec
 RG 114
 DW 76.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.50 usec
 PL1 -3.00 dB
 SFO1 400.2522014 MHz

F2 - Processing parameters
 SI 16384
 SF 400.2500593 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CK 40.00 cm
 FIP 9.000 ppm
 F1 3602.25 Hz
 F2P 1.000 ppm
 F2 400.25 Hz
 PPMCM 0.20000 ppm/cm
 HZCM 80.05001 Hz/cm

nmr400a h-1 decoupled c-13

Data Parameters
44180-89-b
2
1

Acquisition Parameters
20020801
16.16
spect
5 mm QNP 1H
zgpgc
65536
CDC13
254
0
26315.789 Hz
0.401547 Hz
1.2452340 sec
8192
19.000 usec
6.00 usec
300.0 K
0.10000000 sec
0.03000000 sec

==== CHANNEL f1 =====
13C
2.00 usec
0.00 dB
100.6539737 MHz

==== CHANNEL f2 =====
waltz16
1H
93.00 usec
120.00 dB
18.00 dB
400.2520012 MHz

Processing parameters
32768
100.6429588 MHz
EM
0
1.00 Hz
0
1.40

parameters

206.1

nmr400a h-1

8c

Current Data Parameters
NAME 44180-122-f2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011214
Time 16.02
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 40.3
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500533 MHz
WDW no
SSB 0
LB 0.40 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 8.000 ppm
F1 3202700 Hz
F2P 0.600 ppm
F2 0.000 Hz
PPMCM 0.20000 ppm/cm
HZCM 80.05611 Hz/cm

nmr400a c-13

Current Data Parameters
NAME 44180-122-f2
EXPNO 2
PROCNO 1

154.9

F2 - Acquisition Parameters
Date_ 20011214
Time 16.07
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 219
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6299737 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6299737 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D plot parameters
CX 40.00
F1P 160.000
F1 16102.87
F2P 0.000
F2 0.00
PPMC 4.00000
HZCM 402.57184

77.4
77.1
76.8

67.6

34.3

32.5

29.0

28.5

22.9

22.6

14.1

44307-98-6 fraction.3;CDC13
nmr400b h-1

Current Data Parameters
NAME 44307-98-6
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011205
Time 0.33
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 80.6
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.870208 MHz
NCN 0
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CH 40.00 cm
F1P 9.756 ppm
F1 3898.71 Hz
F2P -0.250 ppm
F2 -99.97 Hz
FFNMH 0.25020 ppm/cm
HSCN 99.96751 Hz/cm

44307-98-6 fraction 3;CDCl₃
nmr400b c-13 (h-1 decoupled)

ppm

Current Data Parameters
NAME 44307-98-6
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011206
Time 8.42
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl₃
NS 255
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24131.37 Hz
F2P -10.000 ppm
F2 -1005.48 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.42120 Hz/cm

44007-110 cyclohexanone methyl enol
ether, months later; CDC13
nmr400b h-1

Current Data Parameters
NAME 44307-118
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020628
Time 10.56
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 8
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

1009

44307-128-5 fraction 7;CDCl3
nmr400b h-1

10b

Current Data Parameters
NAME 44307-128-5
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020205
Time 15.15
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 16
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
DL 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
PL 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700208 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

nmr400b c-13 (h-1 decoupled)

ppm

Current Data Parameters
 NAME 44307-128-5
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020205
 Time 15.18
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgdc
 TD 65536
 SOLVENT CDCl3
 NS 118
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 3.00 usec
 PL1 5.00 dB
 SFO1 100.5584112 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 14.70 dB
 SFO2 399.8719994 MHz

F2 - Processing parameters
 SI 32768
 SF 100.5473900 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CK 40.00 cm
 P1P 240.000 ppm
 F1 24131.37 Hz
 F2P -10.000 ppm
 F2 -1005.48 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 628.42120 Hz/cm

154.7

93.5

77.4
77.1
76.8
 33.9
29.9
29.0
28.7
28.5
28.0
25.8
23.6
23.1
23.0
22.9
22.7
22.6
14.0

10b

ppm 220 200 180 160 140 120 100 80 60 40 20 0

nmr400a h-1

Current Data Parameters
NAME 44180-89-f1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020731
Time 15.13
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 18
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 16
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.250593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 8.000 ppm
F1 3202.00 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.20000 ppm/cm
HZCM 80.05001 Hz/cm

129

129

nmr400a h-1 decoupled c-13

Current Data Parameters
 NAME 44180-89-f1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020731
 Time 15.16
 INSTRUM spect
 PROBRD 5 mm QNP 1H
 PULPROG zgpgc
 TD 65536
 SOLVENT CDCl3
 NS 104
 DS 0
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

***** CHANNEL f1
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.6259737 MHz

***** CHANNEL f2 ---
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 93.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.2520012 MHz

