Modular Synthesis of Cyclic Peptidomimetics Inspired by β -Turns

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Supporting Information

Experimental Details

Copies of ^1H - and ^{13}C -NMR Spectra

Experimental Details

General Procedure for Reduction of Amino Acids.¹ Phenylalaninol. To a cold solution of LiBH_4 (1.31 g, 60.5 mmol, 2 equiv) in 40 mL of THF was added trimethyl silyl chloride (15.3 mL, 60.5 mmol, 2 equiv). The ice/water bath was removed and the mixture allowed to stir at room temperature for 20 min. Then the mixture was recooled to 0 °C and phenylalanine (5 g, 30.2 mmol, 1 equiv) was added and the reaction mixture was stirred for 12 h. The reaction mixture was quenched with methanol and the reaction mixture was concentrated. The residue was partitioned between 2.5 M aqueous sodium hydroxide solution and methylene chloride, washed with brine, and dried over anhydrous Na_2SO_4 . The organic layers were filtered and concentrated to afford an oil that was used without further purification.

General Procedure for Azidation of Amino Alcohols.²

(S)-2-Azido-3-phenylpropan-1-ol (1c). NaN_3 (11.2 g, 172 mmol, 4.9 equiv) was dissolved in 35 mL of water and cooled to 0 °C. CH_2Cl_2 (40 mL) was added to the vigorously stirred solution followed by trifluoromethanesulfonic anhydride (10.0 g, 5.96 mL, 35.4 mmol). The reaction mixture was allowed to stir for 2 h at 0 °C. The organic phase was separated and the aqueous layer extracted with methylene chloride (2 $\times$ 10 mL). The combined organic layers were washed with sodium bicarbonate solution and water and dried over anhydrous sodium sulfate. The yield was taken to be 50% for the purpose of determining the amounts of triflyl azide used in subsequent reactions (base titration of triflic acid formed during reaction indicated that about 50% of the anhydride had hydrolyzed during the reaction). To a solution of phenylalaninol (1.41 g, 9.2 mmol, 1 equiv) in methylene chloride (5 mL) was added 4-(dimethylamino)pyridine (4.96 g, 40.6 mmol, 4.4 equiv) at room temperature. Subsequently triflyl azide was added dropwise. The reaction mixture was allowed to stir at room temperature for 2 h. The reaction mixture was concentrated and the known azido alcohol **1c** was isolated using flash chromatography. Colorless oil; yield 93%; R_f 0.66 (1:1, EtOAc/hexane); $[\alpha]_D^{25}$ +2.4 (c 1.0, CHCl_3). IR (neat): 3375, 2105 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 2.94-2.83 (m, 2H); 3.2 (br s, 1H); 3.62-3.57 (dd, J = 7.8, 12.2 Hz, 1H); 3.78-3.73 (t, J = 8.4 Hz, 2H); 7.41-7.29 (m, 5H). ^{13}C NMR (100.6 MHz, CDCl_3): 37.4, 64.8, 65.7, 127.6, 128.9, 129.8, 137.6.

(S)-2-Azido-3-methylbutan-1-ol (1a). Colorless oil; yield 90%; R_f 0.86 (1:1, EtOAc/hexane); $[\alpha]_D^{25} +4.9$ (c 3.1, CHCl_3). IR (neat) 3359, 2097 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 0.95 (d, $J = 3.4$ Hz, 3H); 0.97 (d, $J = 3.7$ Hz, 3H); 1.87-1.76 (septet, $J = 6.6$ Hz, 1H); 2.87 (br s, 1H); 3.27-3.24 (m, 1H); 3.62-3.56 (m, 1H); 3.75-3.71 (m, 1H). ^{13}C NMR (100.6 MHz, CDCl_3): 18.8, 19.9, 30.2, 63.9, 71.1; MS (EI) calcd for $\text{C}_5\text{H}_{11}\text{N}_3\text{O}$ 129.0; found, m/e 129.0.

(S)-2-Azido-4-methylpentan-1-ol (1b). Colorless oil; yield 86%; R_f 0.83 (1:1, EtOAc/hexane); $[\alpha]_D^{25} -6.9$ (c 2.4, CHCl_3). IR (neat): 3374, 2104 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 0.92 (d, $J = 1.2$ Hz, 3H); 0.94 (d, $J = 1.3$ Hz, 3H); 1.29-1.22 (m, 1H); 1.45-1.38 (m, 1H); 1.77-1.74 (septet, $J = 1.8$ Hz, 1H); 2.95 (br s, 1H); 3.52-3.46 (m, 2H); 4.11-4.04 (m, 1H). ^{13}C NMR (100.6 MHz, CDCl_3): 22.3, 23.4, 25.3, 39.7, 63.9, 65.9; MS (EI) calcd for $\text{C}_6\text{H}_{13}\text{N}_3\text{O}$ 143.10; found, m/e 143.0.

(S)-2-Azido-3-(4-(benzyloxy)phenyl)propan-1-ol (1d). White solid; yield 87%; mp 48–50 $^{\circ}\text{C}$; R_f 0.69 (1:1, EtOAc/hexane); $[\alpha]_D^{25} -9.0$ (c 0.7, CHCl_3). IR (neat): 3426, 2105 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 2.46 (s, 1H); 2.88-2.77 (m, 2H); 3.57 (br s, 1H); 3.74-3.66 (m, 2H); 5.09 (s, 2H); 6.97 (d, $J = 8.3$ Hz, 2H); 7.17 (d, $J = 8.3$ Hz, 2H), 7.49-7.38 (m, 5H). ^{13}C NMR (100.6 MHz, CDCl_3): 40.3, 54.7, 66.6, 70.5, 115.4, 127.9, 128.4, 129.0, 130.6, 131.4, 137.5, 157.9. HRMS calcd for $\text{C}_{16}\text{H}_{17}\text{N}_3\text{O}_2$ ($\text{M}^+ + \text{H}$), 283.1321; found, 283.1319.

(S)-Benzyl-4-azido-5-hydroxypentylcarbamate (1e). Colorless oil; yield 71%; R_f 0.64 (1:1, EtOAc/hexane); $[\alpha]_D^{25} +0.38$ (c 5.2, CHCl_3). IR (neat): 3330, 2104, 1698 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 1.59-1.43 (m, 4H); 3.16 (d, $J = 5.1$ Hz, 2H); 3.28 (s, 1H); 3.55 (d, $J = 7.1$ Hz, 1H); 3.67 (d, $J = 2.5$ Hz, 1H); 4.15 (s, 1H); 5.07 (s, 2H); 5.48-5.24 (m, 1H); 7.33 (s, 5H). ^{13}C NMR (100.6 MHz, CDCl_3): 26.8, 28.0, 41.0, 63.3, 65.3, 66.9, 128.4, 128.7, 129.0, 136.9, 157.3. HRMS calcd for $\text{C}_{13}\text{H}_{19}\text{N}_4\text{O}_3$ ($\text{M}^+ + \text{H}$), 279.1457; found, 279.1458.

