Supporting Information

Highly Diastereoselective *Anti*-Aldol Reactions Utilizing Titanium Enolate of *Cis*-2-Arylsulfonamido-1-acenaphthenyl Propionate

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General Information

All melting points were recorded on a Thomas-Hoover melting point apparatus and are uncorrected. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker Avance 400 and 500 spectrometers. IR spectra were recorded on a Mattson Genesis II FT-IR spectrometer. Optical rotations were recorded on a Perkin-Elmer 341 or an Autopol III automatic polarimeter. Anhydrous solvents were obtained as follow: THF and diethyl ether by distillation from sodium and benzophenone; pyridine, dichloromethane and *N,N*-diisopropylethylamine from CaH_2 . All other solvents were reagent grade. Aldehydes were distilled before using. All moisture sensitive reactions including aldol reactions were carried out in a flame dried flask under nitrogen atmosphere. Column chromatography was performed with Whatman 240-400 mesh silica gel under low pressure of 3-5 psi. TLC was carried out with E. Merck silica gel 60-F-254 plates.

Preparation of *cis*-2-amino-1-acenaphthenol **9** and *ent*-**9**

($\pm$) *N*-2-Azido-1-acenaphthenol (**2**). To a stirred solution of **1** (5.26 g, 80% purity, 27.7 mmol) in DMSO (100 mL) were added H_2O (1 mL) and NBS (9.86 g, 55 mmol) at 0 °C. The resulting solution was warmed to 23 °C and stirred for 0.5 h before being poured into cold H_2O . The mixture was extracted twice with Et_2O . The combined organic layers were washed with H_2O and concentrated to give an orange residue which was purified by short path silica gel chromatography to provide practically pure bromohydrin. This bromohydrin was dissolved in Et_2O (100 mL) and the resulting mixture was treated with powdered NaOH (2.5 g, 62.5 mmol) at 23 °C for 3 h and quenched by addition of H_2O (40 mL). The mixture was separated and the aqueous layer was extracted twice with Et_2O . The combined organic layers were washed with brine, dried over MgSO_4 and evaporated to give 3.7 g of epoxide as a brown solid which was treated with NaN_3 (2.25 g, 34.7 mmol) in the presense of NH_4Cl (1.85 g, 34.7 mmol) in 80% aqueous EtOH (70 mL) at 60 °C for 3 h. The reaction mixture was cooled to ambient temperature and diluted with cold H_2O (50 mL). The mixture was extracted twice with EtOAc, dried over MgSO_4 and concentrated to give 4.6 g (78%) of analytically pure **2** as a brown oil.

($\pm$) *N*-Boc-2-amino-1-acenaphthenol (**3**). To a stirred solution of racemic azido alcohol **2** (4.32 g, 20.5 mmol) in EtOAc (50 mL) were added $(\text{Boc})_2\text{O}$ (4.95 g, 22.7 mmol) and 10 % Pd-C (400 mg) at 23 °C. The resulting mixture was hydrogenated under a H_2 atmosphere for 40 h. The reaction mixture was filtered through a Celite pad and rinsed

thoroughly with EtOAc. Evaporation of the solvents gave a solid residue which was recrystallized from EtOAc/hexane to give 4.33 g (80%) of **3** as a white solid, mp 140-142 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.75 (d, *J* = 8.2 Hz, 2H), 7.60-7.52 (m, 3H), 7.36 (d, *J* = 6.8 Hz, 1H), 5.54 (s, 1H), 5.28 (s, 1H), 4.04 (br s, 1H), 1.53 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 157.6, 142.2, 139.9, 136.7, 131.4, 129.1, 128.4, 125.3, 125.0, 121.1, 119.5, 84.8, 81.0, 66.4, 28.8; FT-IR (film, NaCl) 3404, 3329, 1689 cm⁻¹. Anal. Calcd for C₁₇H₁₉NO₃: C, 71.56; H, 6.71; N, 4.91. Found: C, 71.17; H, 7.15; N, 4.60.

Enzymatic Resolution of Racemic *trans*-*N*-Boc-2-amino-1-acenaphthenol (3**).** To a flask equipped with a reflux condenser were added **3** (4.33 g, 15.3 mmol), vinyl acetate (30 mL) and DME (30 mL). The resulting mixture was treated with immobilized Amano lipase on Celite (6.5 g, 20% by weight of Lipase PS 30) at 37 °C for 40 h. After cooling to room temperature, the reaction mixture was filtered through Celite and the filter cake was rinsed with EtOAc. Removal of the solvents under reduced pressure provided a residue which was chromatographed over silica gel (5% EtOAc/CH₂Cl₂) to afford 2.42 g (49%) of **5** as a white solid and 2.08 g (48%) of **4** as a white solid.

(1*R*, 2*R*)-*N*-Boc-2-amino-1-acetoxy-acenaphthene (5**).** Mp = 133-135 °C; [α]_D²³ - 142.0 (*c* 0.51, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.78 (m, 2H), 7.56-7.49 (m, 4H), 6.48 (s, 1H), 5.58 (s, 1H), 5.06 (s, 1H), 2.16 (s, 3H), 1.49 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 171.4, 155.9, 140.9, 139.7, 137.3, 131.2, 128.9, 128.8, 125.9, 125.3, 122.7, 120.9, 83.3, 80.5, 61.8, 28.8, 21.6; FT-IR (film, NaCl) 3441, 1736, 1712 cm⁻¹. Anal. Calcd for C₁₉H₂₁NO₄: C, 69.71; H, 6.47; N, 4.28. Found: C, 69.90; H, 6.46; N, 4.25.

(1*S*, 2*S*)-*N*-Boc-2-amido-1-acenaphthenol (4**).** Mp = 151-152 °C; [α]_D²³ - 153.8 (*c* 0.34, CHCl₃); Anal. Calcd for C₁₇H₁₉NO₃: C, 71.56; H, 6.71; N, 4.91. Found: C, 71.21; H, 6.62; N, 4.88. Spectral data were identical to those of **3**.

(1*R*, 2*R*)-*N*-Boc-2-amino-1-acenaphthenol (6**).** To a stirred solution of **5** (2.26 g, 6.89 mmol) in MeOH (40 mL) and H₂O (30 mL) was added triethylamine (9.7 mL, 68.9 mmol). The resulting solution was stirred at 23 °C for 27 h. After this period, the reaction mixture was concentrated under reduced pressure. The residue was diluted with 1N HCl and CH₂Cl₂, and the layers were separated. The organic extract was washed with aqueous NaHCO₃ and brine, dried over Na₂SO₄ and concentrated to furnish 1.95 g (99%) of **6** as a white solid. Mp = 151-152 °C; [α]_D²³ + 144.5.8 (*c* 0.67, CHCl₃); Anal. Calcd for C₁₇H₁₉NO₃: C, 71.56; H, 6.71; N, 4.91. Found: C, 71.70; H, 6.95; N, 4.94. Spectral data were identical to those of **4**.

