Supporting Information

Concise Asymmetric Synthesis of 6-Hydroxy - 3, 8-dioxabicyclo[3.2.1]octane Derivatives

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General Experimental. ^1H NMR and ^{13}C NMR were recorded on a Bruker AC-300 FT (^1H : 300 MHz, ^{13}C : 75.46 MHz), or Bruker AC-400 FT (^1H : 400 MHz, ^{13}C : 100 MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz respectively. IR spectra were recorded on a Perkin-Elmer, 2000FTIR. High resolution mass spectra were obtained on LC-TOF spectrometer(Micromass). HPLC was recorded on a Agilent 1100(Chiralcel OD-H column, UV detector). Acetonitrile was distilled from CaH_2 . Allylic bromide, crotyl bromide, and methallyl bromide were purchased from Aldrich and used as supplied. α -bromomethylstyrene¹ and 4-chloro- α -bromomethylstyrene^{1,2} were prepared according to literature. (R)-2,3-O-isopropylideneglyceraldehyde was prepared according to literature and immediately used without further purification.³ Other reagents and solvents were used as supplied.

Preparation of 2a-e. General procedure 1 mmol of the freshly prepared (R)- 2,3-O-isopropylideneglyceraldehyde, 1mmol SnCl_2 and 5ml distilled water were mixed together in flask, and stirred vigorously at room temperature. Then 2mmol of Zn powder was added. Five minutes later, 1.2mmol allylic substrate was slowly added. The reaction mixture was stirred for 30minutes at room temperature. The mixture was filtered, and the filtrate was extracted by EtOAc (4 $\times$ 10ml). The combined organic layers was dried (MgSO_4) and concentrated. Chromatography of the residue on silica gel gave **2** as a colorless oil. Yields were included in table 1.

(S)-1-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)but-3-en-1-ol (2a) ^1H NMR (CDCl_3 , 300MHz) δ = 5.85(m, 1H), 5.15(m, 2H), 4.03-3.93(m, 3H), 3.77(m, 1H), 2.35-2.17(m, 2H), 2.01(br s, 1H), 1.43(s, 3H), 1.36(s, 3H).

(S)-3-methyl-1-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)but-3-en-1-ol (2b) ^1H NMR (CDCl_3 , 300MHz) δ = 4.93(s, 1H), 4.86(s, 1H), 4.08-3.77(m, 4H), 2.37-2.11(m, 3H), 1.83(s, 3H), 1.48(s, 3H), 1.41(s, 3H). ^{13}C NMR (CDCl_3 , 75MHz) δ =141.90, 113.57, 109.36, 78.39, 68.86, 65.99, 41.70, 26.55, 25.24, 22.30. IR (film) ν = 3471, 3076, 2986, 2936, 1649, 1455, 1372, 1253, 1215, 1157, 1067 cm^{-1} . HRMS calc. $\text{C}_{10}\text{H}_{18}\text{O}_3$: 186.1256. Found: 186.1260.

(S)-3-phenyl-1-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)but-3-en-1-ol(2c) ^1H NMR (acetone- d_6 , 300MHz) δ = 7.48(dd, J = 8.5, 1.2 Hz, 2H), 7.36-7.26(m, 3H), 5.38(d, J =1.5Hz, 1H), 5.19(d, J =1Hz, 1H), 4.0-3.9(m, 3H), 3.60(m, 1H), 3.25(s, 1H), 3.01(dd, J =14.5, 3.2Hz, 1H), 2.47(dd, J =14.5, 9.1Hz, 1H), 1.33(s, 3H), 1.24(s, 1H). ^{13}C NMR (CDCl_3 , 75MHz) δ = 144.71, 140.18, 128.59, 127.85, 126.33, 115.43, 109.26,

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(2) Arnoldi, A.; Carzaniga, R.; Morini, G.; Merlini, L.; Farina G. *J. Agric. Food Chem.* **2000**, 48, 2547.

(3) Jackson, D. Y. *Synth. Commun.* **1988**, 18, 337.

78.34, 69.66, 65.56, 39.62, 26.68, 25.35. IR (film) ν =3482, 3082, 2986, 2933, 1626, 1574, 1495, 1454, 1371, 1257, 1215, 1156, 1063 cm^{-1} . HRMS calc. $\text{C}_{15}\text{H}_{20}\text{O}_3$: 248.1412. Found: 248.1416.