(S)-Benzyl-5-azido-6-hydroxyhexylcarbamate (1f). Colorless oil; yield 93%; R_f 0.60 (1:1, EtOAc/hexane); $[\alpha]_D^{25} +4.9$ (c 3.5, CHCl_3). IR (neat): 3338, 2103, 1697 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 1.50-1.28 (m, 6H); 3.18 (s, 2H); 3.44 (s, 1H); 3.55 (d, $J = 7$ Hz, 1H); 3.67 (d, $J = 8.6$ Hz, 1H); 3.8 (br s, 1H); 5.09 (s, 2H); 5.17 (s, 1H); 7.35 (s, 5H). ^{13}C NMR (125.75 MHz, CDCl_3): 22.9, 29.6, 29.9, 40.5, 63.9, 64.8, 66.9, 128.0, 128.08, 128.47, 136.38, 156.6. HRMS calcd for $\text{C}_{14}\text{H}_{21}\text{N}_4\text{O}_3$ ($\text{M}^+ + \text{H}$): 293.1614; found, 293.1613.

(S)-2-Azido-4-(methylthio)butan-1-ol (1g). Colorless oil; yield 82%; R_f 0.74 (1:1, EtOAc/hexane); $[\alpha]_D^{20}$ +30 (c 2.9, CHCl_3). IR (neat): 3392, 2123 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 1.61-1.54 (m, 2H); 1.93 (s, 3H); 2.49-2.36 (m, 2H); 3.46-3.40 (m, 2H); 3.54 (dd, J = 2.6, 10.2 Hz, 1H); 3.79 (br s, 1H). ^{13}C NMR (125.7 MHz, CDCl_3): 15.1, 29.5, 30.2, 62.6, 64.8. HRMS calcd for $\text{C}_5\text{H}_{12}\text{N}_3\text{O}_1\text{S}_1$ ($\text{M}^+ + \text{H}$): 162.0701; found, 162.0676.

(S)-Benzyl-2-azido-3-hydroxypropanoate (1h). Colorless oil; yield 90%; R_f 0.81 (1:1 EtOAc/hexane); $[\alpha]_D^{20}$ +26 (c 1.4, CHCl_3). IR (neat): 3435, 2112, 1743 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 2.83 (br s, 1H); 3.94 (d, J = 4.5 Hz, 2H); 4.11 (t, J = 4.5 Hz, 1H); 5.31 (s, 2H); 7.40-7.30 (m, 5H). ^{13}C NMR (125.75 MHz, CDCl_3): 62.6, 63.4, 67.7, 127.0, 128.3, 128.6, 134.8, 164.7. HRMS calcd for $\text{C}_{14}\text{H}_{21}\text{N}_4\text{O}_3$ ($\text{M}^+ + \text{NH}_4$): 239.1144; found, 239.1146.

Ketones 2a – 2c. Compounds 2a and 2c are commercially available from Aldrich. Compound **2b** was synthesized according to literature procedure.³

Benzyl-2-oxo-2-(4'-oxopiperidin-1-yl)ethylcarbamate (2d). To a stirred solution of 4-piperidone hydrochloride (5 g, 32.5 mmol) in THF (50 mL) was added Et_3N (18.2 mL, 130 mmol). The solution was transferred to a flask containing 2-benzylloxycarbonylacetic acid (8.17 g, 39.1 mmol), DCC (10.14 g, 49 mmol), and HOBt (6.59 g, 48 mmol). After 12 h the reaction mixture was diluted with ethyl acetate (250 mL), filtered to remove dicyclohexylurea, and concentrated. Ethyl acetate (250 mL) was added to the residue, which was then washed with water, saturated aqueous sodium bicarbonate, 1N HCl, and brine. The organic layer was dried

over anhydrous sodium sulfate and concentrated in vacuo. Column chromatography afforded the title compound. White solid; mp 115-117 °C, yield 72%; R_f 0.5 (EtOAc, 100%); IR (neat): 1718, 1642, 1425 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 2.51 (s, 4H); 3.71 (s, 2H), 3.92 (s, 2H), 4.14 (s, 2H); 5.15 (s, 2H); 5.80 (s, 1H), 7.74-7.28 (m, 5H). ^{13}C NMR (125.75 MHz, CDCl_3): 40.5, 40.6, 41.0, 42.5, 42.7, 66.9, 127.9, 128.1, 128.4, 136.2, 156.1, 166.8, 205.6. HRMS calcd for $\text{C}_{15}\text{H}_{19}\text{N}_2\text{O}_4$ ($\text{M}^+ + \text{H}$): 291.1345; found, 291.1365.

General Procedure for the Syntheses of 1,4-Diazepin-5-ones. To a solution of *N*-benzyl-4-piperidone (0.100 g, 0.52 mmol) and azido alcohol (**1a-h**; 0.15 g, 0.84 mmol) in CH_2Cl_2 (2 mL) was added trifluoromethanesulfonic acid (0.39 g, 0.23 mL, 2.64 mmol) dropwise at 0 °C. The solution was allowed to warm to room temperature and stirred for 12 h. The solution was concentrated under reduced pressure and a saturated solution of NaHCO_3 (20 mL) was added. The reaction was stirred for an additional 12 h at room temperature whereupon 25 mL of CH_2Cl_2 was added. The organic layers were washed with water, dried over anhydrous Na_2SO_4 , and concentrated to give a crude yellow oil. Column chromatography afforded the desired compounds.

(S)-1-Benzyl-4-(1'-hydroxy-3'-phenylpropan-2'-yl)-1,4-diazepan-5-one (3a). Colorless oil; yield 84%; R_f 0.13 (MeOH/EtOAc, 1:9); $[\alpha]_D^{25}$ +40 (c 4.5, CHCl_3). IR (neat): 3380, 1623 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 2.35 (s, 2H); 2.49 (d, J = 5.1 Hz, 2H); 2.58 (s, 2H); 2.91-2.80 (m, 2H); 3.31 (d, J = 3.9 Hz, 2H); 3.44 (s, 2H); 3.94-3.63 (m, 2H); 4.25 (br s, 1H); 4.72 (s, 1H); 7.31-7.19 (m, 10H). ^{13}C NMR (100.6 MHz, CDCl_3): 35.3, 39.0, 46.4, 50.9, 56.1, 59.5, 63.2, 63.3, 126.9, 127.9, 128.8, 128.9, 129.2, 129.4, 138.4, 138.6, 176.7. HRMS calcd for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}_2$ ($\text{M}^+ + \text{H}$): 339.2073; found, 339.2058.

(S)-1-Benzyl-4-(1'-hydroxy-3'-phenylpropan-2'-yl)-5-oxo-1,4-diazepan-1-carboxylate (3b). Colorless oil; yield 46%; R_f 0.13 (MeOH/EtOAc, 1:9); $[\alpha]_D^{25}$ +3.7 (c 0.9, CHCl₃). IR (neat): 3400, 1644 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): 2.57 (m, 2H); 2.88 (m, 2H); 3.75-3.29 (complex, 9H); 4.73 (br s, 1H); 5.11 (s, 2H); 7.36-7.18 (m, 10H). ¹³C NMR (100.6 MHz, CDCl₃): 21.5, 35.1, 40.0, 42.1, 47.3, 59.8, 63.5, 67.9, 128.4, 128.8, 128.9, 129.2, 129.6, 136.7, 138.2, 155.7, 176.0. HRMS calcd for C₂₂H₂₇N₂O₄ (M⁺ + H): 383.1971; found, 383.1958.