(1*S*, 2*R*)-Acenaphtheno-1,3-oxazolidin-2-one (7**).** To a stirred solution of **6** (3.51 g, 12.3 mmol) in CH₂Cl₂ (60 mL) were added methanesulfonyl chloride (1.4 mL, 24.6 mmol) and triethylamine (5.2 mL 36.9 mmol) at 0 °C under a stream of N₂. The resulting solution was warmed to 23 °C and stirred for 4 h at the same temperature. The reaction mixture was quenched by addition of H₂O and the layers were separated. The organic layer was washed with brine, dried over Na₂SO₄ and evaporated to provide a yellow solid residue. Purification by silica gel chromatography (15% EtOAc/CH₂Cl₂) furnished 2.39 g of **7** (92%) as a white solid. Mp = 232-233 °C; [α]_D²³ - 127.1 (*c* 0.52, CHCl₃); ¹H NMR

(400 MHz, DMSO- d_6) δ 8.52 (s, 1H), 7.92 (d, J = 7.2 Hz, 1H), 7.86 (d, J = 8.2 Hz, 1H), 7.67-7.59 (m, 3H), 7.48 (d, J = 6.8 Hz, 1H), 6.33 (d, J = 7.2 Hz, 1H), 5.67 (d, J = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 158.9, 143.0, 140.6, 137.6, 132.0, 129.4, 126.7, 125.6, 123.1, 121.7, 81.4, 59.3; FT-IR (film, NaCl) 3225, 1759, 1752, 1696 cm^{-1} . Anal. Calcd for $\text{C}_{13}\text{H}_9\text{NO}_2$: C, 73.92; H, 4.29; N, 6.63. Found: C, 73.85; H, 4.39; N, 6.62.

(1R, 2S)-Acenaphtheno-1,3-oxazolidin-2-one (8). The procedure described for **7** was used for **4** (1.09 g, 6.7 mmol) to afford 1.21 g of **8** (86 %) as a white solid. Mp = 230-231 °C; $[\alpha]_D^{23} + 124.2$ (c 0.52, CHCl_3). Anal. Calcd for $\text{C}_{13}\text{H}_9\text{NO}_2$: C, 73.92; H, 4.29; N, 6.63. Found: C, 73.93; H, 4.39; N, 6.63. Other spectroscopic data were identical with those of **7**.

(1S, 2R)-2-amino-1-acenaphthenol (9). A solution of **7** (2.11 g, 10 mmol) in 50% aqueous MeOH (100 mL) was treated with NaOH (1.6 g, 40 mmol) at 80 °C for 18 h. After removal of the volatile solvent on a rotary evaporator, the mixture was extracted three times with CHCl_3 . The combined organic layers were dried over Na_2SO_4 and concentrated until a suspension was formed. To this suspension was added hexane and the resulting solution was cooled to 0 °C and filtered. The filter cake was rinsed with hexane and dried under vacuum to give 1.60 g (87%) of **9** as a pale yellow solid. Mp = 152-153 °C; $[\alpha]_D^{23} + 29.0$. (c 0.61, MeOH); ^1H NMR (400 MHz, DMSO- d_6) δ 7.74 (d, J = 8.1 Hz, 1H), 7.70 (d, J = 8.1 Hz, 1H) 7.54-7.45 (m, 4H), 5.41 (br s, 2H) 5.17 (d, J = 6.2 Hz, 1H), 4.61 (d, J = 6.2 Hz, 1H), 2.00 (s, 1H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 147.2, 145.8, 136.2, 130.9, 128.6, 128.5, 124.4, 123.7, 121.4, 120.7, 73.1, 58.0.

(1R, 2S)-2-amino-1-acenaphthenol (ent-9). The procedure used for **9** was employed for **8** (634 mg, 3 mmol) to deliver 472 mg (85 %) of **ent-9**, mp = 152-153 °C; $[\alpha]_D^{23} - 27$ (c 0.55, MeOH). The ^1H NMR and ^{13}C NMR spectra were identical with **9**.

Preparation of (1S, 2R)-N-(2-Hydroxy-acenaphthen-1-yl)-4-methyl-benzenesulfonamide (10). To a stirred suspension of **9** (1.48 g, 8 mmol), DMAP (49 mg, 0.4 mmol), and *p*-toluenesulfonyl chloride (1.51 g, 8 mmol) in CH_2Cl_2 (50 mL) and CH_3CN (10 mL) at 0 °C was added triethylamine (1.7 mL, 16 mmol). The resulting solution was stirred at the same temperature for 4 h and quenched by addition of 2N HCl. The layers were separated and the organic layer was washed with H_2O and brine, dried over Na_2SO_4 , and concentrated to provide a residue, which afforded 2.1 g (77 %) of **10** upon recrystallization from CH_2Cl_2 /hexane. Mp = 198-199 °C; $[\alpha]_D^{23} - 19.0$ (c 0.2, CHCl_3); ^1H NMR (500 MHz, $\text{CDCl}_3 + \text{CD}_3\text{OD}$) δ 7.87 (d, J = 8.3 Hz, 2H), 7.69 (d, J = 8.3 Hz, 1H), 7.64 (d, J = 8.3 Hz, 1H), 7.49-7.40 (m, 3H), 7.33 (d, J = 8.1 Hz, 2H), 7.08 (d, J = 7.0 Hz, 1H), 5.24 (d, J = 6.2 Hz, 1H), 5.05 (d, J = 6.2 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (125 MHz, $\text{CDCl}_3 + \text{CD}_3\text{OD}$) δ 144.2, 142.3, 141.0, 137.9, 136.1, 131.2, 130.2, 128.7, 128.6, 127.7, 125.6, 124.9, 121.8, 121.2, 72.5, 59.2, 21.9; FT-IR (film, NaCl) 3305, 1327, 1149 cm^{-1} .