(S)-3-(4-chlorophenyl)-1-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)but-3-en-1-ol (2d) ¹H NMR (CDCl_3 , 300MHz) δ = 7.41-7.30(m, 4H), 5.45(s, 1H), 5.23(s, 1H), 4.06-3.95(m, 3H), 3.77-3.73(m, 1H), 2.88(dd, J =14.4, 0.9Hz, 1H), 2.54(dd, J =14.4, 9.0Hz, 1H), 1.87(br s, 1H), 1.45(s, 3H), 1.35(s, 3H). ¹³C NMR (CDCl_3 , 75MHz) δ = 143.61, 138.67, 133.65, 128.62, 127.57, 115.75, 109.23, 78.24, 69.69, 65.51, 39.43, 26.61, 25.23. IR (film) ν = 3468, 3085, 3036, 2987, 2934, 2891, 1735, 1626, 1594, 1493, 1454, 1371, 1257. HRMS calc. $\text{C}_{15}\text{H}_{19}\text{O}_3^{35}\text{Cl}$: 282.1023. Found: 282.1020.

(1R, 2R)-2-methyl-1-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)but-3-en-1-ol (2e) yield 36%. ¹H NMR (CDCl_3 , 300MHz) δ = 5.90-5.80(m, 1H), 5.15(s, 1H), 5.10(d, J =2.8Hz, 1H), 4.10-3.90(m, 3H), 3.60(t, J =5.2, 1H), 2.40(m, 1H), 1.87(s, 1H), 1.42(s, 3H), 1.36(s, 3H), 1.08(d, J =7.0Hz, 3H). ¹³C NMR (CDCl_3 , 75MHz) δ = 140.40, 116.16, 108.78, 77.33, 74.99, 65.73, 40.68, 26.66, 25.43, 16.67. IR (film) ν = 3480, 3077, 2985, 2934, 2889, 1640, 1457, 1419, 1372. HRMS calc. $\text{C}_{10}\text{H}_{18}\text{O}_3$: 186.1256. Found: 186.1264.

Preparation of (S)-1-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)but-3-enyl benzoate (2a')
1mmol of **2a** was dissolved in 4ml pyridine in a flask with stirrer. 1.2mmol of benzoyl chloride was dissolved in 2ml CH_2Cl_2 , and added to the flask via addition funnel. After addition, the mixture was stirred for 2 hours, then removed to a separatory funnel. 30 ml ether was added, and then organic layer was washed with distilled water (4 $\times$ 20ml), dried(MgSO_4), concentrated. The residue was purified by chromatography on silica gel to give 0.25g **2a'** as a light yellow oil(90%).

2a' ¹H NMR (CDCl_3 , 300MHz) δ = 8.05-7.41(m, 5H), 5.84(m, 1H), 5.27(m, 1H), 5.08(m, 2H), 4.28(ddd, J =6.06, 6.51, 6.18Hz, 1H), 4.08(dd, J =8.37, 6.51Hz, 1H), 3.94(dd, J = 8.37, 6.18Hz, 1H), 2.56-2.44(m, 2H), 1.46(s, 3H), 1.37(s, 3H). ¹³C NMR (CDCl_3 , 75MHz) δ = 165.65, 134.36, 133.09, 129.94, 129.54, 128.23, 118.14, 109.46, 76.12, 73.67, 65.87, 35.26, 26.30, 25.09. IR (film) ν = 3073, 2985, 2935, 1788,⁴ 1721, 1643, 1600, 1491, 1452, 1371, 1274, 1213, 1174 cm^{-1} . HRMS calc. $\text{C}_{16}\text{H}_{20}\text{O}_4$: 276.1366. Found: 276.1257.

Preparation of 3a'-e. General procedure 1mmol **2** and 4ml 1M HCl were mixed together in a flask, and stirred at room temperature for two hours. Then the water was evaporated under reduced pressure. 1ml Saturated Na_2CO_3 and the water was again evaporated under reduced pressure. 10ml of EtOAc was added to dissolve the product. The solid was filtered, and washed with 20 ml EtOAc. The filtrate was dried (NaSO_4), evaporated to give **3a'-e** as white to pale yellow solid, which was pure enough to be used without further purification.