(S)-[9H-Fluoren-9-yl]methyl-4-[1'-hydroxy-3'-phenylpropan-2'-yl]-5-oxo-1,4-diazepane-1-carboxylate (3c). White foam; yield 74%; R_f 0.13 (MeOH/EtOAc, 1:9); $[\alpha]_D^{25}$ +2.8 (c 1, CHCl₃). IR (neat): 3416, 1700, 1629 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): 2.32 (m, 1H); 2.46 (m, 1H); 2.88 (d, J = 8.3 Hz, 2H); 3.34-2.87 (complex, 9H); 4.73 (m, 1H); 5.08 (s, 2H); 7.36-7.15 (m, 10H). ¹³C NMR (100.6 MHz, CDCl₃): 35.0, 39.9, 42.1, 47.4, 47.8, 64.0, 67.6, 120.4, 125.2, 127.2, 127.5, 128.1, 129.1, 129.3, 138.2, 141.8, 144.2, 175.8. HRMS calcd for C₂₉H₃₁N₂O₄ (M⁺ + H): 471.2284; found, 471.2276.

(S)-1-Benzyl-4-(1'-hydroxy-3'-methylbutan-2'-yl)-1,4-diazepan-5-one (4). Yellow oil; yield 89%; R_f 0.9 (CH_2Cl_2 :MeOH, 9:1); $[\alpha]_D^{25}$ 13.6 (c 2.1, CHCl_3). IR (neat): 3394, 1627 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 0.88 (d, J = 6.5 Hz, 3H); 0.97 (d, J = 6.5 Hz, 3H); 1.84-1.78 (m, 1H); 2.78-2.61 (m, 6H); 3.41 (d, J = 3.3 Hz, 2H); 3.61-3.52 (m, 4H); 3.82 (dd, J = 3.2, 11.6 Hz, 1H), 4.11-4.07 (m, 1H); 7.30-7.25 (m, 5H). ^{13}C NMR (100.6 MHz, CDCl_3): 20.3, 20.8, 27.4, 38.7, 45.5, 50.8, 55.9, 62.3, 63.1, 63.8, 127.7, 128.8, 129.4, 138.3, 176.9. HRMS calcd for $\text{C}_{17}\text{H}_{27}\text{N}_2\text{O}_2$ ($\text{M}^+ + \text{H}$): 291.2073; found, 291.2073.

(S)-1-Benzyl-4-(1'-hydroxy-3'-methylpentan-2'-yl)-1,4-diazepan-5-one (5). Yellow oil; yield 87%; R_f 0.36 (MeOH/EtOAc 1:9); $[\alpha]_D^{25}$ 5.2 (c 11.2, CHCl_3). IR (neat): 3394, 1627 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 0.90 (d, J = 6.9 Hz, 3H); 0.92 (d, J = 3.6 Hz, 3H); 1.20-1.15 (septet, J = 4.7 Hz, 1H); 1.34-1.29 (m, 1H); 1.51 (br s, 1H), 2.56 (s, 2H); 2.63 (s, 2H); 2.70 (s, 2H); 3.32 (s, 2H); 3.60-3.35 (m, 5H); 4.69 (t, J = 4.1 Hz, 1H), 7.31-7.25 (m, 5H). ^{13}C NMR (125.75 MHz, CDCl_3): 22.1, 22.8, 25.0, 37.2, 38.5, 43.4, 50.6, 54.1, 55.9, 62.7, 63.6, 127.1, 128.3, 128.9, 138.0, 176.4. HRMS calcd for $\text{C}_{18}\text{H}_{29}\text{N}_2\text{O}_2$ ($\text{M}^+ + \text{H}$): 305.2229; found, 305.2204.

(S)-1-Benzyl-4-[3-(4-(benzyloxy)phenyl)-1'-hydroxypropan-2'-yl]-1,4-diazepan-5-one (6). Yellow oil; yield 63%; R_f 0.39 (MeOH/EtOAc 1:9); $[\alpha]_D^{25}$ +39.3 (c 2.9, CHCl_3). IR (neat): 3373, 1627 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 2.40 (s, 2H); 2.54 (d, J = 5.6 Hz, 1H); 2.63 (s, 2H); 2.83 (d, J = 6.5 Hz, 2H); 3.31 (d, J = 5.1 Hz, 2H); 3.75-3.45 (m, 6H); 4.62 (d, J = 4.7 Hz, 1H), 5.05 (s, 2H), 6.93 (d, J = 1.4 Hz, 2H), 7.13 (d, J = 8.4 Hz, 2H), 7.45-7.28 (m, 10H). ^{13}C NMR (100.6 MHz, CDCl_3): 34.3, 39.0, 46.8, 50.9, 56.0, 60.3, 63.2, 63.8, 70.4, 115.3, 127.9, 128.4, 128.8, 128.9; 129.5, 129.7, 130.4, 138.3, 157.9, 176.8. HRMS calcd for $\text{C}_{28}\text{H}_{33}\text{N}_2\text{O}_3$ ($\text{M}^+ + \text{H}$): 445.2491; found, 445.2469.

(S)-4-(5-Amino-1-hydroxypentan-2-yl)-1-benzyl-1,4-diazepan-5-one (7). Yellow oil; yield 84%; R_f 0.51 (MeOH/EtOAc, 1:9); $[\alpha]_D^{25}$ +7.8 (c 1.2, CHCl_3). IR (neat): 3411, 1623 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 1.71-1.68 (m, 1H); 2.81-2.61 (m, 2H); 3.90-3.41 (s, 6H); 4.68 (s, 1H); 7.35-7.07 (m, 5H). ^{13}C NMR (125.75 MHz, CDCl_3): 24.4, 28.2, 38.9, 44.1, 50.9, 52.9, 56.6, 57.0, 62.8, 63.0, 127.2, 128.7, 129.0, 138.2, 174.6. MS (EI) calcd for $\text{C}_{17}\text{H}_{27}\text{N}_3\text{O}_2$ 305.2; found, m/e 305.0.

(S)-Benzyl-5-(4'-benzyl-7'-oxo-1',4'-diazepan-1'-yl)-6-hydroxyhexylcarbamate (8).

Yellow oil; yield 88%; R_f 0.54 (MeOH/EtOAc, 1:9); $[\alpha]_D^{25}$ $\square$ 6.9 (c 0.97, CHCl_3). IR (neat): 3329, 1716, 1628 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 1.68-1.33 (m, 6H); 2.72-2.63 (m, 6H); 3.22-3.18 (m, 2H); 3.38 (s, 1H); 3.95-3.49 (m, 3H); 4.51 (s, 1H); 4.93 (s, 1H); 5.10 (s, 2H); 7.37-7.11 (m, 10H). ^{13}C NMR (125.75 MHz, CDCl_3): 23.1, 27.6, 29.4, 38.3, 40.5, 44.1, 50.4, 55.8, 56.4, 62.7, 63.6, 66.5, 127.2, 128.0, 128.3, 128.4, 128.9, 136.5, 137.8 (2C), 156.4, 176.3. HRMS calcd for $\text{C}_{26}\text{H}_{36}\text{N}_3\text{O}_4$ ($\text{M}^+ + \text{H}$): 454.2706; found, 454.2723.

(S)-1-Benzyl-4-(1'-hydroxy-4'-(methylthio)butan-2'-yl)-1,4-diazepan-5-one (9).