Preparation of propionate ester (11). To a stirred solution of **10** (1.98 g, 5.84 mmol) in CH_2Cl_2 (44 mL) at 0 °C, was added propionyl chloride (0.61 mL 7 mmol) and pyridine

(0.81 mL, 10 mmol). The resulting mixture was stirred at 0 °C for 2 h and the reaction was quenched with 2N HCl. The layers were separated and the organic layer was washed with aqueous NaHCO₃ and brine, and concentrated to give a residue which was chromatographed over silica gel and triturated in hexane at 0 °C to furnish 2.20 g (95%) of **11** as a white solid. Mp = 148-149 °C; $[\alpha]_D^{23} + 195.0$ (c 0.6, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.87 (d, *J* = 8.2 Hz, 2H), 7.77 (d, *J* = 7.0 Hz, 1H), 7.73 (d, *J* = 8.2 Hz, 1H), 7.52-7.49 (m, 3H), 7.39-7.36 (m, 3H), 6.26 (d, *J* = 6.5 Hz, 1H), 5.47 (dd, *J* = 10.6, 6.5 Hz, 1H), 5.32 (d, *J* = 10.6 Hz, 1H), 2.48 (s, 3H), 2.15 (m, 2H), 1.04 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 173.4, 144.3, 140.4, 139.3, 138.2, 136.3, 131.1, 130.4, 128.8, 127.5, 126.3, 125.4, 123.3, 121.3, 72.9, 58.5, 27.7, 22.0, 9.2; FT-IR (film, NaCl) 3284, 1738, 1336, 1163 cm⁻¹.

General procedure for aldol reaction

To a stirred solution of **11** (396 mg, 1 mmol) in CH₂Cl₂ (10 mL) at 0 °C was added a 1M solution of TiCl₄ (1.1 mL, 1.1 mmol) dropwise under a N₂ atmosphere and the resulting solution was stirred for an additional 5 min. To this solution was added *N,N*-diisopropylethylamine (0.52 mL, 3 mmol) dropwise. To a separate flask charged with aldehyde (2 mmol) and CH₂Cl₂ (15 mL) at -78 °C was added a 1M solution of TiCl₄ (2.2 mL, 2.2 mmol) dropwise. After stirring 5 min at the same temperature CH₃CN (115 μL, 2.2 mmol) was added dropwise and the mixture was stirred further for 5 min. After this period, the above titanium enolate solution was added to this solution dropwise via cannula over 15 min. The reaction mixture was stirred at -78 °C for 2 h and quenched by addition of aqueous NH₄Cl. The resulting mixture was allowed to warm to 23 °C and the layers were separated. The aqueous layer was extracted with CH₂Cl₂. The combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated to afford crude aldol product. Either silica gel chromatography (5% to 10% EtOAc/CH₂Cl₂) or recrystallization from CH₂Cl₂/hexane after short path silica gel column chromatography yielded diastereomerically pure *anti*-aldol product **12**.

12a (R = *i*Bu). Mp = 130-131 °C; $[\alpha]_D^{23} + 81.2$ (c 0.51, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.88 (d, *J* = 8.2 Hz, 2H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.72 (d, *J* = 8.2 Hz, 1H), 7.51-7.47 (m, 3H), 7.35-7.33 (m, 3H), 6.43 (d, *J* = 6.5 Hz, 1H), 6.16 (d, *J* = 9.6 Hz, 1H), 5.35 (dd, *J* = 6.5, 9.6 Hz, 1H), 3.71-3.67 (m, 1H), 2.50 (qn, *J* = 7.0 Hz, 1H), 2.48 (s, 3H), 1.72 (m, 1H), 1.33-1.27 (m, 1H), 1.12-1.06 (m, 4H), 0.84 (d, *J* = 6.8 Hz, 3H), 0.71 (d, *J* = 6.5 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 173.8, 143.8, 140.3, 138.5, 137.5, 136.0,

130.7, 129.8, 128.5, 128.3, 127.3, 126.0, 124.8, 122.4, 121.0, 77.2, 72.9, 71.5, 57.9, 46.6, 43.5, 24.2, 23.6, 21.6, 21.3, 13.7; FT-IR (film, NaCl) 3522, 3279, 1731, 1335, 1161 cm^{-1} .

12b (R = Me). Mp = 136-138 $^{\circ}\text{C}$; $[\alpha]_{\text{D}}^{23} + 46.7$ (c 1.8, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.87 (d, $J = 8.3$ Hz, 2H), 7.77 (d, $J = 8.1$ Hz, 1H), 7.72 (d, $J = 8.1$ Hz, 1H), 7.52-7.37 (m, 4H), 7.33 (d, $J = 8.1$ Hz, 2H), 6.45 (d, $J = 6.5$ Hz, 1H), 6.33 (d, $J = 9.2$ Hz, 1H), 5.31 (dd, $J = 6.5, 9.6$ Hz, 1H), 3.86-3.81 (m, 1H), 2.71 (br s, 1H), 2.47 (qn, $J = 7.6$ Hz, 1H), 2.46 (s, 3H), 1.15 (d, $J = 6.3$ Hz, 3H), 1.09 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.1, 144.1, 140.9, 138.8, 137.9, 136.5, 131.2, 130.2, 128.9, 128.7, 127.8, 126.4, 125.1, 122.6, 121.5, 73.3, 63.9, 58.4, 48.4, 22.0, 21.2, 14.4; FT-IR (film, NaCl) 3487, 3285, 1732, 1335, 1162 cm^{-1} .

12c (R = Et). Mp = 118-120 $^{\circ}\text{C}$; $[\alpha]_{\text{D}}^{23} + 73.5$ (c 0.39, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 7.89 (d, $J = 8.3$ Hz, 2H), 7.79 (d, $J = 8.1$ Hz, 1H), 7.73 (d, $J = 8.3$ Hz, 1H), 7.53-7.35 (m, 6H), 6.47 (d, $J = 6.6$ Hz, 1H), 6.14 (d, $J = 9.4$ Hz, 1H), 5.34 (dd, $J = 6.6, 9.4$ Hz, 1H), 3.61-3.57 (m, 1H), 2.54 (qn, $J = 7.2$ Hz, 1H), 2.47 (s, 3H), 1.61-1.51 (m, 1H), 1.40-1.35 (m, 1H), 1.11 (d, $J = 7.2$ Hz, 3H), 0.91 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.2, 144.2, 140.8, 138.9, 137.9, 136.5, 131.2, 130.2, 128.9, 128.7, 127.8, 126.4, 125.2, 122.7, 121.5, 75.2, 73.4, 58.3, 46.3, 27.7, 22.0, 14.6, 9.6, 14.9; FT-IR (film, NaCl) 3532, 3282, 1729, 1334, 1161 cm^{-1} .

12d (R = *n*-Pr). Mp = 140-141 °C; $[\alpha]_D^{23} + 72.0$ (*c* 0.34, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.89 (d, *J* = 8.2 Hz, 2H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.73 (d, *J* = 8.2 Hz, 1H), 7.52-7.35 (m, 6H), 6.47 (d, *J* = 6.5 Hz, 1H), 6.09 (d, *J* = 9.6 Hz, 1H), 5.35 (dd, *J* = 6.5, 9.6 Hz, 1H), 3.66 (m, 1H), 2.54 (qn, *J* = 7.1 Hz, 1H), 2.48 (s, 3H), 2.41 (br s, 1H), 1.44-1.34 (m, 4H), 1.12 (d, *J* = 7.1 Hz, 3H), 0.84 (t, *J* = 6.9 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 174.2, 144.2, 140.8, 138.9, 137.9, 136.5, 131.2, 130.2, 128.9, 128.7, 127.8, 126.4, 125.2, 122.7, 121.5, 73.6, 73.4, 58.3, 46.6, 37.1, 22.0, 18.9, 14.5, 14.3; FT-IR (film, NaCl) 3525, 3289, 1730, 1335, 1161 cm⁻¹.