F2 - Processing parameters
 S1 32768
 SF 100.6429588 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 F1P 240.000 ppm
 F1 24154.31 Hz
 F2P -10.000 ppm
 F2 -1006.43 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 629.01849 Hz/cm

12b

44307-125-8 fraction 10;CDCl₃
nmr400c h1

Current Data Parameters
NAME 44307-125-8
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020131
Time 16.34
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 8
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 32
DW 76.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SFO1 400.1322007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3901.27 Hz
F2P -0.250 ppm
F2 -100.03 Hz
PPMCH 0.25000 ppm/cm
HZCM 100.03250 Hz/cm

Dm

Current Data Parameters
 NAME 44307-125-8
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020131
 Time 16.36
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgdc
 TD 65536
 SOLVENT CDC13
 NS 87
 DS 0
 SWH 26246.719 Hz
 FIDRES 0.400493 Hz
 AQ 1.2485108 sec
 RG 8192
 DW 19.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

***** CHANNEL f1 *****
 MUC1 13C
 P1 2.50 usec
 PL1 0.00 dB
 SF01 100.6237964 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 MUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SF02 400.1322007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127796 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 F1P 240.000 ppm
 F1 24147.07 Hz
 F2P -10.000 ppm
 F2 -1006.12 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 628.82983 Hz/cm

161.4

12b

77.2
76.9
76.6
66.6
59.8

36.9
33.8
32.2
29.8
29.0
28.8
28.5
28.3
28.2
25.6
25.4
22.5
22.4
21.7
13.9

ppm 220 200 180 160 140 120 100 80 60 40 20 0

72454-77-1 enantiomer 2; CDC13
nmr400b h-1

Current Data Parameters
NAME 72454-77-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021009
Time 14.45
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 57
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
FIP 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

72454-77-1 enantiomer2 :CDCl3
nmr400b h-1 decoupled c-13

72454-95-1 Enantiomer 2;CDCl₃
nmr400b h-1

7

Current Data Parameters
NAME 72454-95-1
EXNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 26021009
Time 14.03
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.306774 Hz
AQ 2.4904180 sec
RG 57
CW 75.000 usec
CB 6.00 usec
TE 300.0 K
C1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
RF1 399.8724592 MHz

F1 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3996.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HSCN 99.96751 Hz/cm

72454-95-1 enantiomer 2;CDCl3
nmr400b h-1 decoupled c-13

Current Data Parameters
NAME 72454-95-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021009
Time 14.06
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 72
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

199.66

144.38

134.20
130.54
128.96
127.62
126.94

77.47
77.35
77.15
76.83
73.93

33.26
31.93
27.79

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24131.38 Hz
F2P -10.000 ppm
F2 -1005.47 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.42120 Hz/cm

ppm 220 200 180 160 140 120 100 80 60 40 20 0

44307-117a LC sample ;CDCl₃
nmr400b h-1

Current Data Parameters
NAME 44307-117a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020802
Time 16.14
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 90.5
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
FIP 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.37 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

44307-117a LC sample;CDCl₃
nmr400b h-1 decoupled c-13

Current Data Parameters
NAME 44307-117a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020802
Time 16.21
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl₃
NS 176
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz

===== CHANNEL f2 ---
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24131.38 Hz
F2P -10.000 ppm
F2 -1005.47 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.42120 Hz/cm

204.72

143.04
135.12
133.10
130.59
129.78
126.86

77.44
77.12
76.80
75.98

42.30

34.65
32.26

23.60

1.89

44307-122-2 awu D1=20: CDC13
nmr400b h-1

Current Data Parameters
NAME 44307-122-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020128
Time 8.40
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 40.3
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 20.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700208 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

ppm

Current Data Parameters
 NAME 44307-122-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20020125
 Time 15.19
 INSTRUM spect
 PROBED 5 mm QNP 1H
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 108
 DS 4
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

***** CHANNEL f1 *****

NUC1 13C
 P1 3.00 usec
 PL1 5.00 dB
 SFO1 100.5584112 MHz

***** CHANNEL f2 *****

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 14.70 dB
 SFO2 399.8719994 MHz

F2 - Processing parameters

SF 32768
 SF 100.5473900 MHz
 MDW RM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CK 40.00 cm
 F1P 240.000 ppm
 F1 24131.37 Hz
 F2P -10.000 ppm
 F2 -1005.48 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 628.42120 Hz/cm

44307-119-3 fraction 3; CDCl₃
nmr400b h-1

Current Data Parameters
NAME 44307-119-3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020130
Time 9.25
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SWH 6578.847 Hz
FIDRES 0.220774 Hz
AQ 2.4904160 sec
RG 32
CW 75.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1001000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
RFQ1 399.8724532 MHz