Yellow oil; yield 55%; R_f 0.28 (EtOAc/MeOH, 9:1); $[\alpha]_D^{25}$ $\square$ 2.0 (c 1.1, CHCl_3). IR (neat): 3411, 1623 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 1.83-1.27 (m, 2H), 2.10 (s, 3H); 2.78-2.47 (m, 8H), 3.44 (t, J = 4.6 Hz, 1H), 4.56-4.45 (m, 6H); 4.56 (s, 1H), 7.37-7.26 (m, 5H). ^{13}C NMR (125.75 MHz, CDCl_3): 15.5, 27.9, 30.9, 41.2, 50.5, 51.2, 52.8, 61.9, 62.7, 63.2, 127.2, 128.3, 128.9, 137.7, 176.3. HRMS calcd for $\text{C}_{17}\text{H}_{27}\text{N}_2\text{O}_2\text{S}$ ($\text{M}^+ + \text{H}$): 323.1793; found 323.1782.

(S)-Benzyl-3-(4'-benzyl-7'-oxo-1',4'-diazepan-1'-yl)-6-hydroxybutanoate (10). Yellow oil; yield 62%; R_f 0.33 (MeOH/EtOAc, 1:9); $[\alpha]_D^{25}$ 11.5 (c 3, CHCl₃). IR (neat): 3373, 1739, 1650 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): 2.75-2.28 (m, 6H); 3.59-3.44 (m, 4H); 4.06-3.94 (m, 2H); 4.98 (d, J = 4.5 Hz, 1H); 5.18 (dd, J = 1.4, 12.8 Hz, 2H); 7.55-7.29 (m, 10H). ¹³C NMR (100.6 MHz, CDCl₃): 38.0, 48.7, 50.3, 55.6, 60.9, 61.4, 62.8, 67.2, 127.3, 128.4, 128.5, 128.6, 128.9, 129.1, 135.2, 137.9, 169.8, 175.7. FAB calcd for C₂₃H₂₈N₂O₄ (M^+ - CH₂Ph + H): 306.16; found 307.1.

(S)-(9H-Fluoren-9-yl)methyl-4-(1-hydroxy-3-methylbutan-2-yl)-5-oxo-1,4-diazepane-1-carboxylate (11). White foam; yield 63%; R_f 0.2 (EtOAc/MeOH, 9:1); $[\alpha]_D^{25}$ 1.7 (c 1, CHCl₃). IR (neat): 3456, 1695, 1638 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): 0.87 (d, J = 6.6 Hz, 3H), 0.99 (d, J = 6.5 Hz, 3H), 1.84 (m, 1H), 2.09 (m, 1H), 2.52 (m, 2H), 3.58-3.34 (complex, 6H), 4.09 (m, 1H), 4.23 (m, 2H), 7.33 (t, J = 7.4 Hz, 2H), 7.41 (t, J = 7.4 Hz, 2H), 7.57 (d, J = 7.4 Hz, 2H), 7.77 (d, J = 7.5 Hz, 2H). ¹³C NMR (100.6 MHz, CDCl₃): 20.3, 20.7, 27.3, 39.8, 42.4, 46.2, 47.5, 47.9, 62.1, 63.6, 67.3, 67.6, 120.4, 125.2, 127.5, 128.2, 141.8, 144.3, 155.8, 175.9. HRMS calcd for C₂₅H₃₁N₂O₄ ($M+H^+$): 423.2275, found 423.2284.

(S)-(9H-Fluoren-9-yl)methyl-4-(1-hydroxy-3-methylpentan-2-yl)-5-oxo-1,4-diazepane-1-carboxylate (12). Colorless oil, yield 68%; R_f 0.22 (EtOAc/MeOH, 9:1); $[\alpha]_D^{20}$ +0.8 (c 1.05, CHCl_3). IR (neat): 3441, 1685, 1629 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 0.92 (d, J = 6.4, 6H), 1.18 (m, 1H), 1.32 (m, 1H), 2.46 (m, 1H), 2.51 (m, 3H), 3.74-2.97 (complex, 8H), 4.23 (t, J = 5.6 Hz, 1H), 4.57 (quartet, J = 7.4, 5.9, 2H), 4.72 (m, 1H), 7.32 (t, J = 7.4, 2H), 7.40 (t, J = 7.3, 2H), 7.56 (d, J = 7.2, 2H), 7.76 (d, J = 7.5, 2H). ^{13}C NMR (100.6 MHz, CDCl_3): 22.6, 23.7, 25.5, 37.6, 39.9, 42.5, 44.7, 47.9, 54.8, 64.3, 67.6, 120.4, 125.2, 127.5, 128.2, 141.8, 144.2. HRMS calcd for $\text{C}_{26}\text{H}_{32}\text{N}_2\text{O}_4$ ($\text{M}^+ + \text{H}^+$): 437.2418, found 437.2418.

(S)-Benzyl-4-(1-hydroxy-4-methylpentan-2-yl)-5-oxo-1,4-diazepan-1-carboxylate (13). Colorless oil, yield 52%; R_f 0.31 (EtOAc/MeOH, 9:1); $[\alpha]_D^{20}$ +1.1 (c 1.03, CHCl_3). IR (neat): 3436, 1695, 1649 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): 0.90 (d, J = 3.9, 3H), 0.91 (d, J = 3.7, 3H), 1.17 (m, 2H), 1.33 (m, 1H), 1.47 (m, 1H), 2.68 (m, 2H), 3.63-3.35 (complex, 7H), 3.74 (dd, J = 14.7, 6.6, 2H), 4.74 (m, 1H), 5.13 (s, 2H), 7.34 (m, 5H). ^{13}C NMR (100.6 MHz, CDCl_3): 22.6, 23.7, 25.5, 37.7, 40.0, 42.3, 44.5, 47.8, 54.6, 63.8, 68.0, 128.4, 128.9, 129.4, 136.7, 155.8, 176.1. HRMS calcd for $\text{C}_{22}\text{H}_{27}\text{N}_2\text{O}_4$ ($\text{M}^+ + \text{H}^+$): 349.2127, found 349.2121.

(S)-Benzyl-2-[(4'-(1'-hydroxy-4'-methylpentan-2'-yl)-5-oxo-1,4-diazepan-1-yl)-2-oxoethylcarbamate (14). White solid, 79%; m.p 102-104 °C; R_f 0.4 (EtOAc:MeOH, 9:1); $[\alpha]_D^{23}$ 23.1 (c 3.1, CHCl₃). IR (neat): 3406, 1718, 1637 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): 0.93 (d, J = 3.5 Hz, 3H); 0.94 (d, J = 3.1 Hz, 3H); 1.26-1.21 (septet, J = 5 Hz, 1H); 1.51-1.37 (m, 2H); 1.70 (br s, 2H); 2.09 (br s, 1H); 2.78-2.73 (m, 2H); 3.59-3.37 (m, 6H); 4.12-4.06 (m, 2H), 4.78-4.75 (q, J = 4.5 Hz, 1H), 5.14 (s, 2H); 5.83 (s, 1H); 7.39-7.28 (m, 5H). ¹³C NMR (125.75 MHz, CDCl₃): 22.1, 23.2, 25.1, 37.1, 39.2, 42.6; 43.8, 45.3, 47.7, 54.3, 63.8, 66.9, 127.9, 128.3, 128.4, 136.2, 156.2, 167.1, 175.1. HRMS calcd for C₂₁H₃₂N₃O₅ (M⁺+H): 406.2342; found, 406.2344.