12e (R = *i*Pr). Mp = 133-134 °C; $[\alpha]_D^{23} + 66.8$ (*c* 0.7, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.88 (d, *J* = 8.2 Hz, 2H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.71 (d, *J* = 8.3 Hz, 1H), 7.52-7.45 (m, 3H), 7.35-7.32 (m, 3H), 6.46 (d, *J* = 6.6 Hz, 1H), 6.15 (d, *J* = 9.5 Hz, 1H), 5.33 (dd, *J* = 6.6, 9.5 Hz, 1H), 3.42 (m, 1H), 2.65 (qn, *J* = 7.2 Hz, 1H), 2.61 (br s, 1H), 2.46 (s, 3H), 1.73-1.69 (m, 1H), 1.09 (d, *J* = 7.1 Hz, 3H), 0.88 (d, *J* = 7.0 Hz, 3H), 0.86 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 174.7, 144.1, 140.8, 138.9, 138.0, 136.5, 131.1, 130.2, 128.9, 128.7, 127.8, 126.3, 125.2, 122.7, 121.4, 78.5, 73.4, 58.3, 44.0, 30.7, 22.0, 20.1, 16.2, 14.9; FT-IR (film, NaCl) 3529, 3288, 1731, 1336, 1162 cm⁻¹.

12f (R = cyhex). Mp = 163-164 °C; $[\alpha]_D^{23} + 34.8$ (*c* 0.82, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.71 (d, *J* = 8.2 Hz, 1H), 7.50-7.43 (m, 3H), 7.35-7.33 (m, 3H), 6.42 (d, *J* = 6.5 Hz, 1H), 6.14 (d, *J* = 9.5 Hz, 1H), 5.33 (dd, *J* = 9.5, 6.5 Hz, 1H), 3.35 (m, 1H), 2.68 (qn, *J* = 6.8 Hz, 1H), 2.59 (br s, 1H), 2.46 (s, 3H), 1.72-1.25 (m, 6H), 1.10 (d, *J* = 7.2 Hz, 3H), 1.08-0.94 (m, 5H); ¹³C NMR (125 MHz, CDCl₃) δ 174.8, 144.1, 140.8, 138.9, 138.0, 136.5, 131.2, 130.2, 128.9, 128.7, 127.7, 126.4, 125.2, 122.9, 121.4, 78.5, 73.4, 58.3, 43.0, 41.0, 30.2, 27.2, 26.7, 26.5, 22.0, 20.1, 15.0; FT-IR (film, NaCl) 3501, 3341, 1715, 1337, 1161 cm⁻¹.

12g (R = Ph). Mp = 170-172 °C; $[\alpha]_D^{23} + 54.4$ (c 0.55, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.89 (d, *J* = 8.2 Hz, 2H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.49-7.19 (m, 11H), 6.46 (d, *J* = 5.8 Hz, 1H), 6.23 (d, *J* = 9.1 Hz, 1H), 5.32 (dd, *J* = 5.8, 9.1 Hz, 1H), 4.67 (dd, *J* = 3.2, 9.0 Hz, 1H), 2.92 (d, *J* = 3.2 Hz, 1H), 2.82 (qn, *J* = 7.5 Hz, 1H), 2.47 (s, 3H), 0.93 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 173.4, 143.7, 141.0, 140.6, 138.3, 137.4, 136.1, 130.7, 129.8, 128.6, 128.3, 127.4, 126.7, 126.0, 124.7, 122.3, 121.2, 76.3, 72.9, 58.0, 47.9, 21.6, 14.3; FT-IR (film, NaCl) 3504, 3279, 1735, 1335, 1161 cm⁻¹.

12h (R = Ph(CH₂)₂). Mp = 132-133 °C; $[\alpha]_D^{23} + 88.1$ (c 0.84, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.90-7.87 (m, 2H), 7.77 (d, *J* = 7.6 Hz, 1H), 7.72 (d, *J* = 8.3 Hz, 1H), 7.51-7.45 (m, 3H), 7.32-7.06 (m, 8H), 6.45 (d, *J* = 6.5 Hz, 1H), 6.12 (d, *J* = 9.7 Hz, 1H), 5.36 (dd, *J* = 6.5, 9.7 Hz, 1H), 3.68-3.67 (m, 1H), 2.79-2.73 (m, 1H), 2.60-2.54 (m with qn at 2.55, *J* = 7.0 Hz, 2H), 2.45 (s, 3H), 1.73-1.62 (m, 2H), 1.10 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 174.3, 144.3, 142.0, 140.6, 138.9, 137.9, 136.4, 131.2, 130.3, 128.9, 128.8, 128.7, 127.7, 126.4, 125.3, 122.9, 121.4, 73.4, 73.2, 71.8, 58.3, 46.5, 32.2, 22.1, 14.3; FT-IR (film, NaCl) 3505, 3283, 1733, 1335, 1162 cm⁻¹.

13i (R = BnOCH₂). Mp = 109-111 °C; $[\alpha]_D^{23} + 100.6$ (c 0.73, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.76 (d, *J* = 7.9 Hz, 1H), 7.71 (d, *J* = 8.3 Hz, 1H), 7.52-7.15 (m, 11H), 6.36 (d, *J* = 6.5 Hz, 1H), 5.80 (d, *J* = 10.2 Hz, 1H), 5.41 (dd, *J* = 6.5, 10.2 Hz, 1H), 4.30 (dd, *J* = 4.4, 9.6 Hz, 2H), 3.96 (m, 1H), 3.34 (dd, *J* = 4.4, 9.6 Hz, 1H), 3.26 (dd, *J* = 6.8, 9.6 Hz, 1H), 2.69 (d, *J* = 3.4 Hz, 1H), 2.58 (dt, *J* = 2.1, 7.0 Hz, 1H), 2.47 (s, 3H), 1.09 (d, *J* = 7.1 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 174.1, 144.2,

140.6, 138.9, 138.2, 138.0, 136.4, 131.2, 130.3, 128.9, 128.8, 128.7, 128.2, 128.1, 127.6, 126.4, 125.3, 123.0, 121.4, 73.7, 73.6, 71.8, 58.4, 42.5, 22.0, 11.5; FT-IR (film, NaCl) 3508, 3283, 1732, 1336, 1163 cm^{-1} .