(4) Absorption at 1788 cm^{-1} is due to benzoic anhydride

Preparation of 4a'-e. General procedure 1mmol **3** was dissolved in 5ml anhydrous acetonitrile and the solution was cooled to 0 $^{\circ}\text{C}$ by ice bath and stirred vigorously. 0.7g NaHCO_3 was quickly added into the flask in one portion. 20ml saturated iodine solution in anhydrous acetonitrile was slowly added to the system via a addition funnel with pressure-equalization arm over 8 hours. Then the ice bath was removed, and 1.2g NaHCO_3 and 2ml distilled water was added to the reaction system. Solid NaHSO_4 was added in small portions until the total disappearance of the rest iodine. The mixture was extracted by EtOAc (3 $\times$ 20ml). The combined organic layers was dried (MgSO_4) in the dark, and concentrated. The residue was purified by chromatography on silica gel to give **4a'-e** (yield 64-86%).

(2R, 3S, 5R)-tetrahydro-2-(hydroxymethyl)-5-(iodomethyl)-furan-3-yl benzoate (4a') yield 72%. ^1H NMR (CDCl_3 , 300MHz) δ = 8.02(d, J = 7.1Hz, 2H), 7.58(t, J = 7.3Hz, 1H), 7.45(m, 2H), 5.43(d, J =6.2Hz, 1H), 4.22(m, 1H), 4.09(m, 1H), 3.87(dd, J =11.9, 3.6Hz, 1H), 3.82(dd, J =11.9, 4.0Hz, 1H), 3.46(dd, J =10.5, 5.4Hz, 1H), 3.38(dd, J =10.5, 4.2Hz, 1H), 2.28(dd, J =13.8, 5.13Hz, 1H), 2.25(br s, 1H), 2.06(ddd, J =13.8, 6.2, 2.0Hz, 1H). ^{13}C NMR (CDCl_3 , 75MHz) δ = 166.21, 133.58, 133.38, 129.66, 128.50, 86.05, 77.16, 63.27, 39.43, 9.6. IR (film) ν = 3415, 2961, 2919, 2857, 1715, 1453, 1280 cm^{-1} . HRMS calc. $\text{C}_{13}\text{H}_{15}\text{O}_4\text{I}$: 362.0019. Found: 362.0027.

(2R, 3S, 5R)-tetrahydro-2-(hydroxymethyl)-5-(iodomethyl)-5-methylfuran-3-ol (4b) yield 64%. ^1H NMR (CDCl_3 , 300MHz) δ = 4.35(ddd, J =4.4, 6.4, 1.7Hz, 1H), 3.91(d, J =1.7Hz, 1H), 3.77(d, J =11.6 Hz, 1H), 3.67(d, J =11.6 Hz, 1H), 3.45(s, 2H), 3.19(br s, 2H), 2.18-2.11(m, 2H), 1.42(s, 3H). ^{13}C NMR (CDCl_3 , 75MHz) δ = 85.69, 81.60, 72.81, 62.52, 44.87, 27.55, 17.51. IR (film) ν =3396, 2972, 2931, 2876, 1448, 1420, 1376, 1293, 1220, 1188, 1154, 1085, 1044 cm^{-1} . HRMS calc. $\text{C}_7\text{H}_{13}\text{O}_3\text{I}$: 271.9913 Found: 271.9901.

(2R, 3S, 5R)-tetrahydro-2-(hydroxymethyl)-5-(iodomethyl)-5-phenylfuran-3-ol (4c) yield 86%. ^1H NMR (acetone- d_6 , 300MHz) δ =7.42(d, J =7.7Hz, 2H), 7.29-7.16(m, 3H), 4.18(dd, J =4.6, 6.4Hz, 1H), 3.99(t, J =4.7Hz, 1H), 3.87(d, J =10.3Hz, 1H), 3.62(d, J =10.3Hz, 1H), 3.50(d, J =4.7Hz, 2H), 2.95(s, 2H), 2.46(dd, J =13.2, 4.6Hz, 1H), 2.36(dd, J =13.2, 6.4Hz, 1H). ^{13}C NMR (acetone- d_6 , 75MHz) δ =145.89, 128.37, 127.49, 125.86, 87.59, 84.91, 72.92, 63.10, 45.86, 20.43. IR (film) ν = 3370, 2939, 1662, 1601, 1540, 1493, 1448, 1412, 1313, 1190, 1091, 1026 cm^{-1} . HRMS calc. $\text{C}_{12}\text{H}_{15}\text{O}_3\text{I}$: 334.0066. Found: 334.0080.