(S)-1-Benzyl-4-[1'-(benzyloxy)-4'-methylpentan-2'-yl]-1,4-diazepan-5-one. Sodium hydride (60% in mineral oil, 0.272 g, 11.3 mmol) was suspended in DMF (5 mL) and hexane (1 mL), cooled to -78 °C, and **5** (1.15 g, 3.7 mmol) in DMF was added dropwise. The resulting mixture was stirred for 15 min, at which time benzyl bromide (0.71 g, 0.23 mL, 4.2 mmol) was added dropwise and the reaction mixture was allowed to stir at room temperature for 3 h. The reaction mixture was diluted with ethyl acetate (50 mL), washed with ammonium chloride solution, and dried over anhydrous Na₂SO₄. The crude mixture was purified through column chromatography to afford the title compound. Yellow oil; yield 81%; R_f 0.78 (EtOAc/hexane

1:1); $[\alpha]_D^{25}$ +11.4 (c 11, CHCl₃). IR (neat): 1640 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): 0.92 (d, *J* = 6.6 Hz, 3H); 0.95 (d, *J* = 6.4 Hz, 3H); 1.23-1.19 (m, 1H); 1.51-1.42 (m, 2H); 2.71-2.45 (m, 6H); 3.52-3.32 (m, 6H); 4.40 (d, *J* = 11.1 Hz, 1H); 4.50 (d, *J* = 11.3 Hz, 1H); 4.92 (s, 1H); 7.33-7.13 (m, 10H). ¹³C NMR (125.75 MHz, CDCl₃): 22.2, 23.4, 25.0, 37.7, 38.8, 43.7, 50.6, 51.0, 56.3, 62.8, 71.5, 72.7, 127.1, 127.5, 127.6, 128.3, 128.6, 128.9, 138.0, 138.2, 175.1. HRMS calcd for C₂₅H₃₅N₂O₂ (M⁺+H): 395.2699; found, 395.2679.

(S)-1,6-Dibenzyl-4-[1'-(benzyloxy)-4'-methylpentan-2'-yl]-1,4-diazepan-5-one. To a solution of the above *O*-benzyl 1,4-diazepan-5-one (0.540 g, 1.36 mmol) in THF/HMPA mixture (5 mL/1 mL) at -78 °C was added LiHMDS (1.15 g, 6.84 mmol). After being allowed to stir for 15 min, benzyl bromide (0.325 mL, 2.73 mmol) was added dropwise. The reaction mixture was allowed to stir at -78 °C for 1 h, when it was allowed to warm to room temperature and allowed to stir for 2 h. The reaction mixture was diluted with saturated aqueous NH₄Cl and extracted with ethyl acetate (50 mL). The organic layer was washed with brine and water and dried over anhydrous Na₂SO₄. Column chromatography afforded the desired compound as a mixture of diastereomers (2:1). Yellow oil; yield 56%; *R_f* 0.70 (EtOAc, 100%); IR (neat): 1646 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): 1.08-0.85 (m, 6H); 1.31 (m, 1H); 1.32-1.31 (m, 2H); 2.09 (br s, 2H); 2.84 (br s, 1H); 2.86 (d, *J* = 9.5 Hz, 2H); 3.16 (m, 1H); 3.56-3.36 (m, 8H); 4.60-4.48 (m, 2H); 4.60 (br s, 1H) 7.93-7.22 (m, 15H). ¹³C NMR (125.75 MHz, CDCl₃): 22.1, 23.2, 25.0, 36.0, 37.5, 38.3, 42.7, 46.1, 50.6, 55.2, 56.0, 56.4, 62.6, 71.1, 71.9, 72.8, 125.8, 125.8, 126.9, 127.5, 127.6, 127.7, 128.2, 128.2, 129.0, 138.2, 140.3, 175.9, 176.3. HRMS calcd for C₃₂H₄₁N₂O₂ (M⁺+H): 485.3168; found, 485.3141.

(S)-1,6-Dibenzyl-4-(1'-hydroxy-4'-methylpentan-2'-yl)-1,4-diazepan-5-one (15).

Mixture of diastereomers (2:1), yellow oil; yield 63%; R_f 0.27 (EtOAc, 100%); IR (neat): 3324, 1639 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): 0.95-0.87 (m, 6H); 1.01-1.00 (m, 1H); 1.43 (m, 2H); 1.84 (s, 1H); 2.68-2.66 (m, 1H); 3.01-2.89 (m, 1H); 3.46-3.29 (m, 6H); 4.60-4.44 (m, 2H); 5.08 (br s, 1H). ^{13}C NMR (125.75 MHz, CDCl_3): 21.9, 22.5, 23.1, 25.0, 35.7, 37.5, 38.3, 47.6, 48.5, 70.9, 72.7, 72.8, 125.8, 127.5, 128.3, 129.1, 137.9, 138.1, 140.3, 175.6. HRMS calcd for $\text{C}_{25}\text{H}_{35}\text{N}_2\text{O}_2$ ($\text{M}^+ + \text{H}$): 395.2699; found, 395.2688.

Solid Phase Synthesis of (S)-3-[7-Oxo-1,4-diazepan-1-yl]propyl-2-(tert-butoxycarbonyl)-3-phenylpropanoate hydro chloride (16). A mixture of Merrifield resin (2 g, 3 mmol), piperidone hydrochloride (1.4g, 9 mmol) and diisopropylethylamine (2.6 mL, 15 mmol) in DMF (15mL) was shaken at 50 °C for 12 h. The resin was filtered and then resubjected to the above procedure to ensure complete loading. The resin was then washed with DMF (100 mL), CH_2Cl_2 (100 mL), and ether (100 mL) and dried under vacuum for several hours to a constant weight of 2.42 g. To a suspension of above resin and 1-azidopropan-3-ol (1.7 g, 9 mmol) in CH_2Cl_2 was added $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (1.9 mL, 15 mmol) at room temperature. After shaking for 1 day, it was filtered and treated with 15% KOH (10 mL) in THF (10 mL) for 2 h. The resin was filtered, washed with water (200 mL), THF (100 mL), CH_2Cl_2 (100 mL), and ether (100 mL), and then dried. A mixture of the resulting resin (1.1 g, 1.2 mmol, 1 equiv) and BocN-Phe-OH (0.95 g, 3.6 mmol), and DMAP (0.04 g, 0.3 mmol, 0.1 equiv) in CH_2Cl_2 (10mL) was shaken together at room temperature for a day. The resin was filtered, washed with water (100 mL), aqueous NaHCO_3 (100 mL), MeOH (100 mL), THF (100 mL), CH_2Cl_2 (100 mL) and ether (100 mL), and dried to constant weight. The resin was suspended in CH_2Cl_2 (10 mL) and allowed to

stand for 5 min. Chloroethylchloroformate (0.4 mL, 3.7 mmol) was introduced and the suspension allowed to stir for 12 h, after which time the resin was filtered off and washed with CH₂Cl₂. The residue obtained after the concentration of filtrate was subjected to flash chromatography with EtOAc to furnish 0.21 g of the chloroformate derivative. This was allowed to reflux in 5 mL of methanol for 1 h, after which evaporation of the solvent furnished 0.19 g of title compound (33% yield from piperidinone). White foam; yield 33%; IR (neat): 3406, 1718, 1637 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): 1.17-1.11 (m, 2H); 1.41 (s, 9H); 1.93-1.59 (m, 6H); 3.06 (s, 4H); 3.78-3.37 (m, 6H); 4.07 (br s, 1H); 4.52-4.44 (m, 2H), 5.08 (d, *J* = 7.3 Hz, 1H); 7.31-7.17 (m, 5H). ¹³C NMR (100.6 MHz, CDCl₃): 25.3, 25.9, 27.5, 28.7, 34.3, 34.6; 38.7; 46.2; 49.6, 55.0, 62.8, 80.4, 127.4, 129.0, 129.6, 136.4, 155.6, 172.3. HRMS calcd for C₂₂H₃₄N₃O₅ (M⁺+H): 420.2498; found, 420.2487.