F2 - Processing parameters
SI 16384
SF 399.8700008 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CE 40.00 cm
F1P 9.750 ppm
F2P 3698.73 Hz
F3 -9.250 ppm
F2 -99.87 Hz
F2NCH 0.25000 ppm/cm
EQCN 99.96751 Hz/cm

44307-119-3 fraction 3;CDCl3
nmr400b c-13 (h-1 decoupled)

karady 72434-90-f2 cdcl3
nmr600 c-13

063-

karady 72434-88-3 cdcl3
nmr600 h-1

14b

Current Data Parameters
NAME kar00663
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021025
Time 13.19
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6477449 sec
RG 16
DM 40.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.50 usec
PL1 6.00 dB
SFO1 600.0337060 MHz

F2 - Processing parameters
SI 32768
SF 600.0300089 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
CY 22.00 cm
P1P 9.750 ppm
P1 5850.29 Hz
F2P -0.250 ppm
F2 -150.01 Hz
PPMCH 0.25000 ppm/cm
HZCH 150.00751 Hz/cm

karady 72434-88-3 cdcl3
nmr600 c-13

ppm

Current Data Parameters
NAME karco663
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021025
Time 14.59
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 3262
DS 4
SWH 40000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6384500 sec
RG 8192
DM 12.500 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.00 usec
PL1 0.00 dB
SFO1 150.8942005 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 83.00 usec
PL2 120.00 dB
PL12 28.00 dB
SFO2 600.0330002 MHz

F2 - Processing parameters
SI 65536
SF 150.8776666 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 36210.64 Hz
F2P -10.000 ppm
F2 -1508.77 Hz
PPMCM 6.25000 ppm/cm
HZCM 942.98541 Hz/cm

73626-20 + S-aug

R

16

nmr400a h-1

Current Data Parameters
NAME 73434-122-down
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030325
Time 17.19
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 203.2
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.252014 MHz

F2 - Processing parameters
SI 16384
SF 400.250593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
FLR 8.000 ppm
F1 3202.00 Hz
F2 3.000 ppm
F2 1200.75 Hz
PEMCM 0.12500 ppm
H2M 50.03128 Hz

15a

nmr400a h-1 decoupled c-13

Current Data Parameters
 NAME 73434-122-down
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030325
 Time 17.24
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgdc
 TD 65536
 SOLVENT CDCl3
 NS 447
 DS 0
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 DW 19.000 usec
 DE 5.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.629377 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 93.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.2520012 MHz

F2 - Processing parameters
 SI 32768
 SF 100.629588 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 FIP 240.000 ppm
 F1 24154.31 Hz
 F2P -10.000 ppm
 F2 -1006.43 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 629.01849 Hz/cm

187.4

169.5

161.3

136.5
 135.8
 129.0
 128.7
 127.6
 127.3
 122.1
 119.7
 117.8

82.2
 77.3
 77.0
 76.7
 69.8
 67.9

57.6

159

73434-123-2 S,S-hydroxychromanoneMPA
nmr400a h-1

Current Data Parameters
NAME 73434-123-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030325
Time 17.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6578.947 Hz
FIDRES 0.300774 Hz
AQ 2.4904180 sec
RG 71.8
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3902.44 Hz
F2P -0.250 ppm
F2 -100.06 Hz
PPHCH 0.25000 ppm/cm
HZCM 100.06252 Hz/cm

nmr400a h-1 decoupled c-13

Current Data Parameters
NAME 73434-123-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030325
Time 17.39
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 208
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6259737 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
FIP 240.000 ppm
F1 24154.31 Hz
F2P -10.000 ppm
F2 -1006.43 Hz
PPMCM 6.25000 ppm/cm
HZCM 629.01849 Hz/cm

73434-89-bb R,S-hydroxytetraloneMPA
nmr400a h-1

Current Data Parameters
NAME 73434-89-bb
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030324
Time 17.21
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 406.4
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
P1 3902.44 Hz
P2P -0.250 ppm
P2 -100.06 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.06252 Hz/cm

73434-89-bb ⁸,S-hydroxytetraloneMPA
nmr400a h-1 decoupled c-13

Current Data Parameters
NAME 73434-89-bb
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030324
Time 17.30
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpgc
TD 65536
SOLVENT CDCl3
NS 546
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6539737 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24154.31 Hz
F2P -10.000 ppm
F2 -1006.42 Hz
PPMCM 6.25000 ppm/cm
HZCM 629.01849 Hz/cm

143.0
136.2
134.0
131.4
128.7
128.6
127.9
127.3
127.0

82.3
77.3
77.0
76.7
75.1

57.6

28.8
27.7

16b

nmr400a h-1

Current Data Parameters
NAME 72434-92-up
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021025
Time 11.12
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 32
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 456.1
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 ***
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
ST 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 9.000 ppm
F1 3602.25 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.22500 ppm/cm
HZCM 90.05627 Hz/cm

nmr400a h-1 decoupled c-13