(2R, 3S, 5R)-5-(4-chlorophenyl)-tetrahydro-2-(hydroxymethyl)-5-(iodomethyl)furan-3-ol (4d) yield 84%. ^1H NMR (CDCl_3 , 300MHz) δ =7.35-7.29(m, 4H), 4.33(d, J =1.7 Hz, 1H), 4.19(ddd, J = 1.7, 4.3, 5.7Hz, 1H), 3.75(d, J = 10.4 Hz, 1H), 3.73(s, 2H), 3.63(d, J =10.4 Hz, 1H), 2.65(dd, J =12.4, 4.3 Hz, 1H), 2.57(dd, J = 12.4, 5.7 Hz, 1H), 2.49(s, 2H). ^{13}C NMR (CDCl_3 , 75MHz) δ =142.84, 133.62, 128.56, 126.78, 86.41, 84.52, 73.10, 63.16, 45.50, 18.81. IR (film) ν =3384, 2929, 1704, 1490, 1401, 1361, 1191, 1092, 1046, 1014 cm^{-1} . HRMS calc. $\text{C}_{12}\text{H}_{14}\text{O}_3\text{I}^{35}\text{Cl}$: 367.9676. Found: 367.9654.

(2R, 3S, 4S, 5R)-tetrahydro-2-(hydroxymethyl)-5-(iodomethyl)-4-methylfuran-3-ol (4e) yield 77%. ^1H NMR (CDCl_3 , 300MHz) δ = 4.32(dd, J = 5.3, 5.8Hz, 1H), 3.97(m, 1H), 3.8-3.6(m, 3H), 3.31(dd, J = 11.0, 5.8Hz, 1H), 3.19(dd, J =11.0, 5.3Hz, 1H), 2.44(m, 1H), 1.87(s, 2H), 1.05(d, J = 7.3Hz, 3H). ^{13}C NMR (CDCl_3 , 75MHz) δ = 84.16, 81.99, 74.59, 63.20, 40.97, 7.10, 5.49. IR (film) ν =3395, 2938, 2927, 2905, 1456, 1456, 1097, 1046 cm^{-1} . HRMS calc. $\text{C}_7\text{H}_{13}\text{O}_3\text{I}$: 271.9909. Found: 271.9925.

Preparation of 5a'-e. General procedure 1 mmol **4** was dissolved in 5 ml MeOH, then 0.35g AgNO₃ , 0.5g K₂CO₃ was added . The system was stirred at room temperature for 2 hours. The solvent was evaporated under reduced pressure and 20 ml EtOAc was added to dissolve the product. The precipitate was filtered out, and the filtrate was concentrated and purified by chromatography on silica gel to give **5**.

(1R, 5R, 6S)-6-benzoyloxy-3,8-dioxabicyclo[3.2.1]octane (5a') yield 79%. ¹H NMR (CDCl₃, 400MHz) δ=8.04(d, *J*= 7.9Hz, 2H), 7.56(t, *J*= 7.5Hz, 1H), 7.45(m, 2H), 5.66(dd, *J*= 2.8, 7.7Hz, 1H), 4.44(d, *J*= 7.4Hz, 1H), 4.23(s, 1H), 3.84-3.75(m, 3H), 3.46(d, *J*= 11.3Hz, 1H), 2.61(dd, *J*= 7.7, 13.3Hz, 1H), 2.21(ddd, *J*=2.8, 7.4, 13.3Hz, 1H). ¹³C NMR (CDCl₃, 75MHz) δ=166.59, 133.11, 129.95, 129.64, 128.35, 80.45, 77.48, 76.33, 70.72, 69.11, 36.43. IR (film) ν=2960, 2922, 2856, 1715, 1451, 1278, 1211, 1177, 1115, 1073 cm⁻¹. HRMS calc.C₁₃H₁₄O₄: 234.0895. Found: 234.0878. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (90:10 Hexanes: isopropanol, 0.5 ml/min, 254 nm); major enantiomer t_r = 13.7min, minor enantiomer t_r = 15.2min; 95% ee. The result was confirmed by comparison with that of (1S, 5S, 6R)-6-benzoyloxy-3,8-dioxabicyclo[3.2.1]octane prepared from (S)-2,3-O-isopropylidene glyceraldehydes.⁵