References

1. (a) Giannis, A; Sandhoff, K. *Angew Chem., Int. Ed. Engl.* **1989**, 28, 218-220. (b) Qualgliato, D.A.; Andrae, P.M.; Matelan, E.M. *J. Org. Chem.* **2000**, 65, 5037-5042. (c) Meyers, A.I.; McKennon, M.J. *J. Org. Chem.* **1993**, 58, 3568-3571.
2. (a). Zaloom, J.; Roberts, D.C. *J. Org. Chem.* **1981**, 46, 5173-5176 (b). Vasella, A.; Witzig, C.; Chiara, J.L.; Lomas, M. M. *Helv. Chim. Acta.* **1991**, 74, 2073-2077.
3. Cooper, C.S.; Klock, P.L.; Chu, D.T.W.; Hardy, D.J.; Swanson, R. N.; Plattner, J.J. *J. Med. Chem.* **1992**, 35, 1392-1398.

DS 2
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RG 64
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DE 4.50 usec
TE 300.0 K
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SI-17

1a

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DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
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P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
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TE 300.0 K
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F2 - Processing parameters
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Current Page

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LB 0.30 Hz
GB 0
PC 1.00

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CY 12.50 cm
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F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

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===== CHANNEL f2 =====
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NAI
EXI
PRI
F2
Da:
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IN
PR
PU
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SO
NS
DS
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DW 104.400 usec
DE 4.50 usec
TE 300.0 K
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===== CHANNEL f1 =====
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===== CHANNEL f2 =====
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F2 - Processing parameters
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CY 12.50 cm
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F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

EXPNO
PROCNO

F2 -
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Time_
INST
PROG
PUL
TD
SQ
NS
DE
S
F
I

D1 0.0300000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SF01 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
MDM EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 GF
SF 500.1300000 MHz
MDM OSINE
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 10.008 ppm
F1 5005.33 Hz
F2P -2.008 ppm
F2 -1004.29 Hz
PPNCH 0.60081 ppm/cm
HZCH 300.48077 Hz/cm

NAME
EXPNO
PROCNO

F2 - Acquisition Parameters
Date_ 20031229
Time 19.52
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 247
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 2048
DM 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SF01 125.7719472 MHz

***** CHANNEL f2 *****
CPOPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SF02 500.1325000 MHz

F2 - Processing parameters
SI 65536
SF 125.7578008 MHz
MDM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 GF
SF 500.1300000 MHz
MDM no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 242.517 ppm
F1 30498.43 Hz
F2P -7.539 ppm
F2 -948.11 Hz
PPNCH 12.50282 ppm/cm
HZCH 1572.32703 Hz/cm

CAPPU
PROCNO

F2 - Acqui
Date
Time
INSTR
PROB
PULPR
TO
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
ON
DE
TE
D1

32768
CDC13
16
2
6009.615 Hz
0.183399 Hz
2.7264309 sec
32
83.200 usec
6.00 usec
300.0 K
0.03000000 sec

1g

***** CHANNEL f1 *****
NUC1
P1
PL1
SFO1

1H
7.70 usec
-4.00 dB
500.132005 MHz

F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC

32768
500.1300000 MHz
EM
0
0.30 Hz
0
1.00

F1 - Processing parameters
SI
MC2
SF
WDW
SSB
LB
GB

1024
OF
500.1300000 MHz
GSI
0
0.30 Hz
0

1D NMR plot parameters
CX
CY
F1P
F1
F2P
F2
PPMCH
HZCM

20.00 cm
0.00 cm
11.000 ppm
5501.43 Hz
-1.000 ppm
-500.13 Hz
0.60000 ppm/cm
300.07800 Hz/cm

NAME
EXPNO
PROCNO

F2 - Acquisition Parameters
Date
Time
INSTRUM
PROBHD
PULPROG
TO
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
ON
DE
TE
D1
d11
d12

20031210
19.16
spect
5 mm BBO BB-1H
zgpg30
65536
CDC13
248
4
31446.541 Hz
0.479836 Hz
1.0420883 sec
2048
15.900 usec
6.00 usec
300.0 K
0.10000000 sec
0.03000000 sec
0.00002000 sec

***** CHANNEL f1 *****
NUC1
P1
PL1
SFO1

13C
6.50 usec
5.00 dB
125.7719472 MHz

***** CHANNEL f2 *****
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
SFO2

waltz16
1H
95.00 usec
-4.00 dB
19.00 dB
30.00 dB
500.1325000 MHz

F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC

65536
125.7578008 MHz
EM
0
1.00 Hz
0
1.40

F1 - Processing parameters
SI
MC2
SF
WDW
SSB
LB
GB

1024
OF
500.1300000 MHz
no
0
0.30 Hz
0

1D NMR plot parameters
CX
CY
F1P
F1
F2P
F2
PPMCH
HZCM

20.00 cm
0.00 cm
242.517 ppm
30498.43 Hz
-7.539 ppm
-948.11 Hz
12.50282 ppm/cm
1572.32703 Hz/cm

1h

FIDRES 0.183399 Hz
AQ 2.7264309 sec
RG 32
DW 83.200 usec
DE 6.00 usec
TE 296.2 K
D1 0.03000000 sec
HOREST 0.00000000 sec
HOREX 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SF01 500.1320005 MHz

F1 - Acquisition parameters
ND0 2
TO 256
SF01 500.1325 MHz
FIDRES 23.475060 Hz
SM 12.016 ppm
FNAME undefined

F2 - Processing parameters
S1 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
S1 1024
MC2 OF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 11.000 ppm
F1 5501.43 Hz
F2P -1.000 ppm
F2 -500.13 Hz
PPMCH 0.60000 ppm/cm
HZCM 300.07800 Hz/cm

X
E
PNAME

F2 - Acquisition Parameters
Date 20040519
Time 18.56
INSTRUM spect
PROBHD 5 mm BBO 95-1H
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
MS 224
NS 4
SM 31445.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420803 sec
RG 2048
DW 15.900 usec
DE 6.00 usec
TE 296.2 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
HOREST 0.00000000 sec
HOREX 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SF01 125.7687360 MHz

***** CHANNEL f2 *****
DPPROG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SF02 500.1325000 MHz

F1 - Acquisition parameters
ND0 2
TO 256
SF01 500.1325 MHz
FIDRES 23.475060 Hz
SM 12.016 ppm
FNAME undefined

F2 - Processing parameters
S1 65536
SF 125.7578000 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
S1 1024
MC2 OF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 242.517 ppm
F1 30498.43 Hz
F2P -7.539 ppm
F2 -948.11 Hz
PPMCH 12.50262 ppm/cm
HZCM 1572.32703 Hz/cm

2d

```

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.132000 MHz

F1 - Acquisition parameters
AQ 2
TO 256
SFO1 500.1325 MHz
FIDRES 23.475060 Hz
SM 12.016 ppm
FWDDE undefined

F2 - Processing parameters
SI 32768
SF 500.130000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
HC2 OF
SF 500.130000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

ID MMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 11.000 ppm
F1 5501.43 Hz
F2 -1.000 ppm
F2 500.13 Hz
PWCN 0.60000 ppm/cm
H2CN 300.07800 Hz/cm