Establishment of Stereochemistry of Aldol Products

Relative configurations of aldol products were assigned based upon observed vicinal coupling constants ($J_{2,3} = 6.8\text{--}7.6$ Hz for *anti*-aldols, $J_{2,3} = 2.1$ Hz for *syn*-aldol) and also confirmed indirectly by correlation of corresponding diols and acids, resulting from removal of chiral auxiliary, with known compounds. Further confirmation of relative stereochemistry was done by correlation of coupling constant, J_{AC} (*syn* = 1.6 Hz, *anti* = 11.0 Hz), J_{BC} (*syn* = 3.0 Hz, *anti* = 5.0 Hz) of the isopropylidene derivatives with literature values.^{15,16} Absolute stereochemistry was confirmed by comparison of optical rotation of the resulting diols and carboxylic acids with reported values.

Ref 15 : Barret, A. G. M.; Rys, D. J. *J. Chem. Soc. Perkin Trans. I* **1995**, 8, 1009. Ref 16 : Marshall, J. A.; Wang, X.-j. *J. Org. Chem.* **1992**, 57, 1242.

Acid (**14**, **18**)

^1H NMR (500 MHz, CDCl_3) δ 3.77 (m, 1H), 2.52 (m, 1H), 1.86 (m, 1H), 1.44 (m, 1H), 1.28 (m, 1H), 1.25 (d, $J = 7.2$ Hz, 3H), 0.94 (d, $J = 7.1$ Hz, 3H), 0.92 (d, $J = 6.6$ Hz, 3H). $[\alpha]^{23}_{\text{D}}$ 17.4 (*c* 0.72, CHCl_3); lit^{3b} -18.2 (*c* 2.1, CHCl_3) for the opposite enantiomer. Ref 3b : Ghosh, A. K.; Fidanze, S.; Onish, M.; Hussain, K. A. *Tetrahedron Lett.* **1997**, 41, 7171.

^1H NMR (500 MHz, CDCl_3) δ 3.43 (dd, $J = 5.4, 6.5$ Hz, 1H), 2.68 (m, 1H), 1.80 (m, 1H), 1.24 (d, $J = 7.2$ Hz, 3H), 0.98 (d, $J = 6.8$ Hz, 3H), 0.92 (d, $J = 6.7$ Hz, 3H). $[\alpha]^{23}_{\text{D}}$ 17.4 (*c* 0.72, CHCl_3); lit^{3b} 118.2 (*c* 2.1, CHCl_3). $[\alpha]^{23}_{\text{D}}$ -11.7 (*c* 0.69, CHCl_3); lit¹⁸ -14.3 (*c* 1.0, CHCl_3). Ref 18 : Draanen, N. A. V.; Arseniyadis, S.; Crimmins, M. T.; Heathcock, C. H. *J. Org. Chem.* **1991**, 56, 2499.

14g

^1H NMR (500 MHz, CDCl_3) δ 7.38-7.32 (m, 5H), 4.76 (d, $J = 9.0$ Hz, 1H), 2.85 (m, 1H), 1.03 (d, $J = 7.2$ Hz, 3H). $[\alpha]_D^{23} -40.7$ (c 0.93, CHCl_3); lit¹⁹ -53.4 (c 0.34, CHCl_3). Ref 19 : Yang, Y.-C.; Hung, A.-K.; Chang, C.-S.; Yan, T.-J. *J. Org. Chem.* **1996**, 61, 2038.

17i

^1H NMR (500 MHz, CDCl_3) δ 7.37-7.30 (m, 5H), 4.57 (d, $J = 11.9$ Hz), 4.53 (d, $J = 11.9$ Hz), 4.13 (m, 1H), 3.56-3.50 (m, 2H), 2.71 (m, 1H), 1.22 (d, $J = 7.2$ Hz, 3H). $[\alpha]_D^{23} -12.78$ (c 1.2, CHCl_3); lit^{3b} 12.97 (c 3.7, CHCl_3) for the opposite enantiomer. Ref 3b : Ghosh, A. K.; Fidanze, S.; Onish, M.; Hussain, K. A. *Tetrahedron Lett.* **1997**, 41, 7171.

Diol (**15**, **18**)

15c

^1H NMR (500 MHz, CDCl_3) δ 3.76 (dd, $J = 3.6, 10.8$ Hz, 1H), 3.62 (dd, $J = 7.4, 10.8$ Hz, 1H), 3.50 (dt, $J = 3.4, 7.8$ Hz, 1H), 2.61 (br s, 2H), 1.73 (m, 1H), 1.63 (m, 1H), 1.47 (m, 1H), 0.97 (t, $J = 7.4$ Hz, 3H), 0.88 (d, $J = 7.0$ Hz, 3H). $[\alpha]_D^{23} 21.6$ (c 0.55, CHCl_3); lit²⁰ -22.6 (c 1.22, CHCl_3) for the opposite enantiomer. Ref 20 : Mori, K.; Sano, S.; Yokoyama, Y.; Bando, M.; Kido, M. *Eur. J. Org. Chem.* **1998**, 6, 1135.

15d

^1H NMR (500 MHz, CDCl_3) δ 3.77 (dd, $J = 3.5, 10.7$ Hz, 1H), 3.62 (dd, $J = 7.3, 10.7$ Hz, 1H), 3.57 (m, 1H), 2.55 (br s, 2H), 1.71 (m, 1H), 1.54-1.35 (m, 4H), 0.94 (t, $J = 7.0$ Hz,

3H), 0.88 (d, $J = 7.0$ Hz, 3H). $[\alpha]^{23}_{\text{D}} 32.5$ (c 0.58, CHCl_3); lit²¹ 33.6 (c 1.04, CHCl_3). Ref 21 : Masamune, S.; Sato, T.; Kim, B.-M.; Wollmann, T. S. *J. Am. Chem. Soc.* **1986**, 108, 8279.

15f

^1H NMR (500 MHz, CDCl_3) δ 3.76 (dd, $J = 3.2, 10.6$ Hz, 1H), 3.62 (dd, $J = 7.0, 10.6$ Hz, 1H), 3.33 (dd, $J = 3.9, 7.2$ Hz, 1H), 2.64 (br s, 2H), 1.88-1.11 (m, 12H), 0.88 (d, $J = 7.0$ Hz, 3H). $[\alpha]^{23}_{\text{D}} 24.1$ (c 0.83, CHCl_3); lit²² 24.3 (c 0.94, CHCl_3). Ref 22 : Harada, T.; Kurokawa, H.; Kagamihara, Y.; Tanaka, S.; Inoue, A.; Oku, A. *J. Org. Chem.* **1992**, 57, 1412.

15i

^1H NMR (500 MHz, CDCl_3) δ 7.31-7.18 (m, 5H), 3.80 (dd, $J = 3.5, 10.7$ Hz, 1H), 3.65-3.58 (m, 2H), 2.85 (m, 1H), 2.72-2.68 (m, 3H), 1.90 (m, 1H), 1.81-1.74 (m, 2H), 0.90 (d, $J = 7.0$ Hz, 3H). $[\alpha]^{23}_{\text{D}} 45.3$ (c 0.95, CHCl_3).

18i

^1H NMR (500 MHz, CDCl_3) δ 7.37-7.30 (m, 5H), 4.58 (d, $J = 11.8$ Hz, 1H), 4.55 (d, $J = 11.8$ Hz, 1H), 4.02 (m, 1H), 3.69-3.62 (m, 2H), 3.53-3.52 (m, 2H), 1.7 (m, 1H), 0.94 (d, $J = 7.2$ Hz, 3H). $[\alpha]^{23}_{\text{D}} -2.8$ (c 0.85, CHCl_3).

Isopropylidene Derivatives (16)

16g

$$\left(\begin{array}{l} J_{\text{AC}} = 11.5 \text{ Hz} \\ J_{\text{BC}} = 5.3 \text{ Hz} \\ J_{\text{CD}} = 10.3 \text{ Hz} \end{array} \right)$$

Barret, A. G. M.; Rys, D. J. *J. Chem. Soc. Perkin Trans. 1* **1995**, 8, 1009.

¹H NMR (500 MHz, CDCl₃) δ 3.67 (dd, *J* = 5.0, 11.4 Hz, 1H), 3.46 (dd, *J* = 11.0, 11.4 Hz, 1H), 3.28 (m, 1H), 1.84 (m, 1H), 1.75 (m, 2H), 1.63 (m, 2H), 1.46-1.35 (m, 9H), 1.25-1.18 (m, 4H), 0.72 (d, *J* = 6.7 Hz, 3H). [α]_D²³ 32.0 (*c* 0.75, CHCl₃). Relative configuration was assigned based on ¹H NMR spectral correlation with similar isopropylidene derivatives.¹⁶ Ref 16 : Marshall, J. A.; Wang, X.-j. *J. Org. Chem.* **1992**, 57, 1242.

¹H NMR (500 MHz, CDCl₃) δ 7.30-7.19 (m, 5H), 3.68 (dd, *J* = 5.1, 11.6 Hz, 1H), 3.47 (dd, *J* = 11.4, 11.6 Hz, 1H), 3.40 (m, 1H), 2.62 (m, 1H), 2.72-2.68 (m, 3H), 1.90 (m, 1H), 1.69 (m, 2H), 1.43 (s, 3H), 1.41 (s, 3H), 0.71 (d, *J* = 6.7 Hz, 3H). [α]_D²³ 62.9 (*c* 1.05, CHCl₃). Relative configuration was assigned based on ¹H NMR spectral correlation with similar isopropylidene derivatives.¹⁶ Ref 16 : Marshall, J. A.; Wang, X.-j. *J. Org. Chem.* **1992**, 57, 1242.

¹H NMR (500 MHz, CDCl₃) δ 7.37-7.28 (m, 5H), 4.62 (d, *J* = 12.1 Hz, 1H), 4.49 (d, *J* = 12.1 Hz, 1H), 4.23 (m, 1H), 4.12 (dd, *J* = 2.6, 11.5 Hz, 1H), 3.58 (dd, *J* = 1.4, 11.5 Hz, 1H), 3.47 (dd, *J* = 6.6, 9.6 Hz, 1H), 3.39 (dd, *J* = 6.0, 9.6 Hz, 1H), 1.60 (m, 1H), 1.47 (s, 3H), 1.41 (s, 3H), 1.04 (d, *J* = 6.9 Hz, 3H). [α]_D²³ 11.8 (c 0.34, CHCl₃). Relative

configuration was assigned based on ^1H NMR spectral correlation with similar isopropylidene derivatives.¹⁶ Ref 16 : Marshall, J. A.; Wang, X.-j. *J. Org. Chem.* **1992**, 57, 1242.

Integral

ppm

ppm

7.26132

0.9989

1.0439

1.0893

1.2485

4.5885

6.3532

3.78199

3.77555

3.76877

3.76195

3.75552

2.53555

2.52246

2.52159

2.50833

1.85890

1.85380

1.44125

1.43280

1.42155

1.41259

1.30584

1.29952

1.28661

1.28027

1.27834

1.27166

1.25549

1.24108

0.94822

0.93487

0.92975

0.92298

0.91660

0.90986

4.5939

2.0083

0.9982

2.0630

0.9704

3.4528

ppm

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7.34568
7.34109
7.32830
7.32313
7.31491
7.30786
7.29802
7.26128

4.55561
4.54931
4.14345
4.13349
4.13022
4.12407
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3.53550
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2.71343
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2.69902

1.23402
1.21956

ppm

7.26056

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- 3.76908
- 3.75417
- 3.74781
- 3.64658
- 3.63252
- 3.62589
- 3.61141
- 3.34201
- 3.33439
- 3.32782
- 3.32002
- 2.63663
- 1.87942
- 1.77051
- 1.66103
- 1.53198
- 1.48107
- 1.26636
- 1.26147
- 1.24319
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- 1.21092
- 1.20385
- 1.19653
- 1.17792
- 1.15250
- 1.13262
- 1.12808
- 1.11324
- 1.10937
- 0.89266
- 0.87871

ppm

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7.33657

7.26056

4.63065

4.60648

4.49927

4.47509

4.23367

4.22868

4.13770

4.13254

4.11468

4.10948

3.58698

3.58457

3.56404

3.56145

3.47054

3.46438

3.45134

3.40048

3.38839

3.38129

1.59451

1.59132

1.46388

1.43324

1.40358

1.04774

1.03392

a1d01(02-11-4)
 Pulse Sequence: szpul
 Solvent: CDCl3
 Ambient temperature
 Mercury-300BS "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
 FI size 16384
 Total time 0 min, 44 sec

Diastereomeric Ratio for 12 b
 (anti : syn = 80 : 20)

aldo(02-11-1
 Pulse Sequence: szpu1
 Solvent: CDCl3
 Ambient temperature
 Mercury-300DB "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, 299.8359758 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 16384
 Total time 0 min, 44 sec

Diastereomeric Ratio for 12 c
 (anti : syn = 92 : 8)

aldo1(02-11-5)
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 Mercury-300B8 "Varian"
 PULSE SEQUENCE
 Relax: delay 2.000 sec
 Pulse: 43.1 degrees
 Acq. time 2.105 sec
 Width: 3739.4 Hz
 Resolution
 OBSERVED H1 299.8559758 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FI size 16384
 Total time 0 min, 44 sec

Diastereomeric Ratio for 12 f

(anti : syn = 99 : 1)

Diastereomeric Ratio for 12 h
(anti : syn = 95: 5)

Current Data Parameters
 NAME 02-6-20
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020621
 Time 18.34
 INSTRUM spect
 PROBHD 5 mm BBO BB-1
 PULPROG zg
 TO 16384
 SOLVENT CDCl3
 NS 8
 DS 2
 SMH 4789.272 Hz
 FIDRES 0.292314 Hz
 AQ 1.7105396 sec
 RG 181
 DM 104.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 5.50 usec
 SF01 400.134200 MHz
 NUC1 1H
 PL1 -3.00 dB

F2 - Processing parameters
 SI 8192
 SF 400.132000 MHz
 MDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
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 F2P 0.000 ppm
 F2 0.00 Hz
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 HZCM 160.05261 Hz/cm

Current Data Parameters

NAME 02-5-15
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

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Time 11.05
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 472
DS 2
SMH 24154.590 Hz
FIDRES 0.737140 Hz
AQ 0.6783476 sec
RG 11585.2
DM 20.700 usec
DE 29.57 usec
TE 300.0 K
d11 0.03000000 sec
d12 0.0002000 sec
PL13 19.00 dB
D1 2.00000000 sec
CPOPRG2 waltz16
PCPD2 75.00 usec
SFO2 400.1342220 MHz
NUC2 1H
PL2 120.00 dB
PL12 16.00 dB
P1 3.00 usec
SFO1 100.6242960 MHz
NUC1 13C
PL1 6.00 dB

F2 - Processing parameters

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

1D NMR plot parameters

CX 20.00 cm
F1P 180.000 ppm
F1 18110.39 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.00000 ppm/cm
HZCM 905.51947 Hz/cm

Current Data Parameters

NAME 02-11-4

EXPNO 3

PROCNO 1

F2 - Acquisition Parameters

Date_ 20030113

Time 16.06

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zg

TD 32768

SOLVENT CDCl3

NS 8

DS 2

SMH 6009.615 Hz

FIDRES 0.183399 Hz

AQ 2.7263477 sec

R6 64

DW 83.200 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

P1 5.00 usec

SFO1 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 8.000 ppm

F1 4001.04 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCM 0.40000 ppm/cm

HZCM 200.05200 Hz/cm

Current Data Parameters

NAME 02-11-4

EXPNO 4

PROCNO 1

F2 - Acquisition Parameters

Date_ 20030113

Time 16.32

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zgpg

TD 32768

SOLVENT CDCl3

NS 500

DS 2

SWH 30303.031 Hz

FIDRES 0.924775 Hz

AQ 0.5407220 sec

RG 8192

DM 16.500 usec

DE 6.00 usec

TE 300.0 K

d11 0.03000000 sec

d12 0.00002000 sec

PL13 22.00 dB

D1 2.00000000 sec

CPDPRG2 wait216

PCPD2 80.00 usec

SFO2 500.1320000 MHz

NUC2 1H

PL2 3.00 dB

PL12 22.00 dB

P1 3.80 usec

SFO1 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 180.000 DPM

F1 22636.39 Hz

F2P 0.000 DPM

F2 0.00 Hz

PPMCM 9.00000 DPM/cm

HZCM 1131.81958 Hz/cm

Current Data Parameters

NAME 02-6-3
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20020605
Time 13.14
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 101.6
DM 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 8.000 ppm
F1 4001.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40000 ppm/cm
HZCM 200.06200 Hz/cm

Current Data Parameters

NAME 02-6-3

EXPNO 5

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020605

Time 14.00

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zgpg

TD 32768

SOLVENT CDCl3

NS 1000

DS 2

SMH 30303.031 Hz

FIDRES 0.924775 Hz

AQ 0.5407220 sec

RG 8192

DW 16.500 usec

DE 6.00 usec

TE 300.0 K

d11 0.03000000 sec

d12 0.00002000 sec

PL13 22.00 dB

D1 2.00000000 sec

CPDPRG2 waltz16

PCPD2 80.00 usec

SFO2 500.132000 MHz

NUC2 1H

PL2 3.00 dB

PL12 22.00 dB

P1 3.80 usec

SFO1 125.7715724 MHz

NUC1 13C

PL1 3.00 dB

F2 - Processing parameters

SI 32768

SF 125.7577399 MHz

WDW EM

SSB 0

LB 3.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

FAP 180.000 ppm

F1 22636.39 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCH 9.00000 ppm/cm

HZCM 1131.81958 Hz/cm

Current Data Parameters

NAME 02-6-21

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020625

Time 12:23

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zg

TD 32768

SOLVENT CDCl₃

NS 8

DS 2

SWH 6009.645 Hz

FIDRES 0.183399 Hz

AQ 2.7263477 sec

RG 128

DM 83.200 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

P1 5.00 usec

SFO1 500.1327507 MHz

NUC1 ¹H

PL1 3.00 dB

F2 - Processing parameters

SI 16384

MDW EM

SSB 0

LB 0.20 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

F1P 8.000 ppm

F1 4001.04 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCM 0.40000 ppm/cm

HZCM 200.05200 Hz/cm

Current Data Parameters

NAME	02-6-21
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20020625
Time	13.09
INSTRUM	spect
PROBHD	5 mm BBO BB-1
PULPROG	zgpg
TD	32768
SOLVENT	CDCl3
NS	996
DS	2
SWH	30303.031 Hz
FIDRES	0.92475 Hz
AQ	0.5407220 sec
RG	13004
DW	16.500 usec
DE	6.00 usec
TE	300.0 K
d11	0.03000000 sec
d12	0.0002000 sec
PL13	22.00 dB
D1	2.00000000 sec
CPDPRG2	waltz16
PCPD2	80.00 usec
SFO2	500.132000 MHz
NUC2	¹ H
PL2	3.00 dB
PL12	22.00 dB
P1	3.80 usec
SFO1	125.7715724 MHz
NUC1	¹³ C
PL1	3.00 dB

F2 - Processing parameters

SI	32768
SF	125.7677390 MHz
K0W	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	180.000 ppm
F1	22636.39 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	9.00000 ppm/cm
HZCM	1131.81958 Hz/cm

Current Data Parameters

NAME	02-5-16
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020518
Time	18.55
INSTRUM	Spec
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	2.726347 sec
RG	32
DM	83.200 usec
DE	6.00 usec
TE	300.0 K
D1	1.0000000 sec
P1	5.00 usec
SFO1	500.1327507 MHz
NUC1	¹ H
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	8.000 ppm
F1	4001.04 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.40000 ppm/cm
HZCM	200.05200 Hz/cm

Current Data Parameters

NAME 02-5-16

EXPNO 3

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020518

Time 19.32

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zgpg

TD 32768

SOLVENT CDCl3

NS 800

DS 2

SWH 30303.031 Hz

FIDRES 0.924775 Hz

AQ 0.5407220 sec

R6 8192

DW 16.500 usec

DE 6.00 usec

TE 300.0 K

d11 0.0300000 sec

d12 0.0002000 sec

PL13 22.00 dB

D1 2.0000000 sec

CPDPRG2 waltz16

PCPD2 80.00 usec

SFO2 500.1320000 MHz

NUC2 1H

PL2 3.00 dB

PL12 22.00 dB

P1 3.80 usec

SFO1 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 180.000 ppm

F1 22636.39 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCH 9.00000 ppm/cm

HZCM 1131.81956 Hz/cm

Current Data Parameters

NAME 02-11-5

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20021106

Time 14.51

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zg

TD 32768

SOLVENT CDCl3

NS 2

DS 2

SMH 6009.615 Hz

FIDRES 0.183399 Hz

AQ 2.763477 sec

RG 32

DM 83.200 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

P1 5.00 usec

SFO1 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 9.000 ppm

F1 4501.17 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCM 0.45000 ppm/cm

HZCM 225.05850 Hz/cm

Current Data Parameters

NAME 02-11-5

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20021106

Time 15.06

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zgpg

TD 32768

SOLVENT CDCl3

NS 256

DS 2

SWH 30303.031 Hz

FIDRES 0.924775 Hz

AQ 0.5407220 sec

RG 8192

DM 16.500 usec

DE 6.00 usec

TE 300.0 K

d11 0.03000000 sec

d12 0.0002000 sec

PL13 22.00 dB

D1 2.00000000 sec

CPDPRG2 waltz16

PCPD2 80.00 usec

SFO2 500.1320000 MHz

NUC2 1H

PL2 3.00 dB

PL12 22.00 dB

P1 3.80 usec

SFO1 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

CX 20.00 cm

F1P 180.000 ppm

F1 22636.39 Hz

F2P 10.000 ppm

F2 1257.58 Hz

PPMCM 8.50000 ppm/cm

HZCM 1068.94080 Hz/cm

Current Data Parameters

NAME 02-5-16-B

EXPNO 4

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020521

Time 14.27

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zg

TD 16384

SOLVENT CDCl3

NS 8

DS 2

SWH 4789.272 Hz

FIDRES 0.292314 Hz

AQ 1.7105396 sec

RG 64

DM 104.400 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

P1 5.50 usec

SFO1 400.1342000 MHz

NUC1 1H

PL1 -3.00 dB

F2 - Processing parameters

SI 8192

SF 400.1320090 MHz

WDM EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

F1P 8.000 ppm

F1 3201.05 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCH 0.40000 ppm/cm

HZCM 160.05283 Hz/cm

Current Data Parameters

NAME 02-5-16-B

EXPNO 5

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020521

Time 14.40

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zgpg

TD 32768

SOLVENT CDCl3

NS 1000

DS 2

SWH 24154.590 Hz

FIDRES 0.737140 Hz

AQ 0.6783476 sec

RG 10321.3

DM 20.700 usec

DE 29.57 usec

TE 300.0 K

d11 0.03000000 sec

d12 0.00002000 sec

PL13 19.00 dB

D1 2.00000000 sec

CPDPRG2 waltz16

PCPD2 75.00 usec

SFO2 400.1342220 MHz

NUC2 ¹H

PL2 120.00 dB

PL12 16.00 dB

P1 3.00 usec

SFO1 100.6242960 MHz

NUC1 ¹³C

PL1 6.00 dB

F2 - Processing parameters

SI 32768

SF 100.6132740 MHz

WDW EM

SSB 0

LB 3.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

F1p 180.000 ppm

F1 18110.39 Hz

F2p 0.000 ppm

F2 0.00 Hz

PPMCM 9.00000 ppm/cm

Current Data Parameters
NAME 02-11-5-B
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030211
Time 12:56
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SMH 6009.615 Hz
FIDRES 0.18339 Hz
AQ 2.7263477 sec
RG 114
DW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 5.00 usec
SFO1 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 8.000 ppm
F1 4001.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40000 ppm/cm
HZCM 200.05200 Hz/cm

Current Data Parameters
NAME 02-11-5-B
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030111
Time 16.58

INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 565

DS 2
SMH 30303.031 Hz
FIDRES 0.924775 Hz
AQ 0.5407220 sec
RG 8192

DE 16.500 usec
TE 300.0 K
d11 0.03000000 sec
d12 0.00002000 sec
PL13 22.00 dB
D1 2.00000000 sec

CPDPRG2 waltz16
PCPD2 80.00 usec
SF02 500.132000 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
MDM EM

SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 180.000 ppm
F1 22636.39 Hz
F2P 0.000 ppm
F2 0.00 Hz

PMCH 9.00000 ppm/cm
HZCM 1131.81958 Hz/cm

Current Data Parameters

NAME 02-6-7

EXPNO 3

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020611

Time 11.21

INSTRUM spect

PROBHD 5 mm BBO BB-1

PULPROG zg

TD 32768

SOLVENT CDCl₃

NS 8

DS 2

SMH 6009.615 Hz

FTORES 0.183399 Hz

AD 2.7263477 sec

RG 32

DM 83.200 usec

DE 6.00 usec

TE 300.0 K

D1 1.00000000 sec

P1 5.00 usec

SFO1 500.1327507 MHz

NUC1 ¹H

PL1 3.00 dB

F2 - Processing parameters

SI 16384

SF 500.1300130 MHz

MDM EM

SSB 0

LB 0.20 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

F1P 8.000 ppm

F1 4001.04 Hz

F2P 0.000 ppm

F2 0.00 Hz

PPMCM 0.40000 ppm/cm

HZCM 200.05200 Hz/cm

Current Data Parameters

NAME	02-6-7
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20020611
Time	12.06
INSTRUM	spect
PROBHD	5 mm BBO BB-1
PULPROG	zgpg
TD	32768
SOLVENT	CDCl3
NS	1000
DS	2
SMH	30303.031 Hz
FIDRES	0.924775 Hz
AQ	0.5407220 sec
RG	9195.2
DM	16.500 usec
DE	6.00 usec
TE	300.0 K
d11	0.03000000 sec
d12	0.0002000 sec
PL13	22.00 dB
D1	2.00000000 sec
CPDPRG2	waltz16
PCPD2	80.00 usec
SFO2	500.1320000 MHz
NUC2	1H
PL2	3.00 dB
PL12	22.00 dB
P1	3.80 usec
SFO1	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	180.000 ppm
F1	22636.39 Hz
F2p	0.000 ppm
F2	0.00 Hz
PMCKM	9.00000 ppm/cm
HZCM	1131.81958 Hz/cm