(1R, 5R, 6S)-6-hydroxyl-1-methyl-3,8-dioxabicyclo[3.2.1] octane (5b) yield 82%. ¹H NMR (CDCl₃, 400MHz) δ= 3.83(d, *J*= 1.7 Hz, 1H), 3.67(t, *J*= 4.3Hz, 1H), 3.60(d, *J*=10.9Hz, 1H), 3.41(d, *J*= 10.9Hz, 1H), 3.28(d, *J*= 4.3Hz, 2H), 2.21-2.09(m, 3H), 1.21(s, 3H). ¹³C NMR (CDCl₃, 75MHz) δ=81.69, 73.25, 66.17, 62.78, 49.25, 42.28, 20.88. IR (film) ν=3323, 2958, 2910, 2864, 2831, 2466, 1630, 1467, 1446, 1381, 1341, 1284, 1053 cm⁻¹. HRMS calc.C₇H₁₂O₃: 144.0789. Found: 144.0799. Product was derivatized with benzoyl chloride before the enantiomeric excess was determined. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (90:10 Hexanes: isopropanol, 0.5 ml/min, 254 nm); major enantiomer t_r = 14.1min, minor enantiomer t_r = 15.7min; 97% ee. The result was confirmed by comparison with that of (1S, 5S, 6R)-6-hydroxyl-1-methyl-3,8-dioxabicyclo[3.2.1]octane prepared from (S)-2,3-O-isopropylidene glyceraldehydes.

(1R, 5R, 6S)-6-hydroxyl-1-phenyl-3,8-dioxabicyclo[3.2.1]octane (5c) yield 91%. ¹H NMR (CDCl₃, 300MHz) δ=7.46-7.33(m, 5H), 4.53(d, *J*=2.2Hz, 1H), 4.21(t, *J*=5.5Hz, 1H), 4.04(d, *J*=7.8Hz, 1H), 3.89(d, *J*=7.8Hz, 1H), 3.48(d, *J*=5.5Hz, 2H), 2.40(s, 1H), 2.18(dd, *J*=2.2,10.2Hz, 1H), 2.08(d, *J*=10.2Hz, 1H). ¹³C NMR (CDCl₃, 75MHz) δ=135.61, 128.57, 128.43, 126.26, 87.50, 84.86, 79.49, 77.80, 63.103, 38.99. IR (film) ν=3434, 3061, 3032, 3003, 2938, 1450, 1449, 1339, 1308, 1198, 1084, 1040, 988, 934 cm⁻¹. HRMS calc.C₁₂H₁₄O₃: 207.1021. Found 207.1006. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (90:10 Hexanes: isopropanol, 0.5 ml/min, 254 nm); major enantiomer t_r = 21.6 min, minor enantiomer t_r = 26.4min; 95% ee. The result was confirmed by comparison with that of (1S, 5S, 6R)-6-hydroxyl-1-phenyl-3,8-dioxabicyclo [3.2.1]octane prepared from (S)-2,3-O-isopropylidene glyceraldehydes.

(1R, 5R, 6S)-1-(4-chlorophenyl)-6-hydroxyl-3,8-dioxabicyclo[3.2.1]octane(5d) yield 87%. ^1H NMR (CDCl_3 , 400MHz) δ =7.40-7.33(m, 4H), 4.56(d, J = 2.3Hz, 1H), 4.23(t, J =5.5Hz, 1H), 4.01(d, J = 7.9Hz, 1H), 3.87(d, J =7.9Hz, 1H), 3.52(d, J = 5.5Hz, 2H), 2.18(dd, J =2.3, 10.1Hz, 1H), 2.07(d, J =10.1Hz, 1H), 2.2-2.0 (br s, 1H). ^{13}C NMR (CDCl_3 , 75MHz) δ =134.29, 134.20, 128.79, 127.68, 87.04, 84.90, 79.58, 77.78, 63.12, 39.21. IR (film) ν = 3412, 3003, 2945, 2879, 1722, 1658, 1602, 1496, 1402, 1308, 1198, 1092, 1040, 1014, 988 cm^{-1} . HRMS calc. $\text{C}_{12}\text{H}_{13}\text{O}_3^{35}\text{Cl}$: 240.0553. Found: 240.0559. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (90:10 Hexanes: isopropanol, 0.5 ml/min, 254 nm); major enantiomer t_r = 19.2min, minor enantiomer t_r = 24.2min. >99% ee. The result was confirmed by comparison with that of (1S, 5S, 6R)-1-(4-chlorophenyl)-6-hydroxyl-3,8-dioxabicyclo[3.2.1]octane prepared from (S)-2,3-O-isopropylidene glycerolaldehydes.

(1R, 5R, 6R, 7R)-6-hydroxyl-7-methyl-3,8-dioxabicyclo[3.2.1]octane (5e) yield 84%. ^1H NMR (CDCl_3 , 400MHz) δ =4.15(d, J =2.3Hz, 1H), 4.02(s, 1H), 3.98(t, J =5.7Hz, 1H), 3.80(s, 2H), 3.45(d, J =5.7Hz, 2H), 2.5(br s, 1H), 2.22(qd, J =6.7, 2.3Hz, 1H), 0.99(d, J = 6.7Hz, 3H). ^{13}C NMR (CDCl_3 , 75MHz) δ =85.06, 80.52, 79.59, 70.99, 52.98, 39.24, 9.39. IR (film) ν =3430, 2936, 2883, 1457, 1383, 1289, 1203, 1170, 1123, 1044, 1001, 885 cm^{-1} . HRMS calc. $\text{C}_7\text{H}_{12}\text{O}_3$: 144.0789. Found M: 144.0786. Product was derivatized with benzoyl chloride before the enantiomeric excess was determined. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (90:10 Hexanes: isopropanol, 0.5 ml/min, 254 nm); major enantiomer t_r = 14.5min, minor enantiomer t_r = 17.5min; 98% ee. The result was confirmed by comparison with that of (1S, 5S, 6S, 7S)-6-hydroxyl-7-methyl-3,8-dioxabicyclo[3.2.1]octane prepared from (S)-2,3-O-isopropylidene glycerolaldehydes.

H-H COSY of 5a'

0.0005

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MCNPR	0.0150000 sec
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ID NMR plot parameters	
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RG          11491.4
DM          30.200 usec
DE          1.000 usec
TE          300.2 K
D1          2.0000000 sec
d11         0.0300000 sec
MCREST     0.0000000 sec
MCMRG      0.0150000 sec

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PL1        -6.00 dB
SF01       75.475588 MHz

===== CHANNEL f2 =====
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PL2        -3.00 dB
PL12       16.00 dB
SF02       300.1315007 MHz

F2 - Processing parameters
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PC          1.40

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CY         10.00 cm
F1P        213.235 ppm
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F2P        -6.148 ppm
F2         -463.96 Hz
NUC1NM     9.97193 ppm/cm
NUC2NM     752.55872 Hz/cm
  
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Current Data Parameters
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PROCNO 1

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TO 65536
SOLVENT CDCl3
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DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895986 sec
RG 80.5
DM 62.400 usec
DE 6.00 usec
TE 300.0 K
1E 2.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 8.10 usec
PL1 -3.00 dB
SFO1 400.1326009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300054 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 3
PC 1.00

1D NMR plot parameters
CY 24.00 cm
CX 0.00 cm
FIDP 8.000 ppm
F1 3201.04 Hz
F2 -0.500 ppm
F3 -200.07 Hz
F4NRM 0.35417 ppm/cm
FZCM 141.71272 Hz/cm

C NMR of 5d

Current Data Parameters
NAME pc1040219
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040219
Time 17:25
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 17281
DS 2
SWH 16556.291 Hz
FIDRES 0.505258 Hz
AQ 0.9896436 sec
RG 8192
DM 30.200 usec
DE 1.500 usec
TE 300.2 K
D1 2.0000000 sec
d11 0.0300000 sec
MCREST 0.0000000 sec
MCPRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.40 usec
PL1 -6.00 dB
SF01 75.475598 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 120.00 dB
PL12 16.00 dB
SF02 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677425 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
CY 0.00 cm
F1P 201.387 ppm
F1 15198.20 Hz
F2P 1.798 ppm
F2 135.71 Hz
PCHN 9.07220 ppm/cm
HZCM 684.65863 Hz/cm

Current Data Parameters
NAME pcf040330
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040406
Time 16:13
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8012.620 Hz
FIDRES 0.122266 Hz
AQ 4.0895596 sec
RG 45.3
DM 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.10 usec
PL1 -3.00 dB
SF01 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300064 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 24.00 cm
CY 0.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.07 Hz
OPNOM 0.39563 ppm/cm
HZCM 158.38480 Hz/cm

H NMR of 5e

Current Data Parameters
NAME pcr040330
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040606
Time 15:14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 505
DS 4
SWH 30303.031 Hz
FIDRES 0.924775 Hz
AQ 0.5407385 sec
RG 16384
DM 16.500 usec
DE 30.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.50 usec
PL1 -1.00 dB
SF01 100.622678 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 120.00 dB
PL12 17.00 dB
SF02 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6127698 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 2.00

1D IMF plot parameters
CX 24.00 cm
CY 0.00 cm
ZP 200.000 ppm
F1 20122.55 Hz
F2 -5.000 ppm
F2 -503.06 Hz
SFOH 8.54167 ppm/cm
ZCM 859.40076 Hz/cm

C NMR of 5e