```

```

G
H
F
LC
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 5.00 dB
SFO1 125.768750 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 30.00 dB
SFO2 500.132500 MHz

F1 - Acquisition parameters
AQ 2
TO 256
SFO1 500.1325 MHz
FIDRES 23.475060 Hz
SM 12.016 ppm
FWDDE undefined

F2 - Processing parameters
SI 65536
SF 125.757000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.48

F1 - Processing parameters
SI 1024
HC2 OF
SF 500.130000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

ID MMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 242.511 ppm
F1 30496.43 Hz
F2 -7.530 ppm
F2 948.11 Hz
PWCN 12.50200 ppm/cm
H2CN 1572.32703 Hz/cm

```

NS 16
DS 2
SMH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 11.0000 ppm
F1 4401.43 Hz
F2P -1.0000 ppm
F2 -400.13 Hz
PPHCH 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

Current
NAME
EXPNO
PROCNO

F2 - Acquisition Parameters
Date_ 20031031
Time 9.31
INSTRUM drx400
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 172
DS 2
SMH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 215.0000 ppm
F1 21631.74 Hz
F2P -5.0000 ppm
F2 -503.06 Hz
PPHCH 11.00000 ppm/cm
HZCM 1142.33000 Hz/cm

Hz

NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 15384
SP 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FIP 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

Cur
NAME
EXPNO
PROCNO

F2 - Acquisition Parameters
Date_ 20031129
Time 17.10
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 173
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SP 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FIP 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm

RG 32
DN 83.200 usec
DE 8.00 usec
TE 296.2 K
D1 0.03000000 sec
HOREST 0.00000000 sec
HOREK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -1.00 dB
SFO1 500.1320005 MHz

F1 - Acquisition parameters
AQ 2
TD 256
SFO1 500.1320005 MHz
FIDRES 23.475060 Hz
SF 12.016 ppm
FWDCE undefined

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
HC2 OF
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

1D 1H plot parameters
CI 20.00 cm
CY 0.00 cm
FIP 8.000 ppm
F1 4001.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHCH 0.40000 ppm/cm
H2CH 200.05200 Hz/cm

Current
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_ 20040519
Time 12.50
INSTR msi
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 432
DS 4
SH 3146.541 Hz
FIDRES 0.479836 Hz
AQ 1.8400000 sec
RG 2048
DN 15.900 VWH
DE 6.00 VWH
TE 296.2 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
HOREST 0.00000000 sec
HOREK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.767360 MHz

===== CHANNEL f2 =====
NAMEZ msi16
NUC2 1H
PCPDZ 95.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 30.00 dB
SFO2 500.1320000 MHz

F1 - Acquisition parameters
AQ 2
TD 256
SFO1 500.1320000 MHz
FIDRES 23.475060 Hz
SF 12.016 ppm
FWDCE undefined

F2 - Processing parameters
SI 65536
SF 125.767360 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
HC2 OF
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

1D 1H plot parameters
CI 20.00 cm
CY 0.00 cm
FIP 242.517 ppm
F1 30400.13 Hz
F2P -7.530 ppm
F2 -940.11 Hz
PPHCH 12.50202 ppm/cm
H2CH 1572.32783 Hz/cm

C N E V
F O I P P T E A E
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7284309 sec
RG 32
DM 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.0300000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320005 MHz

F2 - Processing parameters
S1 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
S1 1024
MC2 OF
SF 500.1300000 MHz
WDW QSINE
SSB 0
LB 0.30 Hz
GB 0

10 NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 8.000 ppm
F1 4001.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PWHCH 0.40000 ppm/cm
HZCH 208.05200 Hz/cm

Current Data Parameters
NAME
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031210
Time 19.04
INSTRUM spect
PROBHD 5 mm BBO BB-H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 297
DS 4
SWH 31446.341 Hz
FIDRES 0.478036 Hz
AQ 1.0420883 sec
RG 2048
DM 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.7719472 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SFO2 500.1325000 MHz

F2 - Processing parameters
S1 65536
SF 125.7570000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
S1 1024
MC2 OF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

10 NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 180.000 ppm
F1 22636.40 Hz
F2P 0.000 ppm
F2 0.00 Hz

C1
N
E
FNAME

F2 - Acquisition Parameters

Date_ 20031215
Time 19.04
INSTRUM spect
PROBHD 5 mm BBO 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 244
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420803 sec
RG 2048
DM 15.500 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.7719472 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 55.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SFO2 500.1325000 MHz

F2 - Processing parameters

SI 65536
SF 125.7578008 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters

SI 1024
HC2 OF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

10 NMR plot parameters

CX 20.00 cm
CY 0.00 cm
F1 242.517 ppm
F1 30498.43 Hz
F2P -7.529 ppm
F2 -948.11 Hz
PPHCH 12.50282 ppm/cm
HZCH 1572.32703 Hz/cm

***** CHANNEL 11 *****
 NUC1 1H
 P1 7.72 usec
 PL1 4.00 dB
 SFO1 500.132000 MHz

F1 Acquisition parameters
 WD 2
 IN 256
 SF 500.1320 MHz
 FIDRES 23.475060 Hz
 SFO 12.016 MHz
 FWHM 0.0150000 sec

F2 Processing parameters
 SI 32768
 SF 500.130000 MHz
 WD 64
 IN 32
 SF 0.30 Hz
 FID 0
 PC 1.00

F3 Processing parameters
 SI 1024
 NUC2 13C
 P1 500.130000 MHz
 IN 6514
 SF 125.760360 MHz
 FIDRES 0.03 Hz
 SFO 0.40000 MHz
 FWHM 0.03000 MHz
 PC 200.05200 MHz

Current Acquisition Parameters
 NAME
 EXPNO
 PROCNO

F2 Acquisition Parameters
 Date_ 2004/01/18
 Time 18:17
 INSTRUM spect
 PROBHD 5 mm BBO 1H-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 227
 DS 4
 SWH 31446.541 Hz
 FIDRES 0.479826 Hz
 AQ 1.0420803 sec
 RG 256
 DM 15.900 usec
 DR 6.00 usec
 TE 298.4 K
 D1 0.15000001 sec
 D11 0.03000000 sec
 DELTA 0.05000000 sec
 INCRST 0.00000000 sec
 MCNTR 0.01500000 sec

***** CHANNEL 11 *****
 NUC1 1H
 P1 7.50 usec
 PL1 5.00 dB
 SFO1 125.760360 MHz

***** CHANNEL 12 *****
 CPMPRG2 waltz16
 NUC2 13C
 PCPD2 95.00 usec
 PL2 4.00 dB
 PL12 19.00 dB
 PL13 30.00 dB
 SFO2 500.132000 MHz

F2 Processing parameters
 SI 82538
 SF 125.7578000 MHz
 WD 64
 IN 32
 SF 0.30 Hz
 FID 0
 PC 1.40

F3 Processing parameters
 SI 20.00 MHz
 CY 0.00 MHz
 FID 100.000 ppm
 F1 22536.40 Hz
 F2 0.350 MHz
 F3 0.00 MHz
 FWHM 9.99900 MHz
 PC 1131.00000 MHz

SWH 400.132000 MHz
FIDRES 0.146157 Hz
AQ 3.4210297 sec
RG 32
DN 104.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 7.05 usec
PL1 -3.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 8.000 ppm
F1 3201.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCH 0.40000 ppm/cm
HZCM 160.05200 Hz/cm

Cur

NAME

EXP

PROCNO

F2 - Acquisition Parameters
Date 20041104
Time 9.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 282
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DN 20.050 usec
DE 6.00 usec
TE 298.2 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6225298 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.40 usec
PL2 -3.00 dB
PL12 19.96 dB
PL13 120.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 180.000 ppm
F1 18110.30 Hz
F2P 20.000 ppm
F2 2012.26 Hz
PPMCH 8.00000 ppm/cm
HZCM 804.90216 Hz/cm

Hz

14

NAME: CHANTEL 11

INSTRUM: spect

PROBHD: 5 mm CPDZ 13C

PULPROG: zgpg30

TD: 65536

SOLVENT: CDCl3

NS: 1871

DS: 4

SWH: 3140.541 Hz

FIDRES: 0.47808 Hz

AQ: 1.84200000 sec

RG: 13084

OP: 19.300 usec

DE: 6.00 usec

TE: 298.0 K

D1: 0.15000001 sec

DELTA: 0.05000000 sec

HCOREST: 0.00000000 sec

MCNMR: 0.01500000 sec

----- CHANNEL f1 -----

MU1: 13C

PI: 10.00 usec

PL1: -5.10 dB

SFO1: 125.767560 MHz

----- CHANNEL f2 -----

CPDPRG2: zgpg30

MU2: 1H

PI2: 00.00 usec

PL2: -5.10 dB

PL12: 10.00 dB

PL13: 10.00 dB

SFO2: 500.132000 MHz

----- Acquisition parameters -----

NUC1: 13C

NUC2: 1H

TO: 256

SFO1: 500.132000 MHz

FIDRES: 23.475060 Hz

SM: 12.016 ppm

FMODE: undetuned

----- Processing parameters -----

S1: 32768

SF: 500.1300000 MHz

WDW: EM

SSB: 0

LB: 0.30 Hz

GB: 0

PC: 1.00

----- Processing parameters -----

S1: 1024

HC2: OF

SF: 500.1300000 MHz

WDW: EM

SSB: 0

LB: 0.30 Hz

GB: 0

----- 1D NMR plot parameters -----

CE: 20.00 cm

CY: 0.00 cm

FIP: 0.000 ppm

F1: 4001.04 Hz

F2: 0.000 ppm

F2: 0.00 Hz

PPMCH: 0.40000 ppm/cm

H1CH: 200.05200 1/1/cm

----- Acquisition Parameters -----

NAME: 2004023

TIME: 10.10

INSTRUM: spect

PROBHD: 5 mm CPDZ 13C

PULPROG: zgpg30

TD: 65536

SOLVENT: CDCl3

NS: 1871

DS: 4

SWH: 3140.541 Hz

FIDRES: 0.47808 Hz

AQ: 1.84200000 sec

RG: 13084

OP: 19.300 usec

DE: 6.00 usec

TE: 298.0 K

D1: 0.15000001 sec

DELTA: 0.05000000 sec

HCOREST: 0.00000000 sec

MCNMR: 0.01500000 sec

----- CHANNEL f1 -----

MU1: 13C

PI: 10.00 usec

PL1: -5.10 dB

SFO1: 125.767560 MHz

----- CHANNEL f2 -----

CPDPRG2: zgpg30

MU2: 1H

PI2: 00.00 usec

PL2: -5.10 dB

PL12: 10.00 dB

PL13: 10.00 dB

SFO2: 500.132000 MHz

----- Acquisition parameters -----

NUC1: 13C

NUC2: 1H

TO: 256

SFO1: 500.132000 MHz

FIDRES: 23.475060 Hz

SM: 12.016 ppm

FMODE: undetuned

----- Processing parameters -----

S1: 65536

SF: 125.7576000 MHz

WDW: EM

SSB: 0

LB: 1.00 Hz

GB: 0

PC: 1.40

----- Processing parameters -----

S1: 1024

HC2: OF

SF: 500.1300000 MHz

WDW: EM

SSB: 0

LB: 0.30 Hz

GB: 0

----- 1D NMR plot parameters -----

CE: 20.00 cm

CY: 0.00 cm

FIP: 180.000 ppm

F1: 23803.36 Hz

F2: -10.000 ppm

F2: -1257.56 Hz

PPMCH: 10.00000 ppm/cm

H1CH: 125.71800 1/1/cm

D1 0.0300000 sec
 ----- CHANNEL f1 -----
 NUCL1 1H
 P1 7.70 usec
 PL1 -4.00 dB
 SF01 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300000 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 1024
 MC2 OF
 SF 500.1300000 MHz
 MDW OSINE
 SSB 0
 LB 0.30 Hz
 GB 0

ID NMR plot parameters
 CX 20.00 cm
 CY 0.00 cm
 FIP 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm

PROBHD 1
 F2 - Acquisition Parameters
 Date_ 20040107
 Time 10:27
 INSTRUM spect
 PROBHD 5 mm BBO BB-H
 PULPROG zgpg30
 TO 69536
 SOLVENT CDCl3
 NS 256
 DS 4
 SFO1 31446.541 Hz
 FIDRES 0.470836 Hz
 AQ 1.0400003 sec
 RG 2048
 DN 15.900 usec
 DE 6.00 usec
 TE 300.0 K
 G1 0.10000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

----- CHANNEL f1 -----
 NUCL1 13C
 P1 6.50 usec
 PL1 5.00 dB
 SF01 125.7719472 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUCL2 1H
 PPRG2 95.00 usec
 PL2 -4.00 dB
 PL12 19.00 dB
 PL13 30.00 dB
 SF02 500.1325000 MHz

F2 - Processing parameters
 SI 69536
 SF 125.7578000 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 OF
 SF 500.1300000 MHz
 MDW no
 SSB 0
 LB 0.30 Hz
 GB 0

ID NMR plot parameters
 CX 20.00 cm
 CY 0.00 cm
 FIP 242.517 ppm
 F1 30490.43 Hz
 F2 7.539 ppm
 F2 -940.11 Hz
 PPMCM 12.50202 ppm/cm
 HZCM 1572.32703 Hz/cm

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DM 83.200 usec
DE 5.00 usec
TE 300.0 K
D1 0.03000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SF01 500.1320005 MHz

F2 - Processing parameters
S1 32768
SF 500.1300000 MHz
MDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
S1 1024
MC2 OF
SF 500.1300000 MHz
MDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 10.008 ppm
F1 5005.33 Hz
F2P -2.008 ppm
F2 -1004.29 Hz
PPMCH 0.65081 ppm/cm
H2CH 300.48077 Hz/cm

PROBHD
F2 - Acquisition Parameters
Date_ 20040313
Time_ 9.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 247
DS 4
SMA 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 2048
RG 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 3.00 dB
SF01 125.7719472 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SF02 500.1325000 MHz

F2 - Processing parameters
S1 65536
SF 125.7578000 MHz
MDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
S1 1024
MC2 OF
SF 500.1300000 MHz
MDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 242.517 ppm
F1 30498.43 Hz
F2P -7.538 ppm
F2 -948.11 Hz

Time 16
INSTRUM dr
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 101.6
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SP 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 11.000 ppm
F1 4401.43 Hz
F2P 0.500 ppm
F2 200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020108
Time_ 23.44
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 466
DS 2
SWH 23148.148 Hz
FIDRES 0.153213 Hz
AQ 1.4156276 sec
RG 4096
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6212931 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SP 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FIP 220.000 ppm
F1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm