Asymmetric Schmidt Reaction of Hydroxyalkyl Azides with Ketones

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Synthesis and Characterization of Hydroxyalkyl Azides

Synthesis of Hydroxyalkyl Azides (General Procedure A). A mixture of halide and sodium azide (1.5-3 eq) in DMF was stirred for 18 h at 25 °C. The reaction mixture was partitioned between Et₂O and water. The layers were separated and the organic layer was washed with water, brine, dried (Na₂SO₄) and filtered. The filtrate was concentrated in vacuo and the residue purified by chromatography using 10-50% EtOAc/hexanes or 10-50% Et₂O in petroleum ether as eluent.

Synthesis of Hydroxyalkyl Azides (General Procedure B). A solution of the alcohol dissolved in THF was cooled to 0 °C. PPh₃ (1.1 eq), hydrazoic acid (ca. 1.25 M in benzene) (1.2 eq) and diethyl azodicarboxylate (DEAD) (1.1 eq) were added sequentially. The reaction was warmed to ambient temperature and stirred for 18 h. The reaction mixture was partitioned between Et₂O and water. The layers were separated and the organic layer was washed with water, brine, dried (Na₂SO₄) and filtered. The filtrate was concentrated in vacuo and the residue purified by chromatography using 10-50% EtOAc/hexanes or 10-50% Et₂O/petroleum ether as eluent.

(R)-2-Azido-2-phenyl-ethanol (1). (*S*)-1-phenyl-1,2-dihydroxyethane¹ (0.5 g, 3.6 mmol) was reacted using procedure B to give the title compound (0.491 g, 84%) as a transparent oil in ≥99.5% ee as determined by HPLC. $[\alpha]_D = -221.1$ (*c*, 1.35, CHCl₃).

TLC (3:1 hexane/EtOAc): $R_f = 0.45$; ^1H NMR (400 MHz, CDCl_3) δ 1.93-2.02 (m, 1H), 3.76 (t, $J = 6.3$, 2H), 4.68 (t, $J = 6.0$ Hz, 1H), 7.31-7.44 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 66.5, 67.9, 127.1, 128.7, 129.0, 136.2. IR (neat) 3350, 2090 cm^{-1} . HPLC: (*R*) $t_R = 11.8$ min, (*S*) $t_R = 12.8$ min; Chiracel OD-H; 94% hexane/*i*-PrOH; flow rate 0.5 mL/min; UV 254 nm.

(1*S*, 2*R*)-2-Azido-1,2-diphenylethanol (2). Prepared according to literature precedent.²

(±)-2-Azido-1-phenylethanol ((±)-3). A flask was charged with (±)-styrene oxide (9.48 mL, 83.2 mmol), NaN_3 (7 g, 100 mmol), NH_4Cl (5.76 g, 108 mmol), H_2O (20 mL), and EtOH (80 mL). The flask was fitted with a condenser and the reaction was heated at reflux for 3 h. The reaction was cooled to room temperature and then concentrated. The reaction was diluted with 3:1 hexane/EtOAc (200 mL), dried (MgSO_4), filtered and concentrated. Flash chromatography (3:1 hexane/EtOAc) gave the title compound (2.73 g, 20%) and (±)-2-azido-2-phenylethanol (8.92 g, 66%) as transparent yellow oils. TLC (3:1 hexane/EtOAc): R_f ((±)-3) = 0.48, R_f (1) = 0.61. ^1H NMR (400 MHz, CDCl_3) δ 2.35 (br s, 1H), 3.42-3.52 (m, 2H), 4.87-4.91 (m, 1H), 7.30-7.43 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 58.1, 73.5, 125.9, 128.4, 128.7, 140.5. IR (neat) 3400, 2100 cm^{-1} .

(±)-2-Azido-2-(3,4,5-trimethoxyphenyl) ethanol (4). Yellowish oil. ^1H NMR (500 MHz, CDCl_3) δ 3.79 (m, 2H), 3.86 (s, 3H), 3.89 (s, 6H), 4.57 (dd, $J = 7.2, 5.6$ Hz, 1H), 6.55 (s, 2H); ^{13}C NMR (500 MHz, CDCl_3) δ 56.1, 56.2, 60.7, 66.5, 104.1, 131.9, 138.1, 153.5; IR (neat) 3495, 2960, 2120 cm^{-1} ; MS (CI) m/e 254 ($\text{M}^+ + 1$), 236, 211; HRMS calcd for $\text{C}_{11}\text{H}_{15}\text{N}_3\text{O}_4$ ($\text{M}^+ + 1$): 253.1063, found 253.1058.

(R)-3-Azido-1-phenylpropanol (5). Procedure A on (*R*)-3-chloro-1-phenylpropanol (Aldrich, 2.5 g, 14.6 mmol) gave a transparent yellow-tinged oil (2.56 g, 99%). $[\alpha]_{\text{D}} = +36.3$ (c 0.99, CHCl_3). TLC (3:1 hexane/EtOAc): $R_f = 0.5$. ^1H NMR (400 MHz, CDCl_3) δ 1.91-2.10 (m, 3H), 3.35-3.53 (m, 2H), 4.84 (dd, $J = 8.4, 4.7$ Hz, 1H), 7.28-7.39 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 37.8, 48.4, 71.8, 125.7, 127.9, 128.7, 143.8. IR (neat) 3370, 2065 cm^{-1} . HPLC: $t_{\text{R}}(\text{R}) = 10.2$ min, $t_{\text{R}}(\text{S}) = 9.2$ min; Chiracel OD-H; 95% hexane/*i*-PrOH; flow rate 1 mL/min; UV 254 nm).

(S)-3-Acetoxy-1-phenyl-1-propanol (A). A mixture of (*S*)-3-chloro-2-phenyl-1-propanol (Aldrich, 0.5 g, 2.92 mmol), NaOAc (0.72 g, 8.83 mmol) and NaI (0.44 g, 2.94 mmol) was heated at 120-130 $^{\circ}\text{C}$ in 20 mL DMF for 24 h. The reaction was cooled and diluted with Et_2O (20 mL) and water (15 mL). The layers were separated and the organic

layer was washed with water (2x10 mL), brine, dried (Na₂SO₄) and filtered. The filtrate was concentrated in vacuo to an oil which was subjected to flash column chromatography using 20% EtOAc/hexanes as eluent to provide 0.34 g (60%) of **A** as a pale yellow oil. R_f = 0.4 (50% EtOAc/hexane). $[\alpha]_D = -21.3$ (c 1.75, CHCl₃). IR (CH₂Cl₂) 3520, 1820 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.31-7.19 (m, 5H), 4.67 (dd, J = 5.3, 8.1 Hz, 1H), 4.19-4.13 (m, 1H), 4.06-3.99 (m, 1H), 3.56 (br s, 1H), 1.99-1.88 (m, 2H), 1.94 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 171.8, 144.5, 128.9, 127.9, 126.2, 71.4, 62.1, 38.2, 21.3; MS (CI) 194 (M^+ +1, 20); HRFABMS calcd for C₁₁H₁₅O₃ (M^+ +1): 195.1021; found: 195.1018.

(R)-3-Azido-3-phenyl-1-propanol (6). Reaction of **A** (103 mg, 0.53 mmol), PPh₃ (140 mg, 0.53 mmol) using procedure B, followed by hydrolysis with K₂CO₃/MeOH, gave **6** as a colorless oil. R_f = 0.3 (50% EtOAc/hexane). $[\alpha]_D = +192.7$ (c 0.95, CHCl₃). IR (CH₂Cl₂) 3520, 1820 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.54-7.23 (m, 5H), 4.73 (dd, J = 5.7, 8.8 Hz, 1H), 3.81-3.66 (m, 2H), 2.38 (br s, 1H), 2.15-1.83 (m, 2H); ¹³C NMR (100.6 MHz, CDCl₃) δ 139.7, 129.3, 128.8, 127.3, 63.8, 60.0, 39.1; MS (CI) 178 (M^+ +1, 20); HRFABMS calcd for C₉H₁₂N₃O (M^+ +1): 178.0980; found: 178.0979.

(±)-3-Triisopropylsiloxy-1-(3,4,5-trimethoxyphenyl)propanol (B). TIPSCl (1.75 mL, 8.18 mmol) was added to a solution of 1-(3,4,5-trimethoxyphenyl)-1,3-propanediol³ (1.8 g, 7.43 mmol) and imidazole (1.11g, 16.36 mmol) in 25 mL DMF and the reaction mixture was stirred at room temperature for 6 h.⁴ The reaction mixture was partitioned between Et₂O and water and the organic layer was washed with water (5 x 20 mL) and then brine and dried (Na₂SO₄). Filtration followed by concentration in vacuo furnished an oil which upon flash column chromatography using 10→25 % EtOAc/hexanes as eluent afforded 2.6 g (88%) of **B** as a clear oil. R_f = 0.3 (25% EtOAc/hexane). IR (CH₂Cl₂) 3360 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 6.62 (s, 2H), 4.93 (dd, J = 3.1, 7.9 Hz, 1H), 4.09 (br s, 1H), 3.98-3.85 (m, 2H), 3.87 (s, 6H), 3.83 (m, 3H), 2.00-1.91 (m, 2H), 1.10 (s, 18H), 1.09 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 153.1, 140.3, 136.7, 102.4, 74.4, 62.9, 60.8, 55.9, 40.5, 17.9, 11.7. MS (+FAB) 398 (M⁺, 17); HRFABMS calcd for C₂₁H₃₈O₅Si (M⁺): 398.2567; found: 398.2541.

(±)-3-Azido-3-(3,4,5-trimethoxyphenyl)propanol ((±)-7). Reaction of **B** via procedure B gave (±)-**7** as a clear oil. R_f = 0.25 (50% EtOAc/hexane). IR (CH₂Cl₂) 3380,

2030 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 6.54 (s, 2H), 4.65 (dd, $J = 5.6, 8.8$ Hz, 1H), 3.89 (br s, 1H), 3.89 (s, 6H), 3.85 (s, 3H), 3.85-3.67 (n, 2H), 2.03-1.91 (m, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 153.4, 137.6, 135.1, 103.6, 63.5, 60.7, 59.4, 56.0, 38.8. MS (+FAB) 267 (M^+ , 22); HRFABMS calcd for $\text{C}_{12}\text{H}_{17}\text{N}_3\text{O}_4$ (M^++1): 267.1219; found: 267.1224.

8

(*R*)-3-Azido-2-methyl-1-propanol (8): Reaction of (*S*)-3-bromo-2-methyl-1-propanol (Aldrich, 200 μL , 1.90 mmol) according to procedure A gave 180 mg (82%) of **8** as an oil. $R_f = 0.3$ (25% EtOAc/hexane). $[\alpha]_D = +6.6$ (c 1.3, CHCl_3); IR (CH_2Cl_2) 3300, 2060 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.64-3.52 (m, 2H), 3.41-3.32 (m, 2H), 1.98-1.93 (m, 1H), 1.80 (br s, 1H), 0.99 (d, $J = 6.9$ Hz, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 65.6, 55.0, 36.3, 14.8; MS (CI) 116 (M^++1 , 50); HRFABMS calcd for $\text{C}_4\text{H}_9\text{N}_3\text{O}$ (M^+): 115.0746; found 115.0747.

C

(4*S*)-3-(Phenylacetyl)-4-(phenylmethyl)-2-oxazolidinone (C) is a known compound.⁵

D

(4*S*,2'*R*)-3-((2'-Benzyloxymethyl) phenylacetyl)-4-(phenylmethyl)-2-oxazolidinone (D). Compound **C** (1.0 g, 3.38 mmol) was dissolved in 17 mL of CH₂Cl₂ at 0 °C. TiCl₄ (390 µL, 3.56 mmol) was added and the dark red solution stirred for 5 min, followed by the addition of *N,N*-diisopropylethylamine (650 µL, 3.73 mmol). The reaction mixture was stirred for a further 1 h at which time benzylchloromethyl ether (940 µL, 6.78 mmol) was added to the reaction mixture. The reaction was stirred at 0 °C for a further 12 h, then diluted with 60 mL of 1:1 CH₂Cl₂:aq NaHSO₄. The biphasic mixture was stirred vigorously for 10 min. The layers were separated and the organic layer washed with an aqueous saturated solution of NaHCO₃, water, brine, dried (Na₂SO₄) and filtered. Concentration followed by chromatography using 25% EtOAc/hexanes as eluent furnished 1.4 g (98%) of **D**. *R*_f = 0.5 (25% EtOAc/hexane). [*α*]_D = + 85.5 (*c* 2.0, CHCl₃); IR (CH₂Cl₂) 1770, 1680 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.42-7.08 (m, 15H), 5.49 (dd, *J* = 4.7, 10.1 Hz, 1H), 4.67-4.55 (m, 3H), 4.27 (t, *J* = 9.5 Hz, 1H), 4.08-3.99 (m, 2H), 3.65 (dd, *J* = 4.8, 9.0 Hz, 1H), 3.26 -3.21 (m, 1H), 2.89-2.84 (m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 172.4, 152.8, 138.0, 135.0, 134.9, 129.5, 128.8, 128.3, 127.8, 127.6, 127.5, 127.2, 72.4, 65.6, 55.3, 49.1, 37.6; MS (+FAB) 416 (M⁺+1, 100); HRFABMS calcd for C₂₆H₂₆NO₄ (M⁺+1): 416.1862; found: 416.1873.

E

(S)-1-Benzyloxy-2-phenyl-1,3-propanediol (E). LAH (0.30 g, 7.95 mmol) was added to a solution of compound **D** (1.1 g, 2.65 mmol) in 30 mL THF at 0 °C and the reaction mixture was stirred at 0 °C for 35 min.⁶ The reaction was quenched by addition of EtOAc (20 mL) followed by the addition of water (2 mL). The precipitate was passed through Celite and the solution dried (Na₂SO₄) and filtered. Concentration followed by chromatography using 25→50% EtOAc/hexanes as eluent provided 0.53 g (83%) of **E** as an oil. R_f = 0.5 (50% EtOAc/hexane). $[\alpha]_D$ = - 15.07 (c 1.4, CHCl₃); IR (CH₂Cl₂) 3320, 1060 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.25 (m, 10H), 4.59 (s, 2H), 4.04-3.90 (m, 1H), 3.88-3.79 (m, 3H), 3.27-3.20 (m, 1H), 2.52 (br s, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 139.5, 137.8, 128.6, 128.4, 127.9, 127.7, 127.6, 127.0, 73.6, 73.4, 66.4, 47.8; MS (+FAB) 243 (M⁺+1, 70); HRFABMS calcd for C₁₆H₁₉O₂ (M⁺+1): 243.1385; found: 243.1358.

9

(S)-3-Azido-2-phenyl-1-propanol (9): A solution of compound **E** (0.2 g, 0.83 mmol), CBr₄ (0.38 g, 1.16 mmol) and PPh₃ (0.26 g, 0.99 mmol) in 40 mL CH₂Cl₂ was stirred at room temperature for 24 h.⁷ The solvent was removed in vacuo and the residue was filtered through a plug of silica gel in 10% EtOAc/hexanes. The filtrate was concentrated in vacuo and the residue (0.16 g, R_f = 0.8 (25% EtOAc/hexane)) dissolved in 15 mL THF; 25 mg of 10% Pd/C was then added to the reaction mixture and the

reaction flask purged three times with H₂. The reaction was stirred under hydrogen atmosphere for 5 h at which time the reaction mixture was purged with Ar, filtered through Celite, and the filtrate concentrated to an oil. Flash column chromatography using 25% EtOAc/hexanes as eluent provided 65 mg of (*R*)-3-bromo-2-phenyl-1-propanol as a clear oil (*R_f* = 0.2, 25% EtOAc/hexane). [α]_D = -21.7 (*c* 1.8, CHCl₃); IR (CH₂Cl₂) 3280, 680 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.38-7.23 (m, 5H), 3.95 (d, *J* = 5.9 Hz, 2H), 3.77-3.62 (m, 2H), 3.24-3.17 (m, 1H), 1.60 (br s, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 139.7, 128.8, 128.6, 127.9, 127.6, 121.5, 64.9, 49.9, 34.1). A solution of (*R*)-3-bromo-2-phenyl-1-propanol (50 mg, 0.23 mmol) and NaN₃ (45 mg, 0.70 mmol) in 2 mL DMF was stirred at room temperature for 18 h. The reaction mixture was diluted with 8 mL Et₂O and poured into water. The organic layer was washed with water (3 x 5 mL), brine, dried (Na₂SO₄) and filtered. Concentration followed by chromatography using 10→50% Et₂O/PE as eluent gave 32 mg (45% over three steps) of **9** as an oil. *R_f* = 0.3 (25% EtOAc/hexane). [α]_D -20 (*c* 1.3, CHCl₃). IR (CH₂Cl₂) 3280, 2060 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.43-7.21 (m, 5H), 3.82 (d, *J* = 6.2 Hz, 2H), 3.62 (m, 2H), 3.04 (m, 1H), 1.91 (br s, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 139.0, 128.9, 128.8, 127.9, 127.8, 127.5, 64.5, 53.1, 47.6; MS (CI) 178 (*M*⁺+1, 25); HRFABMS calcd for C₉H₁₂N₃O (*M*⁺+1): 178.0980; found: 178.0984.

10

(±)-3-Azido-2-isopropyl-1-propanol (10). LAH (0.5 g, 13.5 mmol) was suspended in 100 mL Et₂O and the suspension was cooled to 0 °C. Diethyl

isopropylmalonate (0.92 mL, 4.49 mmol) was added dropwise over 5 min. The resultant mixture was warmed to room temperature and stirred for 18 h. The reaction flask was placed in an ice bath and the reaction was quenched by addition of EtOAc (15 mL) followed by water (2 mL). The suspension was filtered through Celite, the filtrate dried (Na_2SO_4) and concentrated to provide 0.53 g of crude 2-isopropyl-1,3-propanediol. The crude 2-isopropyl-1,3-propanediol was then reacted according to procedure B to afford 0.59 g (92%) of **10** as a clear oil. $R_f = 0.3$ (25% EtOAc/hexane). IR (CH_2Cl_2) 3300, 2080 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.73 (dd, $J = 5.0, 12.3$ Hz, 1H), 3.63 (dd, $J = 7.0$ Hz, 10.8 Hz, 1H), 3.51 (dd, $J = 5.0, 12.3$ Hz, 1H), 3.42 (dd, $J = 7.0, 12.2$ Hz, 1H), 1.82-1.75 (m, 1H), 1.55-1.49 (m, 1H), 0.95 (s, 3H), 0.94 (s, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 61.8, 51.2, 46.4, 26.7, 19.9, 19.8; MS (+FAB) 143 (M^+ , 75); HRFABMS calcd for $\text{C}_6\text{H}_{13}\text{N}_3\text{O}$ (M^+): 143.1059; found: 143.1071.

11

(2R, 4S)-4-Azido-2-hydroxypentane (11). Reaction of (*R, R*)-2,4-pentanediol (Aldrich, 1.00 g, 9.61 mmol) according to procedure B gave 0.98 g (79%) of **11** as a clear oil: $[\alpha]_D +59.9$ (c 1.0, CHCl_3); R_f 0.82 (50% EtOAc/Hexane); ^1H NMR (300 MHz, CDCl_3) δ 1.22 (d, $J = 6$ Hz, 3H), 1.32 (d, $J = 6.6$ Hz, 3H), 1.49-1.57 (m, 1H), 1.61-1.71 (m, 1H), 2.17 (br s, 1H), 3.60-3.72 (m, 1H), 3.89-3.99 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 19.4, 23.6, 44.8, 56.2, 66.3; IR (neat) 3360 (br), 2100 (s) cm^{-1} ; MS (EI) m/e 129 (M^+), 105, 83, 69.

(2R, 4R)-2-tert-Butyldimethylsiloxy-4-hydroxypentane (F): A mixture of sodium hydride (58 mg, 2.4 mmol) and (2R, 4R)-2,4-pentanediol (Aldrich, 250 mg, 2.4 mmol) in 50 mL THF was stirred at 0 °C for 10 min, followed by the addition of TBSCl (0.36 g, 2.4 mmol).⁸ The resulting reaction mixture was stirred at room temperature for 18 h. The reaction was diluted with Et₂O (25 mL) and water (25 mL) and the layers separated. The organic layer was washed with brine, dried (Na₂SO₄) and filtered. The filtrate was concentrated in vacuo to an oil. Flash column chromatography using 15% EtOAc/hexanes as eluent afforded 0.38 g (72%) of **F** as an oil. $R_f = 0.9$ (25% EtOAc/hexane). $[\alpha]_D = -8.4$ (c 1.0, CHCl₃); IR (CH₂Cl₂) 3240 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 4.21-4.13 (m, 2H), 3.41 (br s, 1H), 1.68-1.46 (m, 2H), 1.22 (d, $J = 6.3$ Hz, 3H), 1.16 (d, $J = 6.2$ Hz, 3H), 0.89 (s, 9H), 0.10 (s, 3H), 0.09 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 67.5, 64.2, 45.9, 25.7, 23.7, 22.7, 17.8, -4.6, -5.1; MS (CI) 219 ($M^+ + 1$, 100); HRFABMS calcd for C₁₁H₂₇O₂Si ($M^+ + 1$): 219.1780; found: 219.1760.

(2R, 4S)-2-tert-Butyldimethylsiloxy-4-hydroxypentane (G): To a cooled (0 °C) solution of **F** (250 mg, 1.14 mmol), benzoic acid (0.14 g, 1.14 mmol) and PPh₃ (0.33 g, 1.26 mmol) in 25 mL THF was added DEAD (198 μ L, 1.26 mmol) and the resultant reaction mixture was stirred at room temperature for 20 h. The reaction mixture was partitioned between Et₂O and water and the organic layer was washed with brine, dried

(Na₂SO₄) and filtered. The filtrate was concentrated in vacuo to an oil which was dissolved in 20 mL of 8:1 MeOH/water mixture, followed by the addition of K₂CO₃ (157 mg, 1.14 mmol). The reaction mixture was stirred at room temperature for 12 h, partitioned between Et₂O and water, and the organic layer was washed with brine, dried (Na₂SO₄) and filtered. Concentration followed by chromatography using 10→25% EtOAc/hexanes as eluent provided 120 mg (50% over two steps) of **G** as an oil. *R_f* = 0.2 (5% EtOAc/hexane). [α]_D = -0.5 (*c* 1.0, CHCl₃); IR (CH₂Cl₂) 3330 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 4.09-4.05 (m, 1H), 3.97-3.92 (m, 1H), 1.62-1.50 (m, 2H), 1.17 (d, *J* = 6.1 Hz, 3H), 1.15 (d, *J* = 6.3 Hz, 3H), 0.90 (s, 9H), 0.12 (s, 3H), 0.11 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 70.1, 67.5, 47.5, 25.8, 24.6, 23.4, 17.8, -3.9, -4.9; MS (CI) 219 (*M*⁺+1, 100); HRFABMS calcd for C₁₁H₂₇O₂Si (*M*⁺+1): 219.1780; found: 219.1797.

(2*R*, 4*R*)-4-Azido-2-hydroxypentane (12): Reaction of **G** (100 mg, 0.46 mmol) using procedure B gave an oil that was dissolved in 10 mL THF and treated with TBAF (460 μL of 1M solution in THF, 0.46 mmol). After stirring at room temperature for 30 min, the reaction was diluted with Et₂O (10mL) and water (10 mL). The layers were separated and the organic layer was washed with brine, dried (Na₂SO₄) and filtered. Concentration followed by chromatography using 25% Et₂O/PE as eluent furnished 40 mg (67% over two steps) of **12** as an oil. *R_f* = 0.5 (10% EtOAc/hexane). [α]_D = -50.1 (*c* 0.8, CHCl₃); IR (CH₂Cl₂) 3360, 2000 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 4.04-4.00 (m, 1H), 3.98-3.73 (m, 1H), 1.90 (br s, 1H), 1.58-1.52 (m, 2H), 1.30 (d, *J* = 6.6 Hz, 3H),

1.22 (d, $J = 6.2$ Hz, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 64.7, 54.9, 45.1, 24.0, 19.8; MS (CI) 130 ($\text{M}^+ + 1$, 20); HRFABMS calcd for $\text{C}_5\text{H}_{10}\text{N}_3\text{OLi}$ ((M-1)Li): 136.1062; found: 136.1048.

Ring-Expansion Reactions

Iminium ether tetrafluoroborate salt (13). A flask containing 4-methylcyclohexanone (**29**) (348 mg, 3.10 mmol), (*R*)-2-azido-2-phenylethanol (757 mg, 4.65 mmol) and *n*-pentane (10 mL) was cooled to -20 °C under argon and $\text{BF}_3 \cdot \text{OEt}_2$ (1.15 mL, 9.3 mmol) was introduced dropwise. The reaction was stirred at -20 °C for 18 h and then was allowed to warm to room temperature. The reaction was concentrated and chromatographed (30:1 $\rightarrow$ 10:1, $\text{CH}_2\text{Cl}_2/\text{MeOH}$); recrystallization (twice) from 25% EtOAc/hexane gave 0.34 g (33%) of **13** as a white crystalline solid. TLC (30:1 $\text{CH}_2\text{Cl}_2/\text{MeOH}$). $R_f = 0.25$. The ^1H NMR spectrum was sensitive to concentration; the reported data was obtained at $c = 5$ mg/mL. ^1H NMR (400 MHz, CDCl_3) δ 1.09 (d, $J = 6.6$ Hz, 3H), 1.65-1.85 (m, 3H), 1.90-2.00 (m, 2H), 2.90-3.00 (m, 1H), 3.10-3.20 (m, 1H), 3.30-3.40 (m, 1H), 3.60-3.70 (m, 1H), 4.78 (t, $J = 8.6$ Hz, 1H), 5.56 (dd, $J = 10.6, 9.5$ Hz, 1H), 5.74 (dd, $J = 10.6, 8.4$ Hz, 1H), 7.40-7.45 (m, 2H), 7.50-7.55 (m, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 21.5, 25.9, 28.0, 32.0, 34.0, 45.2, 68.4, 78.8, 128.0, 130.1, 130.7, 133.2, 180.2. IR (KBr) 3400, 2950, 1645, 1475 cm^{-1} . MS (EI) m/e 230 (M^+), 214, 104, 91, 77; HRMS m/e calcd for $\text{C}_{15}\text{H}_{20}\text{NO}$: (M^+) 230.1545; found: (M^+) 230.1526. Anal. Calcd for $\text{C}_{15}\text{H}_{20}\text{NOBF}_4$: C, 56.81; H, 6.36; N, 4.42; found: C, 56.90; H, 6.76; N, 4.11. An X-ray

structure of the salt was obtained and the data is presented elsewhere in this Supporting Information.

General procedure for Ring Expansion. (1'*R*, 5*R*)-5-Methyl-1-(1'-phenylhydroxyethyl)-hexahydro-2*H*-azepine-2-one (**14a**) and (1'*R*, 5*S*)-5-methyl-1-(1'-phenylhydroxyethyl)-hexahydro-2*H*-azepine-2-one (**14b**). A reaction vessel was charged with 4-methylcyclohexanone (137 mg, 1.23 mmol), (*R*)-2-azido-2-phenylethanol (301 mg, 1.85 mmol), and CCl₄ (5 mL). The reaction vessel was cooled to -20 °C under argon and BF₃•OEt₂ (0.45 mL, 3.69 mmol) was introduced dropwise. The reaction was stirred at -20 °C for 18 h and then was allowed to warm to room temperature. The solution was concentrated and 15% KOH (3 mL) was added. The reaction was stirred for 15 min and then concentrated, diluted with CH₂Cl₂ (75 mL), dried (MgSO₄), filtered, and concentrated to give a crude yellow oil. HPLC analysis of the crude reaction mixture indicated a 83:17 (**14a** : **14b**) ratio of diastereomers. Flash chromatography (1:1 hexane/EtOAc) afforded compounds (**14a** as a white solid and **14b** as an oil) in a combined yield of 295 mg (97%). TLC (15:1 CH₂Cl₂/MeOH): R_f (**14a**) = 0.63, R_f (**14b**) = 0.72. Major diastereomer (**14a**): mp 91 °C. [α]_D = -74.6 (*c*, 1.44 CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 0.65 (m, 1H), 0.83 (d, *J* = 6.6 Hz, 3H), 1.18-1.30 (m, 1H), 1.41-1.50 (m, 1H), 1.51-1.65 (m, 1H), 1.75-1.85 (m, 1H), 2.55-2.65 (m, 2H), 3.05-3.30 (m, 3H), 4.00-4.15 (m, 2H), 5.78 (dd, *J* = 5.2, 8.9 Hz, 1H), 7.25-7.38 (m, 5H); ¹³C NMR (100.6 MHz, CDCl₃) δ 22.4, 31.3, 36.0, 36.3, 36.5, 43.9, 59.2, 62.1, 127.8, 128.3, 128.6, 137.1, 177.6. IR (neat) 3360, 2900, 1600 cm⁻¹. MS (EI) *m/e* 248 (M⁺+1), 229, 216, 91; HRMS calcd for

$C_{15}H_{22}NO_2$: ($M^+ + 1$) 248.1650; found: 248.1667. Anal. Calcd for $C_{15}H_{21}NO_2$: C, 72.84; H, 8.56; N, 5.66; found: C, 72.64; H, 8.82; N, 5.57. Minor diastereomer (**14b**): $[\alpha]_D = -68.5$ (c 0.84, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 0.94 (d, $J = 6.5$ Hz, 3H), 1.20-1.38 (m, 2H), 1.55-1.65 (m, 1H), 1.65-1.75 (m, 1H), 1.80-1.90 (m, 1H), 2.55-2.70 (m, 2H), 2.82 (br s, 1H), 3.05-3.15 (m, 2H), 3.95-4.05 (m, 1H), 4.17-4.23 (m, 1H), 5.91 (dd, $J = 5.1, 9.3$ Hz, 1H), 7.20-7.38 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 22.5, 31.3, 36.3 (2C), 36.8, 44.1, 58.5, 62.2, 127.6, 127.7, 128.6, 137.4, 177.5. IR (neat) 3380, 2910, 1610 cm^{-1} . MS (EI) m/e 248 ($M^+ + 1$), 229, 216, 91; HRMS calcd for $C_{15}H_{22}NO_2$ ($M^+ + 1$): 248.1650; found ($M^+ + 1$): 248.1653. HPLC: t_R major (**14a**) = 10.0 min, t_R minor (**14b**) = 9.1 min; Econosil-CN; 80% hexane/*i*-PrOH; flow rate 1.5 mL/min; UV 254 nm.

(1'R,5R)-5-tert-butyl-1-(1'-phenylhydroxyethyl)-hexahydro-2H-azepine-2-one (15a) and (1'R,5S)-5-tert-Butyl-1-(1'-phenylhydroxyethyl)-hexahydro-2H-azepine-2-one (15b). Major diastereomer (**15a**): mp 139-140 °C; $[\alpha]_D = -63.1$ (c , 1.52, $CHCl_3$). 1H NMR (400 MHz, $CDCl_3$) δ 0.61 (m, 1H), 0.71 (s, 9H), 1.13-1.30 (m, 2H), 1.52-1.60 (m, 1H), 1.94-2.01 (m, 1H), 2.53-2.78 (m, 2H), 2.78 (br s, 1H), 3.15-3.29 (m, 2H), 4.04-4.15 (m, 2H), 5.71 (dd, $J = 5.3, 8.7$, 1H), 7.25-7.38 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 24.2, 27.4, 29.0, 32.9, 36.9, 44.7, 51.3, 59.7, 62.5, 128.0, 128.3, 128.6, 137.1, 177.9. IR (KBr) 3240, 2940, 1580. MS (EI) m/e 290 ($M^+ + 1$), 271, 258; HRMS calcd for $C_{18}H_{28}NO_2$: ($M^+ + 1$) 290.2120; found: 290.2137. Minor diastereomer (**15b**): $[\alpha]_D = -53.9$ (c 1.16, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 0.84 (s, 9H), 1.15-1.40 (m, 3H), 1.80-1.90 (m, 1H), 1.90-2.05 (m, 1H), 2.50-2.60 (m, 1H), 2.65-2.75 (m, 1H), 2.84 (br s, 1H), 3.08 (m, 1H), 3.16 (m, 1H), 4.00-4.05 (m, 1H), 4.17-4.21 (m, 1H), 5.86 (dd, $J = 5.2, 9.1$

Hz, 1H), 7.21-7.40 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 24.0, 27.5, 29.5, 33.0, 36.5, 44.6, 51.4, 58.6, 62.2, 127.7, 128.6, 137.5, 177.5. IR (neat) 3360, 2930, 1605, 1435, 690. MS (EI) m/e 290 ($\text{M}^+ + 1$), 271, 258; HRMS calcd for $\text{C}_{18}\text{H}_{28}\text{NO}_2$: ($\text{M}^+ + 1$) 290.2120; found: 290.2123. HPLC: t_R major (**15a**) = 10.0 min, t_R minor (**15b**) = 8.8 min Econosil-CN; 83% hexane/*i*-PrOH; flow rate 1.5 mL/min; UV 254 nm.

***N*-Dealkylation of **15a**: (*R*)-Hexahydro-5-*tert*-butyl-2*H*-azepine-3-one (**H**).**

About 2 mL ammonia was condensed into a THF (2 mL) solution containing **15a** (100 mg, 0.27 mmol). Small pieces of Na metal were added until the solution turned blue. The reaction was stirred for 15 min and was allowed to warm to room temperature overnight. The reaction was quenched with saturated NH_4Cl (1 mL). The solution was diluted with EtOAc (25 mL), dried (MgSO_4), filtered, and concentrated. Flash chromatography (EtOAc $\rightarrow$ 90:10 EtOAc/MeOH) gave 40 mg (86%) of **I** as a white solid. The ^1H and ^{13}C spectra matched the literature data.⁹ $[\alpha]_D = +12.3$ (c, 0.52, MeOH). lit.⁹ $[\alpha]_D = +14.7$ (c, 0.5, MeOH).

(1'*R*,2'*S*,5*R*)-5-Methyl-1-(1',2'-diphenylhydroxyethyl)-hexahydro-2*H*-azepine-2-one (16a**) and (1'*R*,2'*S*,5*S*)-5'-methyl-1-(1',2'-diphenylhydroxyethyl)-hexahydro-2*H*-azepine-2-one (**16b**).** TLC (1:1 hexane/EtOAc): R_f (**16a**) = 0.40, R_f (**16b**) = 0.50. Major diastereomer (**16a**): mp 159 °C. $[\alpha]_D = -27.4$ (c 0.562, CHCl_3). ^1H

NMR (400 MHz, CDCl_3) δ 0.35 (q, J = 2.1 Hz, 1H), 0.73 (d, J = 6.4 Hz, 3H), 0.97 (q, J = 11.7 Hz, 1H), 1.38-1.43 (m, 2H), 1.58-1.65 (m, 1H), 2.20 (apparent t, J = 12.6 Hz, 1H), 2.32 (dd, J = 7.7, 13.1 Hz, 1H), 3.06 (d, J = 2.7 Hz, 1H), 3.20-3.33 (m, 2H), 5.41-5.49 (m, 2H), 7.25-7.38 (m, 6H), 7.40-7.50 (m, 4H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 22.5, 30.9, 36.00, 36.04, 36.4, 47.1, 66.4, 73.7, 127.1, 127.8, 128.0, 128.2, 128.4, 129.1, 137.5, 141.4, 175.6. IR (CH_2Cl_2) 3360, 2920, 1615, 1445 cm^{-1} . MS (EI) m/e 324 ($\text{M}^+ + 1$), 306, 278, 216 (100), 91, 77; HRMS m/e calcd for $\text{C}_{21}\text{H}_{26}\text{NO}_2$: ($\text{M}^+ + 1$) 324.1964; found: 324.1958. Anal. Calcd for $\text{C}_{21}\text{H}_{25}\text{NO}_2$: C, 77.99; H, 7.79; N, 4.33; found: C, 78.18; H, 8.08; N, 4.31. Minor diastereomer (**16b**): $[\alpha]_{\text{D}} = -32.5$ (c 0.08, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 0.73 (d, J = 6.4 Hz, 3H), 0.75-0.91 (m, 2H), 1.20-1.30 (m, 1H), 1.39-1.50 (m, 2H), 2.34 (apparent t, J = 12.0 Hz, 1H), 2.47 (dd, J = 8.2, 14.2 Hz, 1H), 3.02 (d, J = 3.4 Hz, 1H), 3.10 (dd, J = 10.3, 15.4 Hz, 1H), 3.23 (dd, J = 6.7, 15.4 Hz, 1H), 5.37 (dd, J = 3.0, 7.5 Hz, 1H), 5.71 (d, J = 7.5 Hz, 1H), 7.23-7.39 (m, 6H), 7.41-7.50 (m, 4H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 22.1, 30.5, 35.2, 36.0, 36.2, 46.5, 65.2, 74.3, 127.5, 127.7, 128.2, 128.3, 128.4, 129.1, 137.5, 141.1, 176.0. IR (CH_2Cl_2) 3360, 2920, 1615 cm^{-1} . MS (EI) m/e 324 ($\text{M}^+ + 1$), 306, 278, 269, 251, 216 (100), 91, 77; HRMS m/e calcd for $\text{C}_{21}\text{H}_{26}\text{NO}_2$: ($\text{M}^+ + 1$) 324.1964; found: 324.1971. HPLC: t_{R} major (**16a**) = 13.5 min, t_{R} minor (**16b**) = 12.1 min; Chirobotic T; 90% hexane/EtOH; flow rate 1 mL/min; UV 254 nm.

(1'R,2'S,5R)-5-tert-Butyl-1-(1',2'-diphenylhydroxyethyl)-hexahydro-2H-azepine-2-one (17a) and (1'R,2'S,5S)-5-tert-butyl-1-(1',2'-diphenylhydroxyethyl)-hexahydro-2H-azepine-2-one (17b). TLC (1:1 hexane/EtOAc): R_{f} (**17a**) = 0.42, R_{f}

(**17b**) = 0.52. Major diastereomer (**17a**): mp 147-148 °C. $[\alpha]_D = -17.7$ (c 0.9, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 0.37 (q, J = 11.6 Hz, 1H), 0.68 (s, 9H), 0.91-1.01 (m, 2H), 1.50-1.56 (m, 1H), 1.74-1.81 (m, 1H), 2.14 (dd, J = 11.5, 14.0 Hz, 1H), 2.36 (dd, J = 7.7, 14.0 Hz, 1H), 3.02 (s, 1H), 3.12 (dd, J = 10.5, 15.2 Hz, 1H), 3.26 (dd, J = 5.4, 15.2 Hz, 1H), 5.41 (br d, J = 8.1 Hz, 1H), 5.48 (br d, J = 7.9 Hz, 1H), 7.24-7.37 (m, 6H), 7.45 (d, J = 7.0 Hz, 2H), 7.49 (d, J = 6.9 Hz, 2H); ¹³C NMR (100.6 MHz, CDCl₃) δ 23.7, 27.3, 28.7, 32.8, 36.6, 47.7, 51.2, 66.7, 73.6, 127.0, 127.8, 128.1, 128.2, 128.4, 128.9, 129.0, 137.7, 141.4, 175.8. IR (CH₂Cl₂) 3340, 2940, 1615, 1445. MS (EI) m/e 366 (M^+ +1), 348, 271, 258 (70), 91.77. Minor diastereomer (**17b**): $[\alpha]_D = +30.0$ (c 0.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 0.66 (s, 9H), 0.69-1.73 (m, 5H), 2.27-2.33 (m, 1H), 2.48-2.53 (m, 1H), 2.83 (br s, 1H), 3.02-3.08 (m, 1H), 3.22-3.20 (m, 1H), 5.35 (br d, J = 8.0 Hz, 1H), 5.84 (br d, J = 8.1 Hz, 1H), 7.26-7.37 (m, 6H), 7.45-7.50 (m, 4H); ¹³C NMR (100.6 MHz, CDCl₃) δ 22.4, 23.2, 28.8, 32.7, 36.5, 46.4, 50.6, 64.0, 74.2, 127.7, 127.8, 128.3, 128.4 (2C), 129.0, 137.7, 141.0, 175.9. IR (neat) 3320, 2940, 1610 cm⁻¹. HPLC: t_R major (**17a**) = 36.3 min, t_R minor (**17b**) = 34.0 min; Chirobotic T; 90% hexane/EtOH; flow rate 1 mL/min; UV 254 nm.

(±)-(1'*R*,5*R*)-5-*tert*-Butyl-1-(3'-phenylhydroxyethyl)-hexahydro-2H-azepine-2-one and (±)-(1'*R*,5*S*)-5-*tert*-butyl-1-(3'-phenylhydroxyethyl)-hexahydro-2H-azepine-2-one (**18a** and **18b**). Flash chromatography (20:1, CH₂Cl₂/MeOH) gave an inseparable mixture of diastereomers as a white solid (0.256 g, 73% yield); the stereostructures were not assigned. TLC (20:1 CH₂Cl₂/MeOH): R_f = 0.42. Major isomer (diagnostic peaks only): ¹H NMR (400 MHz, CDCl₃) δ 0.84 (s, 9H), 2.57-2.64 (m, 1H),

3.56-3.60 (m, 1H), 3.72-3.84 (m, 1H), 4.57 (d, $J = 4.3$, 1H), 4.97-5.01 (m, 1H), 7.25-7.40 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 23.9, 27.5, 29.2, 33.1, 36.1, 51.5, 51.8, 58.4, 74.3, 125.8, 127.5, 128.4, 142.4, 178.6. Minor isomer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 0.81 (s, 9H), 2.43-2.50 (m, 1H), 2.57-2.64 (m, 1H), 3.10 (dd, $J = 5.2, 15.3$ Hz, 1H), 3.46 (dd, $J = 10.8, 15.0$ Hz, 1H), 3.52-3.61 (m, 1H), 3.72-3.84 (m, 1H), 4.86 (d, $J = 4.7$ Hz, 1H), 4.96-5.00 (m, 1H), 7.25-7.40 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 23.9, 36.2, 59.1, 74.1. For mixture: IR (CH_2Cl_2) 3340, 2940, 1615 cm^{-1} . MS (EI) m/e 290 ($\text{M}^+ + 1$), 272, 182 (100), 154, 77. Anal. Calcd for $\text{C}_{18}\text{H}_{27}\text{NO}_2$: C, 74.96; H, 9.09; N, 4.86; found: C, 74.61; H, 9.17; N, 4.63. HPLC: t_R (major) = 16.01 min, t_R (minor) = 17.16 min; Econosil-CN; 90% hexane/*i*-PrOH; flow rate 1 mL/min; UV detector 254 nm UV.

(3'*R*,5*R*)-5-Methyl-1-(3'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (19a) and (3'*R*,5*S*)-5-Methyl-1-(3'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (19b). TLC (1:1 hexane/EtOAc): R_f (**19a**) = 0.15, R_f (**19b**) = 0.20. Major diastereomer (**19a**): $[\alpha]_D = -4.2$ (c 1.02, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 0.99 (d, $J = 6.6$, 3H), 1.12-1.31 (m, 3H), 1.62-1.75 (m, 1H), 1.76-1.98 (m, 4H), 2.42-2.61 (m, 2H), 3.12 (dt, $J = 14.2, 4.4$ Hz, 1H), 3.24 (dd, $J = 6.5, 15.2$ Hz, 1H), 3.51 (dd, $J = 10.9, 15.2$ Hz, 1H), 4.09-4.19 (m, 1H), 4.62 (m, 1H), 7.21-7.28 (m, 1H), 7.31-7.39 (m, 4H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 22.6, 31.4, 35.7, 36.1, 37.2, 37.7, 45.7, 49.3, 69.8, 125.5, 127.0, 128.3, 144.1, 177.1. MS (EI) m/e 262 ($\text{M}^+ + 1$), 244(100); HRMS m/e calcd for $\text{C}_{16}\text{H}_{24}\text{NO}_2$: ($\text{M}^+ + 1$) 262.1807, found: 262.1798. Minor diastereomer (**19b**): $[\alpha]_D = -2.2$ (c 0.5, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 0.97 (d, $J = 6.5$ Hz, 3H), 1.18-1.30 (m, 2H),

1.65-1.95 (m, 5H), 2.50-2.56 (m, 2H), 3.01 (m, 1H), 3.32 (dd, $J = 15.1, 5.9$ Hz, 1H), 3.47 ($J = 15.1, 11.0$ Hz, 1H), 4.10-4.25 (m, 1H), 4.53 (br d, $J = 10.4$ Hz, 1H), 4.75-4.85 (m, 1H), 7.21-7.29 (m, 1H), 7.29-7.39 (m, 4H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 22.6, 31.3, 35.7, 35.8, 36.3, 37.5, 44.9, 48.4, 69.6, 125.7, 127.0, 128.2, 144.1, 177.2. MS (EI) m/e 262 ($\text{M}^+ + 1$), 244; HRMS m/e calcd for $\text{C}_{16}\text{H}_{24}\text{NO}_2$: ($\text{M}^+ + 1$) 262.1807, found: 262.1797. HPLC: t_R major (**19a**) = 19.9 min, t_R minor (**19b**) = 18.2 min; Chirobotic T; 90% hexane/EtOH; flow rate 1 mL/min; UV 254 nm.

Removal of side chain from 19a: oxidation of side chain alcohol. (*R*)-Hexahydro-1-(3'-phenyl-3'-propanone)-5-methyl-2*H*-azapine-2-one (I). Compound **19a** (268 mg, 1.0 mmol) and PCC (431 mg, 2.0 mmol) were reacted in a slurry of CH_2Cl_2 (10 mL) and silica gel (1 g) for *ca.* 3 h. The reaction was filtered and concentrated to afford a crude oil. Flash chromatography (75% hexane/EtOAc) gave 239 mg (92%) of the title compound as a white solid, mp 83-84 °C. TLC (30:1 $\text{CH}_2\text{Cl}_2/\text{MeOH}$): $R_f = 0.75$. $[\alpha]_D = +18.6$ (c, 1.08, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 0.93 (d, $J = 6.6$ Hz, 3H), 1.08-1.28 (m, 2H), 1.55-1.68 (m, 1H), 1.74-1.85 (m, 2H), 2.45-2.55 (m, 2H), 3.21-3.35 (m, 2H), 3.36-3.44 (m, 1H), 3.45-3.55 (m, 1H), 3.70 (apparent pentet, $J = 6.8$ Hz, 1H), 3.83 (apparent pentet, $J = 6.8$ Hz, 1H), 7.46 (t, $J = 7.7$ Hz), 7.56 (t, $J = 7.5$ Hz), 7.95-8.0 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 22.6, 31.3, 36.1, 36.9, 37.7 (2C), 45.3, 50.2, 128.2, 128.6, 133.3, 136.7, 175.8, 199.1. IR (KBr) 2940, 1660, 1630 cm^{-1} . MS (EI) m/e 259 (M^+), 231, 202, 154, 105, 77; HRMS m/e calcd for $\text{C}_{16}\text{H}_{22}\text{NO}_2$: ($\text{M}^+ + 1$) 260.1650; found: 260.1639. Anal. Calcd for $\text{C}_{16}\text{H}_{21}\text{NO}_2$: C, 74.10; H, 8.16; N, 5.40; found: C, 74.01; H, 8.07; N, 5.37.

Removal of side chain from 19a: elimination of side chain. (R)-Hexahydro-5-methyl-2H-azepine-3-one (J). Compound **I** (227 mg, 0.8 mmol) was dissolved in 20 mL of THF. NaH (128 mg, 3.2 mmol) was added and the reaction was heated at 65 °C for ca. 30 min. The reaction was cooled to room temperature and quenched with saturated NH₄Cl (2 mL). The reaction was diluted with CH₂Cl₂ (20 mL) and washed with saturated NH₄Cl (5 mL), H₂O (5 mL), and saturated NaCl (5 mL). The combined aqueous layer was dried (MgSO₄), filtered, and concentrated. Flash chromatography (50% hexane/EtOAc → EtOAc → 95% EtOAc/MeOH) gave 60 mg (69%) of the known⁹ title compound as transparent oil. $[\alpha]_D = +18$ (*c* 1.7, MeOH); lit.⁹ $[\alpha]_D = +18$ (*c* 1.4, MeOH).

(3'*R*,5*R*)-5-Phenyl-1-(3'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (20a) and (3'*R*,5*S*)-5-phenyl-1-(3'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (20b). TLC (15:1 CH₂Cl₂/MeOH): *R*_f (**20a**) = 0.55, *R*_f (**20b**) = 0.58. Major diastereomer (**20a**): $[\alpha]_D = -7.9$ (*c* 1.33, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 1.65-1.90 (m, 3H), 1.95-2.15 (m, 3H), 2.55-2.80 (m, 3H), 3.25 (dt, *J* = 4.8, 14.1 Hz, 1H), 3.33 (dd, *J* = 15.3, 5.8 Hz, 1H), 3.66 (dd, *J* = 15.3, 11.0, Hz, 1H), 4.05-4.15 (m, 1H), 4.56 (br s, 1H), 4.72 (dd, *J* = 3.1, 9.7 Hz, 1H), 7.15-7.18 (m, 2H), 7.20-7.41 (m, 8H); ¹³C NMR (100.6 MHz, CDCl₃) δ 30.6, 36.2, 36.8, 37.8, 45.7, 48.1, 49.5, 70.0, 125.5, 126.6, 126.7, 127.1, 128.4, 128.7, 144.0, 145.7, 176.8. IR (neat) 3350, 2905, 1605, 1480 cm⁻¹. MS (EI) *m/e*

323 (M^+), 306, 203, 118, 91, 77; HRMS Calcd for $C_{21}H_{26}NO_2$: (M^++1) 324.1963; found: 324.1952. Minor diastereomer (**20b**): $[\alpha]_D = +2.5$ (c 0.08, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 1.65-1.85 (m, 3H), 1.86-2.05 (m, 3H), 2.65-2.85 (m, 3H), 3.10 (dt, $J = 3.6, 14.1$ Hz, 1H), 3.32 (dd, $J = 15.1, 4.9$ Hz, 1H), 3.62 (dd, $J = 15.1, 11.1$ Hz, 1H), 4.15-4.25 (m, 1H), 4.57 (dd, $J = 10.2, 2.4$ Hz, 1h), 4.67 (br s, 1h), 7.15-7.40 (m, 10H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 30.7, 35.4, 36.3, 37.5, 45.1, 48.3, 48.7, 69.8, 125.7, 126.61, 126.65, 127.1, 128.3, 128.6, 144.0, 145.8, 176.8. IR (film) 3300, 2900, 1600, 1440 cm^{-1} . MS (EI) m/e 323 (M^+), 306, 214, 91; HRMS Calcd for $C_{21}H_{26}NO_2$: (M^++1) 324.1963; found: 324.1945.

(3'R,5R)-5-tert-Butyl-1-(3'-phenylhydroxypropyl)-hexahydro-2H-azepine-2-one (21a) and (3'R,5S)-5-tert-butyl-1-(3'-phenylhydroxypropyl)-hexahydro-2H-azepine-2-one (21b). TLC (1:1 hexane/EtOAc): R_f (**21a**) = 0.26, R_f (**21b**) = 0.30. Major diastereomer (**21a**): mp 79-80 °C. $[\alpha]_D = -1.68$ (c 1.0, $CHCl_3$). 1H NMR (400 MHz, $CDCl_3$) δ 0.90 (s, 9H), 1.15-1.35 (m, 3H), 1.75-1.88 (m, 1H), 1.89-1.99 (m, 1H), 2.00-2.08 (m, 2H), 2.42-2.47 (m, 1H), 2.60 (dd, J 7.5, 13.8 Hz, 1H), 3.12 (dt, $J = 14.2, 4.5$ Hz, 1H), 3.27 (dd, $J = 15.2, 6.2$ Hz, 1H), 3.51 (dd, $J = 15.2, 10.0$ Hz, 1H), 4.10-4.18 (m, 1H), 4.60 (dd, $J = 3.1, 10.0$ Hz, 1H), 7.20-7.38 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 24.2, 27.5, 30.0, 33.1, 36.0, 37.9, 45.5, 49.7, 51.4, 70.0, 125.5, 127.0, 128.3, 144.1, 177.3. MS (EI) m/e calcd for $C_{19}H_{30}NO_2$: (M^++1) 304.2276, found: 304.2301. Compound **21a** was analyzed by X-ray crystallography; the structure was reported in our initial communication.¹⁰ Minor diastereomer (**21b**): $[\alpha]_D = +3.0$ (c 0.1, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 0.89 (s, 9H), 1.18-1.32 (m, 3H), 1.71-1.82 (m, 1H), 1.85-2.08 (m, 3H),

2.50-2.68 (m, 2H), 3.00 (dt, $J = 14.1, 4.8$ Hz, 1H), 3.26 (dd, $J = 14.8, 6.2$ Hz, 1H), 3.44 (dd, $J = 14.8, 9.5$ Hz, 1H), 4.15-4.23 (m, 1H), 4.52 (dd, $J = 2.9, 10.5$ Hz, 1H), 4.74 (br s, 1H), 7.22-7.38 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 24.1, 27.6, 28.7, 33.2, 36.1, 37.5, 44.8, 48.9, 51.8, 69.6, 125.7, 127.1, 128.3, 144.1, 177.4. HRMS (EI) m/e calcd for $\text{C}_{19}\text{H}_{30}\text{NO}_2$: ($\text{M}^+ + 1$) 304.2276, found: 304.2276. HPLC: t_R major (**21a**) = 13.6 min, t_R minor (**21b**) = 12.1 min; Chirobotic T; 90% hexane/EtOH; flow rate 1 mL/min; UV 254 nm.

(R)-Hexahydro-1-(3'-phenyl-3'-propanone)-5-tert-butyl-2H-azapine-2-one

(K). Prepared from **21a**: TLC (30:1 $\text{CH}_2\text{Cl}_2/\text{MeOH}$): $R_f = 0.5$; mp 100-101 °C. $[\alpha]_D = +17.3$ (c 1.18, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 0.86 (s, 9H), 1.08-1.26 (m, 3H), 1.94 (m, 2H), 2.43 (m, 1H), 2.55 (m, 1H), 3.28 (td, $J = 6.7, 2.6$ Hz, 2H), 3.46 (m, 2H), 3.70 (m, 1H) 3.84 (m, 1H), 7.47 (t, $J = 7.6$ Hz, 2H), 7.57 (m, 1H), 7.98 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 24.1, 27.5, 29.7, 33.1, 36.5, 37.6, 45.2, 50.6, 51.5, 128.2, 128.6, 133.3, 136.8, 175.9, 199.2. IR (KBr) 2920, 1660, 1640, 1615. MS (EI) m/e 302 ($\text{M}^+ + 1$), 301, 244, 216, 196, 105, 77; HRMS (EI) m/e Calcd for $\text{C}_{19}\text{H}_{28}\text{NO}_2$: ($\text{M}^+ + 1$) 302.2120, found: 302.2093. Anal. Calcd for $\text{C}_{19}\text{H}_{27}\text{NO}_2$: C, 75.71; H, 9.03; N, 4.65; found: C, 75.56; H, 8.81; N, 4.55.

(R)-Hexahydro-5-tert-butyl-2H-azepine-3-one (H). This known compound⁹ was prepared from **K**: $[\alpha]_D = +15.2$ (c, 0.5, MeOH); lit.⁹ $[\alpha]_D = +14.7$ (c, 0.5, MeOH).

(1'R,5S)-5-Methyl-1-(1'-phenylhydroxypropyl)-hexahydro-2H-azepine-2-one (22a) and (2'R,5R)-5-methyl-1-(1'-phenylhydroxypropyl)-hexahydro-2H-azepine-2-one (22b). $R_f = 0.3$ (**22b**), 0.2 (**22a**) (50% EtOAc/hexane). Major diastereomer (**22a**): $[\alpha]_D = +153.7$ (c 1.4, CHCl_3); IR (CH_2Cl_2) 3340, 1650 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.37-7.27 (m, 5H), 5.97 (dd, $J = 3.1, 12.4$ Hz, 1H), 3.76-3.71 (m, 1H), 3.44 (dd, $J = 2.8, 11.7$ Hz, 2H), 3.10-2.99 (m, 2H), 2.72-2.66 (m, 1H), 2.61-2.54 (m, 1H), 2.16-2.08 (m, 1H), 1.97-1.89 (m, 1H), 1.83-1.78 (m, 1H), 1.59-1.51 (m, 1H), 1.34-1.21 (m, 2H), 0.80 (d, $J = 6.6$ Hz, 3H), 0.47-0.37 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 177.6, 138.8, 128.5, 128.4, 127.8, 58.3, 52.4, 42.7, 36.3, 36.2, 36.0, 31.9, 31.4, 22.4. Minor diastereomer (**22b**): $[\alpha]_D = +79.4$ (c 0.75, CHCl_3); IR (CH_2Cl_2) 3320, 1610 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.37-7.19 (m, 5H), 5.99 (dd, $J = 2.9, 12.5$ Hz, 1H), 3.82-3.77 (m, 1H), 3.61 (dd, $J = 2.6, 11.6$ Hz, 1H), 2.99-2.90 (m, 2H), 2.71-2.66 (m, 1H), 2.61-2.54 (m, 1H), 2.31-2.22 (m, 1H), 1.93-1.82 (m, 2H), 1.69-1.57 (m, 2H), 1.39-1.23 (m, 2H), 1.03-0.85 (m, 1H), 0.93 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 177.6, 139.4, 128.6, 127.9, 127.6, 58.5, 52.7, 43.3, 36.9, 36.1, 35.9, 32.1, 31.4, 22.4. MS (CI) 262 ($\text{M}^+ + 1$, 78); HRFABMS calcd for $\text{C}_{16}\text{H}_{24}\text{NO}_2$ ($\text{M}^+ + 1$): 262.1807; found: 262.1787. HPLC: t_R major: 22.4 min, t_R minor: 17.5 min; Chirobiotic T; 90% hexane/ethanol; flow rate 1 mL/min; UV 254 nm.

(1'*R*,5*S*)-5-*tert*-Butyl-1-(1'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (23a) and (2'*R*,5*R*)-5-*tert*-butyl-1-(1'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (23b). Major diastereomer (**23a**): $[\alpha]_D = +90.7$ (*c* 1.0, CHCl_3); IR (CH_2Cl_2) 3280, 1600 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.34-7.28 (m, 5H), 5.94 (dd, $J = 3.2, 12.4$ Hz, 1H), 3.93 (d, $J = 7.6$ Hz, 1H), 3.78 (bs, 1H), 3.59-3.37 (m, 1H), 3.09-2.92 (m, 2H), 2.71 (dd, $J = 7.4, 14.5$ Hz, 1H), 2.56-2.49 (m, 1H), 2.17-2.10 (m, 1H), 2.09-1.92 (m, 1H), 1.43-1.39 (m, 1H), 1.28-1.16 (m, 1H), 1.14-1.08 (m, 2H), 0.72 (s, 9H), 0.44-0.34 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 177.7, 138.8, 128.5, 128.3, 127.8, 58.3, 52.3, 51.3, 43.1, 36.4, 32.8, 31.7, 28.4, 27.2, 24.1. Minor diastereomer (**23b**): $[\alpha]_D = +84.9$ (*c* 0.65, CHCl_3); IR (CH_2Cl_2) 3380, 1610 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.54-7.02 (m, 5H), 5.99 (dd, $J = 2.8, 12.6$ Hz, 1H), 4.00-3.97 (m, 1H), 3.86-3.79 (m, 1H), 3.65-3.59 (m, 1H), 3.04-2.90 (m, 2H), 2.77-2.71 (m, 1H), 2.59-2.52 (m, 1H), 2.33-2.25 (m, 1H), 2.09-2.00 (m, 1H), 1.94-1.83 (m, 1H), 1.61 (bs, 1H), 1.36-1.15 (m, 2H), 1.05-0.94 (m, 1H), 0.86 (s, 9H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 177.6, 139.3, 128.6, 127.9, 127.6, 125.6, 58.4, 52.5, 43.7, 36.1, 33.0, 32.2, 29.7, 29.6, 27.5, 24.1. MS (CI) 304 ($\text{M}^+ + 1$, 100); HRFABMS calcd for $\text{C}_{19}\text{H}_{30}\text{NO}_2$ ($\text{M}^+ + 1$): 304.2277; found: 304.2269. HPLC: t_R major: 14.8 min, t_R minor: 11.4 min; Chirobiotic T; 90% hexane/ethanol; flow rate 1 mL/min; UV 254 nm.

Determination of stereochemistry of 23a: Preparation of (S)-H. Compound **23a** (25 mg) was dissolved in 5 mL of THF and the solution cooled to -78°C . 20 mL of NH_3 was condensed into the reaction flask. Pieces of sodium metal were added to the reaction mixture until a blue color persisted. The blue reaction mixture was stirred at -78°C for 15 min and the reaction quenched by the addition of solid NH_4Cl . The mixture was warmed to room temperature and partitioned between Et_2O and water. The organic layer was washed with brine, dried (Na_2SO_4), and filtered. The filtrate was concentrated and subjected to chromatography using EtOAc, then 5% MeOH in CH_2Cl_2 as eluent to furnish 11 mg of the dealkylated lactam **23a** as a white solid (78% yield). Determination of optical rotation of the dealkylated lactam **M** ($[\alpha]_{\text{D}} = -12.2$ (c 0.55, MeOH)) and comparison with reported value⁹ established that compound **23a** was the major diastereomer in the reaction of 4-*tert*-butylcyclohexanone with (*R*)-3-azido-2-phenyl-1-propanol.

5-*tert*-Butyl-(1'-(3,4,5-trimethoxyphenyl)hydroxypropyl)-hexahydro-2H-azepine-2-one (24a,b): Isolated as a mixture of diastereomers. IR (CH_2Cl_2) 3380, 1610

cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) diagnostic peaks: δ 6.54 (s, 1H), 5.87 (dd, $J = 2.9, 12.4$ Hz, 1H), 0.88 (s, 9H) (major diastereomer); δ 6.49 (s, 1H), 5.88 (dd, $J = 3.0, 12.4$ Hz, 1H), 0.75 (s, 9H) (minor diastereomer); ^{13}C NMR (100.6 MHz, CDCl_3) δ 177.8, 153.1, 137.5, 134.6, 105.5, 60.9, 58.3, 56.1, 52.4, 51.4, 43.2, 36.5, 32.9, 32.0, 28.8, 27.2, 24.2 (major diastereomer). MS (+FAB) 394 ($\text{M}^+ + 1$, 45); HRFABMS calcd for $\text{C}_{22}\text{H}_{36}\text{NO}_5$ ($\text{M}^+ + 1$): 394.2593; found: 394.2591.

(2'*R*,5*R*)-5-Methyl-1-(2'-methylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (25a) and (2'*R*,5*S*)-5-methyl-1-(2'-methylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (25b). $R_f = 0.2$ (75% EtOAc/hexane). Major diastereomer (**25a**): $[\alpha]_D = +5.6$ (c 1.6, CHCl_3); IR (CH_2Cl_2) 3290, 1610 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.98 (br s, 1H), 3.76 (dd, $J = 10.4, 13.8$ Hz, 1H), 3.48-3.44 (m, 2H), 3.43-3.33 (m, 1H), 3.31-3.19 (m, 1H), 2.83 (dd, $J = 4.4, 13.9$ Hz, 1H), 2.57-2.54 (m, 2H), 1.85-1.65 (m, 4H), 1.31-1.19 (m, 2H), 0.99 (d, $J = 4.8$ Hz, 3H), 0.97 (d, $J = 4.5$ Hz, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 177.2, 63.0, 50.5, 48.9, 36.2, 35.8, 35.7, 34.1, 31.3, 22.6, 15.1. Minor diastereomer (**25b**): $[\alpha]_D = +9.2$ (c 0.12, CHCl_3); IR (CH_2Cl_2) 3300, 1610 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 4.11 (br s, 1H), 3.73-3.47 (m, 3H), 3.40-3.21 (m, 2H), 3.03 (dd, $J = 4.0, 14.0$ Hz, 1H), 2.59-2.51 (m, 2H), 1.88-1.73 (m, 3H), 1.72-1.63 (m, 1H), 1.28-1.02 (m, 2H), 0.96 (d, $J = 6.7$ Hz, 3H), 0.95 (s, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 177.1, 63.1, 51.1, 50.3, 36.8, 36.2, 35.8, 34.8, 31.3, 22.6, 15.2. MS (CI) 200 ($\text{M}^+ + 1$, 100); HRFABMS calcd for

$C_{11}H_{22}NO_2$ ($M^+ + 1$): 200.1650; found: 200.1644. HPLC: t_R major: 7.7 min, t_R minor: 6.8 min; Chiracel OD-H; 90% hexane/isopropanol; flow rate 1mL/min; UV 220 nm.

(2'*R*,5*R*)-5-*tert*-Butyl-1-(2'-methylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (26a) and (2'*R*,5*S*)-5-*tert*-butyl-1-(2'-methylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (26b). Major diastereomer (**26a**): $[\alpha]_D = +9.3$ (c 1.1, $CHCl_3$); IR (CH_2Cl_2) 3360, 1610 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 3.77 (dd, $J = 8.9, 10.4$ Hz, 1H), 3.49-3.21 (m, 4H), 2.85 (dd, $J = 4.4, 13.9$ Hz, 1H), 2.63-2.49 (m, 2H), 2.18-1.94 (m, 2H), 1.79-1.73 (m, 1H), 1.32-1.20 (m, 3H), 1.00 (d, $J = 6.9$ Hz, 3H), 0.89 (s, 9H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 177.7, 63.5, 52.1, 50.9, 49.9, 36.5, 34.5, 33.5, 29.1, 27.9, 24.6, 15.6. Minor diastereomer (**26b**): $[\alpha]_D = +20.5$ (c 0.4, $CHCl_3$); IR (CH_2Cl_2) 3300, 1610 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 3.69-3.46 (m, 3H), 3.42-3.24 (m, 2H), 3.09 (dd, $J = 4.1, 14.0$ Hz, 1H), 2.64-2.43 (m, 2H), 2.11-1.99 (m, 2H) 1.87-1.79 (m, 1H), 1.66 (br s, 1H), 1.31-1.20 (m, 2H), 1.19-1.04 (m, 1H), 0.98 (d, $J = 7$ Hz, 3H). 0.89 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.2, 63.1, 51.4, 50.9, 50.8, 36.0, 34.9, 33.1, 29.6, 27.5, 24.1, 15.2. MS (CI) 242 ($M^+ + 1$, 100); HRFABMS calcd for $C_{14}H_{28}NO_2$ ($M^+ + 1$): 242.2120; found: 242.2102. HPLC: t_R major: 7.7 min, t_R minor: 6.8 min; Chiracel OD-H; 90% hexane/isopropanol; flow rate 1mL/min; UV 220 nm.

(2'*S*,5*R*)- and (2'*S*,5*S*)-5-Methyl-(2'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (27a,b). Isolated as a mixture of isomers: $R_f = 0.3$ (70% EtOAc/hexane). IR (CH_2Cl_2) 3280, 1610 cm^{-1} ; MS (CI) 262 ($M^+ + 1$, 88); HRFABMS calcd for $\text{C}_{16}\text{H}_{24}\text{NO}_2$ ($M^+ + 1$): 262.1807; found: 262.1784. HPLC: t_R **27a**: 9.1 min, t_R **27b**: 8.7 min; Chiracel OD-H; 93% hexane/ethanol; flow rate 1 mL/min; UV 254 nm. ^1H NMR (400 MHz, CDCl_3 , diagnostic peaks only) for **27a**: δ 4.31 (dd, $J = 10.5, 13.8$ Hz, 1H), 3.06 (dd, $J = 4.5, 13.9$ Hz, 1H). For **27b**: δ 4.04 (dd, $J = 9.0, 13.9$ Hz, 1H), 2.99 (dd, $J = 5.9, 15.3$ Hz, 1H). ^{13}C NMR (100.6 MHz, CDCl_3 , diagnostic peaks only) for **27a**: δ 177.3, 62.9. For **27b**: δ 177.2, 62.4.

(2'*S*,5*S*)- and (2'*S*,5*R*)-5-*tert*-Butyl-(2'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (28a, 28b). Isolated as a mixture of isomers: $R_f = 0.3$ (70% EtOAc/hexane). IR (CH_2Cl_2) 3300, 1620 cm^{-1} ; MS (CI) 304 ($M^+ + 1$, 100); HRFABMS calcd for $\text{C}_{19}\text{H}_{30}\text{NO}_2$ ($M^+ + 1$): 304.2276; found: 304.2299. HPLC: t_R **28a**: 10.2 min, t_R **28b**: 9.7 min; Chiracel OD-H; 94% hexane/ethanol; flow rate 1 mL/min; UV 254 nm. ^1H NMR (400 MHz, CDCl_3 , diagnostic peaks only): δ 4.25 (dd, $J = 10.1, 13.8$ Hz, 1H) (**28a**); δ 4.00 (dd, $J = 8.6, 13.9$ Hz, 1H) (minor diastereomer, diagnostic peaks only); ^{13}C NMR (100.6 MHz, CDCl_3): δ 177.8, 63.5 (**28a**); δ 177.9, 62.8 (**28b**).

Determination of stereochemistry of 28a,b. A mixture of **28a,b** (220 mg, 0.73 mmol) was added to a slurry of PCC (0.31 g, 1.45 mmol) and 0.3 g of silica gel in 15 mL CH_2Cl_2 . The orange reaction mixture was stirred at room temperature for 3 h, at which time the dark brown mixture was diluted with 30 mL Et_2O . The mixture was filtered through a Florisil pad and the pale yellow filtrate was dissolved in 25 mL THF followed by the addition of 52 mg (2.1 mmol) of NaH. The reaction mixture was refluxed for 20 h and then cooled to room temperature. The mixture was partitioned between Et_2O and water and the organic layer was washed with brine, dried (Na_2SO_4) and filtered. The filtrate was concentrated to an oil which upon chromatography using EtOAc, then 5% MeOH in CH_2Cl_2 as eluent afforded 18 mg of the dealkylated lactam **H** in 14% yield over two steps as a white solid. Spectral data for the dealkylated lactam matched that described above. Determination of optical rotation of the mixture of lactams ($[\alpha]_{\text{D}} = -4$ (c 0.5, MeOH)) and comparison with reported value⁹ for the enantiomerically pure *R* lactam (+)-**H** ($[\alpha]_{\text{D}} = +14.7$ (c 0.5, MeOH)) established an *ee* of 27% and established (*S*)-**H** as the major product. In addition, the *ee* of 27% corresponded to a ratio of 63:37, matching that obtained through HPLC analysis within experimental error.

5-Methyl-1-(1'-isopropylhydroxypropyl)-hexahydro-2H-azepine-2-one ((±)-29a,b**):** HPLC of the crude reaction mixture showed a 88:12 ratio of the diastereomeric lactam products. $R_f = 0.2$ (70% EtOAc/hexane). IR (CH_2Cl_2) 3330, 1610 cm^{-1} ; ^1H NMR (peaks for the major diastereomer **29a** from ^1H NMR of the mixture of diastereomers) (400 MHz, CDCl_3) δ 4.01 (br s, 1H), 3.99-3.95 (m, 1H), 3.63-3.56 (m, 1H), 3.42-3.35 (m, 1H), 3.14 (dd, $J = 6.2, 15$ hz, 1H), 2.83 (dd, $J = 4.1, 13.8$ Hz, 1H), 2.62-2.51 (m, 2H), 1.93-1.65 (m, 4H), 1.30-1.13 (m, 3H), 1.01 (d, $J = 6.8$ Hz, 3H), 0.97 (d, $J = 6.6$ Hz, 3H), 0.96 (d, $J = 6.8$ Hz, 3H), 0.85-0.80 (m, 1H); ^{13}C NMR (peaks for **29b** from ^{13}C NMR of the mixture of diastereomers) (100.6 MHz, CDCl_3) δ 177.3, 59.6, 48.5, 47.6, 45.2, 36.3, 35.8, 35.7, 31.4, 27.6, 20.9. MS (CI) 228 ($\text{M}^+ + 1$, 90); HRFABMS calcd for $\text{C}_{13}\text{H}_{26}\text{NO}_2$ ($\text{M}^+ + 1$): 228.1964; found: 228.1986. HPLC: t_R major: 9.1 min, 9.7 min, t_R minor: 6.9 min, 7.3 min; Chiracel OD-H; 93% hexane/isopropanol; flow rate 1 mL/min; UV 220 nm.

5-tert-butyl-1-(1'-isopropylhydroxypropyl)-hexahydro-2H-azepine-2-one ((±)-30a,b**):** Mixture: $R_f = 0.2$ (70% EtOAc/hexane). IR (CH_2Cl_2) 3330, 1650 cm^{-1} ; ^1H NMR (peaks for **30a** from ^1H NMR of the mixture of diastereomers) (400 MHz, CDCl_3) δ 4.09 (br s, 1H), 3.97 (t, $J = 12.6$ Hz, 1H), 3.68-3.60 (m, 1H), 3.42-3.33 (m, 2H), 3.17 (dd, $J =$

6.2, 14.9 Hz, 1H), 2.84 (dd, $J = 4.2, 13.9$ Hz, 1H), 2.64-2.40 (m, 2H), 2.18-1.84 (m, 2H), 1.83-1.73 (m, 1H), 1.63-1.43 (m, 1H), 1.31-1.16 (m, 3H), 1.01 (d, $J = 6.8$ Hz, 3H), 0.96 (d, $J = .8$ Hz, 3H), 0.88 (s, 9H); ^{13}C NMR (peaks for **30b** from ^{13}C NMR of the mixture of diastereomers) (100.6 MHz, CDCl_3) δ 177.1, 59.4, 51.4, 48.6, 47.1, 45.0, 35.8, 32.9, 28.3, 27.3, 23.9, 20.6. MS (+FAB) 270 ($\text{M}^+ + 1$, 75); HRFABMS calcd for $\text{C}_{16}\text{H}_{32}\text{NO}_2$ ($\text{M}^+ + 1$): 270.2433; found: 270.2411. HPLC: t_{R} major: 9.2 min, 9.8 min, t_{R} minor: 7.0 min, 7.4 min; Chiracel OD-H; 93% hexane/isopropanol; flow rate 1mL/min; UV 220 nm.

(1'S,3'R,5S)-5-methyl-(3'-hydroxy-1'-methylbutyl)-hexahydro-2H-azepine-2-one (31a). This was the only isomer isolated from the reaction when run and purified under the usual conditions. $[\alpha]_{\text{D}} = -38.4$ (c 0.52, CHCl_3); IR (CH_2Cl_2) 3300, 1610 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 4.83-4.75 (m, 1H), 3.80-3.59 (m, 1H), 3.39-3.17 (m, 2H), 2.86 (br s, 1H), 2.57-2.45 (m, 2H), 1.93-1.78 (m, 2H), 1.67-1.58 (m, 2H), 1.56-1.40 (m, 1H), 1.28-1.22 (m, 1H), 1.22-1.02 (m, 1H), 1.17 (d, $J = 6.2$ Hz, 3H), 1.13 (d, $J = 6.9$ Hz, 3H), 0.94 (d, $J = 6.6$ Hz, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 176.5, 66.4, 47.1, 44.0, 42.2, 38.2, 37.0, 36.9, 31.9, 24.0, 23.1, 19.7. MS (CI) 214 ($\text{M}^+ + 1$, 100); HRFABMS calcd for $\text{C}_{12}\text{H}_{24}\text{NO}_2$ ($\text{M}^+ + 1$): 214.1807; found: 214.1796. HPLC: t_{R} major: 5.7 min, t_{R} minor: 4.9 min; Chiracel OD-H; 90% hexane/isopropanol; flow rate 1mL/min; UV 220 nm.

32a

(1'*S*, 3'*R*, 5*S*)-5-*tert*-Butyl-1-(3'-hydroxy-1'-methylbutyl)hexahydro-2H-azepin-2-one (32a). This was the only isomer isolated from the reaction when run and purified under the usual conditions. The ^1H spectrum of this compound was concentration-dependent. $[\alpha]_{\text{D}} = -35.4$ (c 0.98, CHCl_3); ^1H NMR (500 MHz, CDCl_3 , $c = 0.025$ M) δ 0.84 (s, 9H), 1.04 (m, 1H), 1.12 (d, $J = 6.9$ Hz, 3H), 1.14 (d, $J = 6.2$ Hz, 3H), 1.19 (m, 1H), 1.41-1.46 (m, 1H), 1.54-1.60 (m, 1H), 1.92-2.01 (m, 2H), 2.38-2.43 (m, 1H), 2.55 (dd, $J = 10.0, 15.0$ Hz, 1H), 2.85 (br s, 1H), 3.14 (dd, $J = 10.6, 15.2$ Hz, 1H), 3.28 (dd, $J = 6.4, 15.3$ Hz, 1H), 3.73-3.77 (m, 1H), 4.72-4.76 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 19.4, 23.5, 24.2, 27.4, 30.4, 32.9, 36.8, 42.1, 43.6, 46.5, 51.7, 66.0, 176.2; IR (CCl_4) 3400 (br), 2960 (s), 1620 (s) cm^{-1} ; MS (EI) m/e 255 ($\text{M}^+ + 1$), 237, 222, 84, 69, 57; HRMS calcd for $\text{C}_{15}\text{H}_{29}\text{NO}_2$: 255.2190, found 255.2209.

32b

(1'*S*, 3'*R*, 5*R*)-5-*tert*-Butyl-1-(3'-hydroxy-1'-methylbutyl)hexahydro-2H-azepin-2-one (32a). An authentic sample of isomer **32b** could be obtained by carrying out the ring-expansion reaction of *tert*-butylcyclohexanone (300 mg, 1.94 mmol) with **11** (0.30 g, 2.32 mmol) as in the general procedure, except that no solvent was used and the reactants were combined at room temperature. Careful flash chromatography (65% EtOAc/Hexane) afforded 10 mg (2%) of **32b** and 445 mg of **32a** (90%). Both isomers had an R_f of ca. 0.28 (65% EtOAc/Hexane). **32b**: $[\alpha]_{\text{D}} = -44.4$ (c 0.16, CHCl_3); ^1H NMR

(500 MHz, CDCl_3) δ 0.86 (s, 9H), 1.11 (d, $J = 6.9$ Hz, 3H), 1.22 (d, $J = 6.2$ Hz, 3H), 1.16-1.27 (m, 2H), 1.42-1.48 (m, 1H), 1.62-1.67 (m, 2H), 1.92-1.99 (m, 2H), 2.27 (br s, 1H), 2.42-2.46 (m, 1H), 2.59 (dd, $J = 7.3, 13.6$ Hz, 1H), 3.19 (dd, $J = 9.4, 15.4$ Hz, 1H), 3.34 (dd, $J = 6.6, 15.2$ Hz, 1H), 3.83 (m, 1H), 4.73-4.78 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 19.1, 23.8 (two peaks), 27.5, 30.0, 33.0, 36.7, 42.0, 43.9, 46.7, 51.3, 66.3, 175.8; IR (CDCl_3) 1615 (s) cm^{-1} ; MS (EI) m/e 255 ($\text{M}^+ + 1$), 237, 222, 84, 69, 57.

(1'*R*,3'*R*,5*R*)-5-Methyl-(3'-hydroxy-1'-methylbutyl)-hexahydro-2*H*-azepine-2-one (33a) and (1'*R*,3'*R*,5*S*)-5-Methyl-(3'-hydroxy-1'-methylbutyl)-hexahydro-2*H*-azepine-2-one (33b). TLC (7:3 EtOAc/hexane) $R_f = 0.2$ (both isomers); IR (CH_2Cl_2) 3300, 1610 cm^{-1} ; **33a** $[\alpha]_D = +95.1$ (c 0.72, CHCl_3); ^1H NMR (400 MHz, CDCl_3) : δ 4.89 (m), 4.1 (m), 3.90-3.70 (m), 2.80 (m), 2.45 (m), 1.88-1.75 (m), 1.45 (m), 0.96 (d, $J = 6.6$ Hz, 3H); **33b** $[\alpha]_D = -47.5$ (c 0.9, CHCl_3); ^1H NMR (400 MHz, CDCl_3) : δ 4.85 (m), 3.79 (m), 3.25 (m), 2.51 (m), 1.92-1.40 (m), 1.2 (m), 0.90 (d, $J = 6.6$ Hz, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) **33a** : δ 178.58, 54.53, 50.95, 48.62, 36.6, 36.4, 31.6, 22.7, 22.1, 18.7; **33b** : δ 176.7, 66.69, 47.2, 44.0, 40.7, 38.2, 37.0, 31.9, 31.9, 23.9, 23.1, 19.8. MS (CI) 214 ($\text{M}^+ + 1$, 100); HRFABMS calcd for $\text{C}_{12}\text{H}_{24}\text{NO}_2$ ($\text{M}^+ + 1$): 214.1807; found: 214.1808. HPLC: t_R major: 12.2 min, t_R minor: 11.4 min; Chiracel OD-H; 95% hexane/ethanol; flow rate 1mL/min; UV 220 nm.

(1'*R*,3'*R*,5*R*)- and (1'*R*,3'*R*,5*S*)-5-*tert*-Butyl-(3'-hydroxy-1'-methylbutyl)-hexahydro-2*H*-azepine-2-one (34a,b). Mixture: $R_f = 0.2$ (70% EtOAc/hexane). Recrystallization from hexanes-EtOAc yielded the pure major diastereomer (stereostructure unassigned). For major diastereomer: $[\alpha]_D = +32.8$ (c 0.21, CHCl_3); IR (CH_2Cl_2) 3300, 1610 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 4.87-4.79 (m, 1H), 4.40 (br s, 1H), 3.48-3.44 (m, 1H), 3.27-3.22 (m, 1H), 3.13-3.06 (m, 1H), 2.69-2.63 (m, 1H), 2.55-2.49 (m, 1H), 2.05-1.98 (m, 2H), 1.43-1.39 (m, 2H), 1.31-1.22 (m, 2H), 1.21-1.10 (m, 1H), 1.20 (d, $J = 6.9$ Hz, 3H), 1.16 (d, $J = 6.3$ Hz, 3H), 0.89 (s, 9H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 178.2, 63.3, 52.0, 45.9, 43.9, 41.9, 36.7, 30.2, 29.7, 27.5, 24.4, 22.1, 18.8. Minor diastereomer (from spectrum of the mixture; diagnostic peaks only) ^{13}C NMR (100.6 MHz, CDCl_3) δ 178.2, 63.6, 44.4, 41.7, 39.9, 36.1, 33.1, 29.8, 27.4, 23.9, 22.2, 18.9, 16.4. MS (CI) 256 ($\text{M}^+ + 1$, 100); HRFABMS calcd for $\text{C}_{15}\text{H}_{30}\text{NO}_2$ ($\text{M}^+ + 1$): 256.2277; found: 256.2287. HPLC: t_R major: 12.0 min, t_R minor: 11.3 min; Chiracel OD-H; 95% hexane/ethanol; flow rate 1 mL/min; UV 220 nm.

(3'*R*,4*R*,6*S*)-4,6-Dimethyl-1-(3'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (35a) and (3'*R*,4*S*,6*R*)-4,6-dimethyl-1-(3'-phenylhydroxypropyl)-

hexahydro-2*H*-azepine-2-one (35b). Major diastereomer (**35a**): mp 89-90 °C. $[\alpha]_D = -11.1$ (c 1.25, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 0.93-1.03 (m, 1H), 0.95 (d, $J = 6.7$ Hz, 3H), 1.03 (d, $J = 6.6$ Hz, 3H), 1.61-1.75 (m, 1H), 1.75-1.88 (m, 2H), 1.88-1.98 (m, 2H), 2.35-2.45 (m, 2H), 2.96 (d, $J = 15.0$ Hz, 1H), 3.09 (dt, $J = 14.2, 4.5$ Hz, 1H), 3.36 (dd, $J = 15.0, 10.0$ Hz, 1H), 4.12-4.20 (m, 1H), 4.61 (dd, $J = 3.1, 10.0$ Hz, 1H), 7.23-7.28 (m, 1H), 7.30-7.39 (m, 4H); ¹³C NMR (100.6 MHz, CDCl₃) δ 21.1, 24.5, 30.3, 34.8, 37.8, 44.6, 46.1, 48.0, 56.9, 69.7, 125.6, 127.1, 128.3, 144.0, 176.0. IR (KBr) 3300, 1595 cm⁻¹. HRMS (EI) m/e calcd for C₁₇H₂₆NO₂: ($M^+ + 1$) 276.1963, found: 276.1944. Flash chromatography gave the minor diastereomer in diastereomerically enriched form only. Minor diastereomer (**35b**, diagnostic peaks only): ¹H NMR (400 MHz, CDCl₃) δ 0.93 (d, $J = 6.6$ Hz, 3H), 1.02 (d, $J = 6.6$ Hz, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 21.1, 34.5, 30.0, 33.3, 37.5, 44.6, 45.2, 48.1, 55.4, 69.6, 125.7, 127.1, 128.3, 144.0, 176.0. Anal. (mixture) Calcd for C₁₇H₂₅NO₂: C, 74.14; H, 9.15; N, 5.09; found: C, 74.38; H, 9.29; N, 5.06.

(3'*R*,4*R*)-4-methyl-1-(3'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (36a) and (3'*R*,6*R*)-6-methyl-1-(3'-phenylhydroxypropyl)-hexahydro-2*H*-azepine-2-one (36b). As described by the general procedure, (*R*)-3-methylcyclohexanone (154 mg, 1.38 mmol), (*R*)-3-azido-1-phenylpropanol (164 mg, 0.92 mmol), and BF₃•OEt₂ (0.46 mL, 3.68 mmol) were reacted to give crude iminium ethers. After hydrolysis and work up, HPLC of the crude reaction mixture indicated a 94:6 ratio of constitutional isomers (**36a**) and (**36b**). Flash chromatography (1:1 hexane/EtOAc→EtOAc→19:1 EtOAc/MeOH) gave 216 mg (90%) combined yield diastereomers (**36a**) and (**36b**).

Although a partial separation of **36a** and **36b** was possible by flash chromatography, it was found that a better separation occurred by flash chromatography of the crude iminium ether reaction mixture (30:1, CH₂Cl₂/MeOH) followed by hydrolysis to the major and minor diastereomers. The major lactam **36a** was never completely removed from the minor component **36b**. TLC (1:1 hexane/EtOAc): R_f (**36a**) 0.16, R_f (**36b**) = 0.20. **36a**: oil. [α]_D = -8.33 (c 1.2, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 1.03 (d, *J* = 6.7 Hz, 3H), 1.22-1.40 (m, 1H), 1.48-1.62 (m, 1H), 1.75-1.90 (m, 6H), 2.40-2.50 (m, 2H), 3.08 (dt, *J* = 14.1, 4.5 Hz, 1H), 3.20-3.30 (m, 1H), 3.40-3.50 (m, 1H), 4.14 (apparent septet, 1H), 4.60 (d, *J* = 10.2 Hz, 1H), 4.72 (apparent d, *J* = 3.2 Hz, 1H), 7.20-7.27 (m, 1H), 7.29-7.39 (m, 4H); ¹³C NMR (100.6 MHz, CDCl₃) δ 22.9, 27.9, 29.6, 32.9, 37.7, 38.3, 44.4, 45.7, 50.1, 69.7, 125.5, 127.0, 128.2, 144.0, 176.1. IR (CHCl₃) 3360, 2930, 1617 cm⁻¹. MS (FIA) *m/e* 262 (M⁺ + 1), 244, 140. **36b** (diagnostic peaks): ¹H NMR (400 MHz, CDCl₃) δ 0.95 (d, *J* = 6.8 Hz, 3H), 4.53 (d, *J* = 10.3 Hz, 1H), 4.74 (apparent d, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 20.4, 22.7, 33.0, 36.8, 37.4, 38.5, 45.3, 55.9.

(3'R,4S)-4-Phenyl-1-(3'-phenylhydroxypropyl)-pyrrolidin-2-one (37a) and (3'R,4R)-4-phenyl-1-(3'-phenylhydroxypropyl)-pyrrolidin-2-one (37b). Structures unassigned. TLC (15:1 CH₂Cl₂/MeOH): R_f (major) 0.55, R_f (minor) 0.60. Major diastereomer: [α]_D = +16.5 (c 0.23, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 1.80-2.01 (m, 2H), 2.60 (dd, *J* = 16.9, 7.6 Hz, 1H), 2.82 (dd, *J* = 16.9, 8.8 Hz, 1H), 3.27 (dt, *J* = 5.0, 14.3 Hz, 1H), 3.45-3.60 (m, 2H), 3.73 (dd, *J* = 9.1, 7.9 Hz, 1H), 3.85-3.95 (m, 2H), 4.62-4.68 (m, 1H), 7.20-7.24 (m, 2H), 7.24-7.30 (m, 2H), 7.30-7.40 (m, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 36.5, 37.2, 38.6, 39.5, 55.0, 70.4, 125.5, 126.6, 127.2, 127.3, 128.4,

129.0, 142.3, 143.9, 175.0. IR (film) 3360, 2920, 1655 cm^{-1} . MS (EI) m/e 295 (M^+), 278, 189, 175, 104, 91, 77; HRMS calcd for $\text{C}_{19}\text{H}_{22}\text{NO}_2$ ($M^+ + 1$) 296.1650; found ($M^+ + 1$) 296.1647. Minor diastereomer: $[\alpha]_D = +13.9$ (c 0.91, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 1.85-2.05 (m, 2H), 2.55 (dd, $J = 16.9, 8.6$ Hz, 1H), 2.83 (dd, $J = 16.9, 9.0$ Hz, 1H), 3.29 (dt, $J = 14.3, 5.2$ Hz, 1H), 3.38 (dd, $J = 9.6, 7.4$ Hz, 1H), 3.56 (pentet, $J = 8.0$ Hz, 1H), 3.75-3.90 (m, 3H), 4.65-4.70 (m, 1H), 7.18-7.23 (m, 2H), 7.24-7.30 (m, 2H), 7.30-7.41 (m, 6H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 36.5, 37.5, 38.8, 39.6, 54.8, 70.6, 125.6, 126.7, 127.2, 127.3, 128.4, 128.9, 142.0, 144.0, 175.0. IR (film) 3360, 2950, 1650, 1480 cm^{-1} . MS (EI) m/e 295 (M^+), 278, 189, 175, 104, 91, 77; HRMS m/e calcd for $\text{C}_{19}\text{H}_{22}\text{NO}_2$ ($M^+ + 1$) 296.1650; found ($M^+ + 1$) 296.1652. HPLC: t_R major = 14.3 min, t_R minor = 12.3 min; Chirobotic T; 80% hexane/EtOH; flow rate 1 mL/min; UV detector 254 nm.

(3'R,4aS,8aS)-2-(3'-Phenylhydroxypropyl)-1,4,4a,5,8,8a-hexahydroisoquinolin-3-one (58a) and (3'R,4aR,8aR)-2-(3'-phenylhydroxypropyl)-1,4,4a,5,8,8a-hexahydroisoquinolin-3-one (58b). Reaction of *cis*-1,3,3a,4,7,7a-hexahydro-2(H)-inden-2-one¹¹ (288 mg, 1.68 mmol) and **5** gave **38a** and **38b** as oils; the isomers were not assigned. TLC (1:3 hexane/EtOAc) R_f (major) = 0.21, R_f (minor) = 0.28. Major diastereomer: $[\alpha]_D = -2.3$ (c 1.54, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 1.75-2.05 (m, 4H), 2.15-2.30 (m, 4H), 2.35 (dd, $J = 17.9, 5.8$ Hz, 1H), 2.51 (dd, $J = 17.9, 5.4$ Hz, 1H), 3.05 (dt, $J = 14.1, 4.6$ Hz, 1H), 3.19 (dd, $J = 12.3, 7.1$ Hz, 1H), 3.34 (dd, $J = 12.3, 4.8$ Hz, 1H), 4.10-4.20 (m, 1H), 4.55 (br d, $J = 10.1$ Hz, 1H), 4.66 (br s, 1H), 5.58-5.68 (m, 2H), 7.20-7.27 (m, 1H), 7.30-7.39 (m, 4H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 26.4, 27.9, 29.4,

30.1, 35.6, 36.7, 44.0, 50.2, 69.9, 124.4, 124.8, 125.7, 127.1, 128.3, 144.1, 170.1. IR (neat) 3350, 2900, 1605 cm^{-1} . MS (EI) m/e 285 (M^+), 267, 207, 179, 165, 91, 77. Minor diastereomer: $[\alpha]_D = +13.6$ (c 0.31, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 1.75-2.05 (m, 4H), 2.15-2.30 (m, 4H), 2.39 (t, $J = 6.0$ Hz, 2H), 3.01 (dt, $J = 14.1, 4.6$ Hz, 1H), 3.23 (d, $J = 14.2$ Hz, 2H), 4.15-4.25 (m, 1H), 4.54 (br d, $J = 8.6$ Hz, 1H), 4.80 (br s, 1H), 5.65 (s, 2H), 7.21-7.26 (m, 1H), 7.30-7.38 (m, 4H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 26.0, 28.3, 29.2, 29.9, 35.0, 36.3, 43.6, 50.6, 69.7, 124.5, 124.7, 125.5, 127.0, 128.3, 144.0, 170.8. IR (neat) 3350, 2895, 1600 cm^{-1} MS (EI) m/e 285 (M^+), 267, 212, 179, 165, 91, 77.

39 (and isomers)

Reaction of 2-Bromo-4-*tert*-butyl-cyclohexanone with 2-Azido-2-phenylethanol. A solution of 2-bromo-4-*tert*-butyl-cyclohexanone¹² (0.173 g, 0.73 mmol) and azido alcohol ($\pm$)-**1** (0.140 g, 0.87 mmol) in 8 mL of CH_2Cl_2 was cooled to 0 °C. $\text{BF}_3 \cdot \text{OEt}_2$ (0.8 mL, 6.256 mmol) was added dropwise over 5 min; gas evolution was observed. The reaction was kept at 0 °C for 30 min, allowed to warm to room temperature, and stirred for 24 h. The solution was concentrated and 15% KOH was added to the residual oil. The reaction mixture was stirred for 1 h at room temperature. Additional CH_2Cl_2 was added and the organic layer was dried (anhydrous Na_2SO_4), filtered, and concentrated to afford crude product that was purified by column chromatography. The products isolated as a mixture of four different diastereomers (120 mg, 47%) with **39** being the major product in ca. 10:2:1:1 ratio of diastereomers. ($\pm$)-**6-Bromo-8-*tert*-butyl-3-phenyl-1-oxa-4-aza-spiro[4.5]dec-3-ene (39)** was purified by recrystallization and isolated as clear crystals (mp 152-153 °C): ^1H NMR (400 MHz,

CDCl₃) δ 0.90 (s, 9H), 1.30 (tt, J = 12.2, 3.0 Hz, 1H), 1.48 (m, 1H), 1.77 (m, 1H), 1.92-2.08 (m, 3H), 2.34 (m, 1H), 4.51 (dd, J = 12.8, 4.4 Hz, 1H), 5.03 (d, J = 13.5 Hz, 1H), 5.16 (d, J = 13.5 Hz, 1H), 7.46 (m, 3H), 7.76 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 22.9, 27.5, 32.5, 35.9, 36.5, 49.0, 60.4, IR 75.5, (neat) 2980 cm⁻¹; MS (CI) m/e 350 (M^+ + 1), 172; HRMS calcd for C₁₈H₂₄BrNO (M^+ + 1): 350.1120, found 350.1105. This compound was confirmed through X-ray crystallography.

Debromination of Crude 39. The crude mixture of bromides (9 mg, 0.02 mmol) was treated with 4 mL of benzene, 1 mg AIBN, and 8 mg (0.2 mmol) of tributyltin hydride. The reaction mixture was heated to reflux for 6 h. Evaporation of the solvent gave a mixture of **57a** and **57b** as a ca. 10 : 1 ratio of diastereomers (NMR). (±)-(8Z)-**tert-Butyl-3-phenyl-1-oxa-4-azaspiro[4.5]dec-3-ene (40a)**: oil; ¹H NMR (400 MHz, CDCl₃) δ 0.90 (s, 9H), 1.10 (m, 1H), 1.37 (m, 2H), 1.73 (m, 4H), 1.91 (m, 2H), 5.01 (s, 1H), 7.40-7.52 (m, 3H), 7.73 (m, 2H); ¹³C NMR (100 MHz) δ 24.1, 28.0, 32.8, 36.5, 47.3, 73.0, 111.3, 121.9, 128.0, 129.1, 131.2955 cm⁻¹; MS (CI) m/e 272 (M^+ + 1), 172; HRMS calcd for C₁₈H₂₅NO (M^+ + 1): 272.2014, found 272.1999. (±)-(8E)-**tert-Butyl-3-phenyl-1-oxa-4-azaspiro[4.5]dec-3-ene (40b)**. Oil; ¹H NMR (400 MHz, CDCl₃) δ 0.92 (s, 9H), 1.14 (m, 1H), 1.29-1.81 (m, 8H), 4.99 (m, 2H), 7.46 (m, 3H), 7.75 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 25.4, 27.7, 32.4, 38.2, 47.6, 72.8, 112.1, 127.7, 128.6, 131.0, 131.6, 164.7; IR (neat) 2960 cm⁻¹; MS

(CI) m/e 272 ($M^+ + 1$), 172; HRMS calcd for $C_{18}H_{25}NO$ ($M^+ + 1$): 272.2014, found 272.2001.

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1

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SMH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 456.1
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

(KF3.2026)
Current Data Parameters
NAME 2az2phenethanol
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 970814
Time 12.45
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1625.5
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.000200 sec
PL13 18.00 dB
D1 0.0500000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 0.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-43

SOLVENT CDCl₃
 NS 16
 OS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 812.7
 DM 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME 1phenyl2azido
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 970918
 Time 11.41
 INSTRUM drx400
 PROBD 5 mm Multinu
 PULPROG zgpg30
 TD 85936
 SOLVENT CDCl₃
 NS 1024
 OS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 512
 DM 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPOPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 13C
 PL2 0.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127713 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 0.50

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

NUC1 1H
P1 7.90 usec
PL1 -4.00 dB
SF01 500.1320005 MHz

===== CHANNEL f1 =====
NUC1 1H
P1 7.90 usec
PL1 -4.00 dB
SF01 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 500.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 4501.17 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.47500 ppm/cm
HZCM 237.56175 Hz/cm

Current Data Parameters
NAME 052p.58
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20000526
Time 15.11
INSTRUM spect
PROBHD 5 mm TXI 1H-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 594
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 1149.4
OW 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 7.00 usec
PL1 4.00 dB
SF01 125.7719472 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 65.00 usec
PL2 4.00 dB
PL12 20.00 dB
PL13 18.00 dB
SF02 500.1325000 MHz

F2 - Processing parameters
SI 65536
SF 125.7577951 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 190.000 ppm
F1 23893.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.50000 ppm/cm
HZCM 1194.69910 Hz/cm

SI-45

5

INSTRUM drx400
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 64
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
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
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME dsr-II-57c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020821
Time 11.30
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 146
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
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 100.6232933 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
F1P 220.000 ppm
F1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

SI-416

A

F2 - Acquisition Parameters
 Date_ 991029
 Time 12.56
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

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
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME RL-1-39
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 991102
 Time 7.58
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 2048
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

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
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -1008.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04639 Hz/cm

SI-47

2FS

PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 526
 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
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.50 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

 F2 - Processing parameters
 SI 32768
 SF 100.6127784 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

 1D NMR plot parameters
 CX 20.00 cm
 FID 200.000 ppm
 F1 20122.55 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1056.43420 Hz/cm

Current Data Parameters
 NAME ks11.291f
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000511
 Time 18.37
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 128
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 161.3
 DM 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 d1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

SI-48

B

TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	64
DW	104.400 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec
P1	7.70 usec
DE	4.50 usec
SFO1	400.1320007 MHz
NUC1	1H
PL1	-6.00 dB

F2 - Processing parameters

SI	16384
SF	400.1300036 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm

Current Data Parameters

NAME	ksii.297j
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	5000000
Time	12.18
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	64
DW	104.400 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec
P1	7.70 usec
DE	4.50 usec
SFO1	400.1320007 MHz
NUC1	1H
PL1	-6.00 dB

F2 - Processing parameters

SI	16384
SF	400.1300036 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm

SI-49

(±)-7

Time 12.49
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
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
SF 400.1299989 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME ksii.297c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20000614
Time 13.41
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 507
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 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 6.90 usec
PL1 -6.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127798 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

SI-50

8

F2 - Acquisition Parameters
 Date_ 1000511
 Time 18.23
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 128
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 45.3
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1299960 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME ks11.292b
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000512
 Time 13.24
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 477
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 D11 0.0300000 sec
 D12 0.0000000 sec
 PL13 12.00 dB
 D1 0.05000000 sec
 CPOPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.50 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127826 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.56 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1056.43420 Hz/cm

SI-
51

D

Parameters
290e
1
1
Parameters
7509
1.33
400
300
g30
32768
CDC13
NS
DS
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.132007 MHz
NUC1 1H
PL1 -6.00 dB
F2 - Processing parameters
SI 16384
SF 400.1300231 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06827 Hz/cm

Current Data Parameters
NAME ks11.290d
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000509
Time 20.23
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 26248.588 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 32768
DW 17.700 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.0500000 sec
CPOPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127806 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.43420 Hz/cm

SI-
52

E

HG 64
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

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

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME ks11.2901
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000509
 Time 20.49
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 537
 DS 2
 SWH 28248.588 Hz
 FIDRES 0.431039 Hz
 AQ 1.1600372 sec
 RG 32768
 DW 17.700 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 5.50 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127772 MHz
 NDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1056.43420 Hz/cm

SI-
53

9

Date_ 1000000
Time 10.36
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
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
P1 7.70 usec
DE 4.50 usec
SFO1 400.132007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300199 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCH 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME ksii.190f
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000102
Time 10.32
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 857
DS 2
SMH 26248.588 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 32768
DM 17.700 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPOPRG2 waltz16
PCPD2 100.00 usec
NUC2 13C
SF02 400.1316005 MHz
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127815 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCH 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

SI
54

10

F2 - Acquisition Parameters
 Date_ 1000511
 Time 18.10
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 128
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 32
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1299977 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 -4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME ks11.291b
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000511
 Time 17.06
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 680
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.00200 sec
 PL13 18.00 dB
 D1 0.0500000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127784 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1056.43420 Hz/cm

SI-55

Current Data Parameters
 NAME CK2_228top
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030207
 Time 7.53
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 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
 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 0.00 cm
 F1P 10.985 ppm
 F1 4395.28 Hz
 F2P -0.985 ppm
 F2 -393.99 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME CK2_228top
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030207
 Time 7.55
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 310
 DS 2
 SWH 23146.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 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 100.6232933 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 0.00 cm
 F1P 220.036 ppm
 F1 22138.42 Hz
 F2P -10.036 ppm
 F2 -1009.73 Hz
 PPMCM 11.50359 ppm/cm
 HZCM 1157.40747 Hz/cm

SI-56

F2 - Processing parameters
 S1 16384
 SF 400.1299896 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 ID NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 3201.04 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.42500 ppm/cm
 HZCM 170.05525 Hz/cm

Current Data Parameters
 NAME Ks11.180
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 991215
 Time 18.00
 INSTRUM dm4400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 1024
 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
 O11 0.035000 sec
 O12 0.000000 sec
 PL13 15.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB
 F2 - Processing parameters
 S1 32768
 SF 100.6127734 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40
 ID NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1056.43408 Hz/cm

Current Data Parameters

```
NAME      KSII.180W
EXPNO      11
PROCNO     1
```

F2 - Acquisition Parameters

Date	99121C
Time	18.25
INSTNUM	drx400
PROBHD	5 mm Multin
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	2
SWH	23148.148 Hz
FIDRES	0.352313 Hz
AQ	1.4156276 sec
RG	32768
DW	21.600 usec
DE	5.0 usec
TE	30.0 K
d11	0.0303200 sec
d12	0.0000200 sec
PL13	0.0000000 sec
d1	0.0500000 sec
CPDPR2	wait16
SPOR2	100.000 usec
SPOR2	400.1316075 MHz
NUC2	1H
PL2	-6.00 dB
PL12	18.00 dB
P1	6.90 usec
DE	4.50 usec
SF01	100.6232933 MHz
NUC1	13C
PL1	-6.00 dB

```
F2 - Processing parameters
SI                      32768
SF                      100.6127713 MHz
WDW                      EM
SSB                      0
LB                      1.00 Hz
GB                      0
PC                      1 40
```

```

10 NMR plot parameters
CX                20.00 cm
F1P              200.000 ppm
F1               20122.55 Hz
F2P              -10.000 ppm
F2               -1006.13 Hz
PPMCM            10.50000 ppm/cm
HZCM             1056.43408 Hz/cm

```

SI-
cm 58
m

12

F2 - Acquisition Parameters
 Date_ 1000102
 Time 9.46
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 64
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 OW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 d1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -5.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300033 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME ks11.190e
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 500000
 Time 10.09
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 799
 DS 2
 SWH 28248.588 Hz
 FIDRES 0.431039 Hz
 AQ 1.1600372 sec
 RG 32768
 OW 17.700 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waitz15
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 1H
 PL2 -5.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127720 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 260.000 ppm
 F1 26159.32 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 13.50000 ppm/cm
 HZCM 1358.27234 Hz/cm

SI-59

13

PROBHD 5 mm
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 256
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

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
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME miniukmeth
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 970827
Time 11.54
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPOPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 13C
PL2 0.00 dB
PL12 18.00 dB
P1 6.50 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 220134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-60

14a

RG 90.5
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 S1 16384
 SF 400.1300056 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME kf4.155maj13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980704
 Time 19.41
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 2048
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.0500000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1318005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 S1 32768
 SF 100.6127727 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

SI-61

14b

FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1.1449.4
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.0500000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

Current Data Parameters
NAME kf4.155min
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 980705
Time 11.35
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300068 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.95000 ppm/cm
HZCM 220.07150 Hz/cm

SI-62

CU
NA
EX
PR

F2
De
T1
T2
P1

PULPROG 2930
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
FIP 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME k14.157maj13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980703
Time 15.06
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D11 0.0300000 sec
D12 0.0000200 sec
PL13 18.00 dB
D1 0.0500000 sec
CPOPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -5.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127688 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
FIP 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-63

0.146157 Hz
 AQ 3.4210291 sec
 RG 30.5
 DM 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

 F2 - Processing parameters
 SI 16384
 SF 400.1300065 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

 1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME k14.157min13C
 EXPNO 1
 PROCNO 1

 F2 - Acquisition Parameters
 Date_ 980704
 Time 8.56
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 1024
 DM 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPOPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

 F2 - Processing parameters
 SI 32768
 SF 100.6127727 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

 1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

SI-64

16a

SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 90.5
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 970829
Time 0.31
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 0.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1008.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

SI-65

NS
DS
SWH
FID
AQ 3.4210291 sec
RG 456.1
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
FIP 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

F2 - Acquisition Parameters
Date_ 970828
Time 10.48
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 0.00 dB
PL12 18.00 dB
P1 6.50 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127713 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
FIP 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1167.04000 Hz/cm

SI-666

Date_ 21.26
Time drx400
INSTRUM 5 mm Multinu
PROBHD zg30
PULPROG 32768
TD CDC13
SOLVENT 16
NS 2
DS 4789.272 Hz
SWH 0.146157 Hz
FIDRES 3.4210291 sec
AQ 90.5
RG 104.400 usec
DM 4.50 usec
DE 300.0 K
TE 1.00000000 sec
D1 7.70 usec
P1 4.50 usec
DE 400.1320007 MHz
SF01 1H
NUC1 -6.00 dB
PL1

F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

F2 - Acquisition Parameters
Date_ 970903
Time 22.02
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 0.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

SI-67

DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME KF3.206min13C
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 980705
Time 19.25
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127692 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.50

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-68

PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 16
OS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 322.5
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.132007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300079 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

F2 - Acquisition Parameters
Date_ 970915
Time 13.49
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 1024
OS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1024
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.0500000 sec
CROPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 0.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

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
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04639 Hz/cm

SI-69

F2 - Acquisition Parameters
 Date_ 980401
 Time 21.34
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 256
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PRMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME K14.083AmajC13
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980401
 Time 23.38
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 512
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.0500000 sec
 CPOPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 5.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127720 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 0.80

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PRMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980401
Time 22.16
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07152 Hz/cm

Current Data Parameters
NAME K14.083AminC13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980401
Time 22.47
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1024
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
S11 0.0300000 sec
S12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPOPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-71

TO 32768
SOLVENT CDC13
NS 16
OS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 256
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
O1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME KF4.106.C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980512
Time 17.46
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1024
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
d1 0.05000000 sec
CROPPAG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127713 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

Processing parameters:

- WJ: 4799.272 Hz
- SWH: 0.146157 Hz
- FIDRES: 3.4210281 sec
- RG: 181
- DW: 104.400 usec
- DE: 4.50 usec
- TE: 300.0 K
- D1: 1.0000000 sec
- P1: 7.70 usec
- DE: 4.50 usec
- SFO1: 400.132007 MHz
- NUC1: 1H
- PL1: -6.00 dB

Processing parameters:

- SI: 16384
- SF: 400.1300100 MHz
- WDW: EM
- SSB: 0
- LB: 0.30 Hz
- GB: 0
- PC: 1.00

1D NMR plot parameters:

- CX: 20.00 cm
- F1P: 10.000 ppm
- F1: 4001.30 Hz
- F2P: -1.000 ppm
- F2: -400.13 Hz
- PPMCM: 0.55000 ppm/cm
- HZCM: 220.07150 Hz/cm

Current Data Parameters:

- NAME: Kf4.120maj.C13
- EXPNO: 2
- PROCNO: 1

F2 - Acquisition Parameters:

- Date_: 980615
- Time: 20.46
- INSTRUM: drx400
- PROBHD: 5 mm Multinu
- PULPROG: zgpg30
- TD: 65536
- SOLVENT: CDCl3
- NS: 1024
- DS: 2
- SWH: 23148.148 Hz
- FIDRES: 0.353213 Hz
- AQ: 1.4156276 sec
- RG: 812.7
- DW: 21.600 usec
- DE: 4.50 usec
- TE: 300.0 K
- d11: 0.0300000 sec
- d12: 0.0000200 sec
- PL13: 18.00 dB
- D1: 0.05000000 sec
- CPDPRG2: waltz16
- PCPD2: 100.00 usec
- SFO2: 400.1316005 MHz
- NUC2: 13C
- PL2: -6.00 dB
- PL12: 18.00 dB
- P1: 6.90 usec
- DE: 4.50 usec
- SFO1: 100.6232933 MHz
- NUC1: 13C
- PL1: -6.00 dB

F2 - Processing parameters:

- SI: 32768
- SF: 100.6127734 MHz
- WDW: EM
- SSB: 0
- LB: 1.00 Hz
- GB: 0
- PC: 1.40

1D NMR plot parameters:

- CX: 20.00 cm
- F1P: 220.000 ppm
- F1: 22134.81 Hz
- F2P: -10.000 ppm
- F2: -1006.13 Hz
- PPMCM: 11.50000 ppm/cm
- HZCM: 1157.04688 Hz/cm

FIORIS 0.140000
AQ 3.4210291 sec
RG 114
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME k14.160min13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980710
Time 0.35
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1024
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPOPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127734 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

10 NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-74

21a

5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146197 Hz
AQ 3.4210291 sec
RG 322.5
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME KF4.017C13na
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980211
Time 11.54
INSTRUM gpc400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPOPRG2 wait16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 0.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127713 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm

F2 -
Date
Time
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 362
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

NAME KF4.017C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980210
Time 20.41
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 0.00 dB
PL12 18.00 dB
P1 5.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127706 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-76

Acquisition parameters:

- RG 104.400 usec
- DE 4.50 usec
- TE 300.0 K
- D1 1.00000000 sec
- P1 7.70 usec
- DE 4.50 usec
- SFO1 400.1320077 MHz
- NUC1 1H
- PL1 -6.00 dB

F2 - Processing parameters:

- SI 16384
- SF 400.1300077 MHz
- WDW EM
- SSB 0
- LB 0.30 Hz
- GB 0
- PC 1.00

1D NMR plot parameters:

- CX 20.00 cm
- F1P 10.000 ppm
- F1 4001.30 Hz
- F2P -1.000 ppm
- F2 -400.13 Hz
- PPMCM 0.55000 ppm/cm
- HZCM 220.07150 Hz/cm

Current Data Parameters:

- NAME KF4.135.C13
- EXPNO 1
- PROCNO 1

F2 - Acquisition Parameters:

- Date_ 980528
- Time 23.37
- INSTRUM drx400
- PROBHD 5 mm Multinu
- PULPROG zgpg30
- TD 65536
- SOLVENT CDCl3
- NS 512
- DS 2
- SWH 23148.148 Hz
- FIDRES 0.353213 Hz
- AQ 1.4156276 sec
- RG 2048
- DW 21.600 usec
- DE 4.50 usec
- TE 300.0 K
- d11 0.0300000 sec
- d12 0.0000200 sec
- PL13 18.00 dB
- D1 0.05000000 sec
- CPDPRG2 waltz16
- PCPD2 100.00 usec
- SFO2 400.1316005 MHz
- NUC2 1H
- PL2 -6.00 dB
- PL12 18.00 dB
- P1 6.90 usec
- DE 4.50 usec
- SFO1 100.6232933 MHz
- NUC1 13C
- PL1 -6.00 dB

F2 - Processing parameters:

- SI 32768
- SF 100.6127713 MHz
- WDW EM
- SSB 0
- LB 1.00 Hz
- GB 0
- PC 1.40

1D NMR plot parameters:

- CX 20.00 cm
- F1P 220.000 ppm
- F1 22134.81 Hz
- F2P -10.000 ppm
- F2 -1006.13 Hz
- PPMCM 11.50000 ppm/cm
- HZCM 1157.04688 Hz/cm

SI-77

Current Data Parameters
 NAME ks11.232g
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000124
 Time 17.46
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 128
 DW 104.400 usec
 DT 4.50 usec
 TE 300.0 K
 O 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300053 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME ks11.232h
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000124
 Time 18.10
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 28248.588 Hz
 FIDRES 0.431039 Hz
 AQ 1.1600372 sec
 RG 32768
 DW 17.700 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 O1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127737 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.59000 ppm/cm
 HZCM 1157.04688 Hz/cm

SI-
78

Current Data Parameters
NAME ks11.232c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000118
Time 17.49
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 28248.588 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 32768
DM 17.700 usec
DE 4.50 usec
TE 300.0 K
d11 0.300000 sec
d12 0.000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127703 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

Current Data Parameters
NAME ks11.232a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000118
Time 13.26
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 512
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
d1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME ks11.280r
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000406
Time 18.48
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 26248.588 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 32768
DW 17.700 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.0500000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

23a

Current Data Parameters
NAME ks11.280p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000406
Time 15.55
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
d1 1.0000000 sec
d11 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300001 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06925 Hz/cm

SI-80

Current Data Parameters
 NAME ks11.281j
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000410
 Time 7.19
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 38847
 DS 2
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 8192
 DM 16.500 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127709 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1096.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

Current Data Parameters
 NAME ks11.280n
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000405
 Time 13.40
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 812.7
 DM 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

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

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

SI-81

Current Data Parameters
 NAME ksii.297e
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20000614
 Time 16.13
 INSTRUM drr400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 161.3
 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
 SF 400.1300039 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME ksii.297g
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20000614
 Time 17.21
 INSTRUM drr400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1173
 DS 2
 SWH 28248.588 Hz
 FIDRES 0.431039 Hz
 AQ 1.1600372 sec
 RG 32768
 DW 17.700 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 6.90 usec
 PL1 -6.00 dB
 SFO1 100.6232933 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 FCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127754 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1056.43420 Hz/cm

SI-82

25a

Current Data Parameters
NAME ks11.233y
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000129
Time 12.38
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 181
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
d1 1.00000000 sec
d11 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300044 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 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

Current Data Parameters
NAME ks11.233z
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000129
Time 13.59
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 28248.568 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 32768
DW 17.700 usec
DE 4.50 usec
TE 300.0 K
d11 0.03000000 sec
d12 0.0000200 sec
PL13 18.00 dB
d1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127728 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-
83

25b

Current Data Parameters
NAME ksii.233w
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000129
Time 12.31
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 256
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME ksii.233v
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000129
Time 12.23
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 26248.568 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 32768
DW 17.700 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CQPPRG2 waitz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-
84

Current Data Parameters
 NAME RL-I-12majorcr
 EXPNO 2
 PROCNO 2

F2 - Acquisition Parameters
 Date_ 990818
 Time 17.42
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 23148.146 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 1024
 DM 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d1 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.50 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

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

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04639 Hz/cm

Current Data Parameters
 NAME RL-I-12majorcr
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990818
 Time 14.26
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 181
 DM 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

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

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -5.000 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME Ks11.233a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000127
 Time 16.44
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 256
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300085 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME Ks11.234a
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000130
 Time 11.49
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 28248.588 Hz
 FIDRES 0.431039 Hz
 AQ 1.1600372 sec
 RG 32768
 DW 17.700 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127737 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

SI-86

Current Data Parameters
 NAME RL-1-24C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990908
 Time 13.09
 INSTRUM drx400
 PROBDH 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 S1 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
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPWCM 0.52500 ppm/cm
 HZCM 210.05825 Hz/cm

Current Data Parameters
 NAME RL-1-24C
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990908
 Time 13.29
 INSTRUM drx400
 PROBDH 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 238
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 812.7
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.0500000 sec
 CPDPRG2 waltz16
 PCPD02 100.00 usec
 SFO2 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 S1 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
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPWCM 11.50000 ppm/cm
 HZCM 1157.04639 Hz/cm

SI-
87

+ 29a,b

Current Data Parameters
 NAME ksii.241n
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000303
 Time 15.47
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 2
 SMH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300006 MHz
 NDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME ksii.241p
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 1000304
 Time 15.57
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 2
 SMH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1316005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232833 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127720 MHz
 NDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

SI-
88

30 a, b

Current Data Parameters
NAME ks11.243k
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000314
Time 10.34
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 574.7
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.132007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300068 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME ks11.281n
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000410
Time 15.48
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 32768
DM 15.500 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127875 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

SI-89

31a

Current Data Parameters
NAME ksii.296b
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000603
Time 14.37
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300021 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME ksii.296c
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000603
Time 15.09
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 85536
SOLVENT CDC13
NS 1396
DS 2
SWH 28248.588 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 32768
DW 17.700 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
CDPRG2 waitz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127746 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

SI-90

32a

NS
DS
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
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
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 0.00 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCM 0.59846 ppm/cm
HZCM 239.46359 Hz/cm

Current Data Parameters
NAME 32a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030207
Time 14.18
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 485
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
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 100.6232933 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 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

SI-
91

Single diastereomer

32b

Time 11.05
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 161.3
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
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 0.00 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCM 0.59846 ppm/cm
HZCM 239.46359 Hz/cm

Current Data Parameters
NAME CK2_229minormaybe
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030208
Time 11.08
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1109
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
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 100.6232933 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 0.00 cm
F1P 220.036 ppm
F1 22138.42 Hz
F2P -10.036 ppm
F2 -1009.73 Hz
PPMCM 11.50359 ppm/cm
HZCM 1157.40747 Hz/cm

SI-92

Current Data Parameters
 NAME 33amajor
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030131
 Time 12.44
 INSTRUM dxt400
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 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
 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 10.985 ppm
 F1 4395.28 Hz
 F2P -0.985 ppm
 F2 -393.99 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME 33amajor
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030131
 Time 12.46
 INSTRUM dxt400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 215
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 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 100.6232933 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 215.000 ppm
 F1 21631.74 Hz
 F2P -5.000 ppm
 F2 -503.06 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.73999 Hz/cm

SI-
93

data Parameters
33bminor
1
1

Position Parameters

20030131
Time 12.58
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 10
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
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
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 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCM 0.59846 ppm/cm
HZCM 239.46359 Hz/cm

Current Data Parameters

NAME 33bminor
EXPNO 1
PROCNO 2

F2 - Acquisition Parameters

20030202
Time 8.17
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1622
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
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 100.6232933 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 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

SI-
94

34a

Current Data Parameters
NAME RL-I-37cr
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 991102
Time 9.02
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 322.5
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

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
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME RL-I-37cr
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 991110
Time 12.50
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 824
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

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
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04639 Hz/cm

Current Data Parameter
 NAME RL-1 17
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 991011
 Time 17.02
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 128
 DM 104.400 usec
 DE 1.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

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
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -0.500 ppm
 F2 -200.07 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 210.06825 Hz/cm

Current Data Parameters
 NAME RL-1-37
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 991029
 Time 8.41
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SMH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 4096
 DM 21.800 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 O1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

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
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04639 Hz/cm

SI-
96

35a

SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16394
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
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME KF4.152.naj13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 980621
Time 23.27
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156278 sec
RG 1024
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.0500000 sec
CPOPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232333 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127706 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-97

36a

SWH 631.411 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 4096
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.0500000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1315005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127734 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

Current Data Parameters
 NAME kf4.167maj1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980718
 Time 0.32
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 90.5
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300079 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

37a

37a

NAME 980403
Time 17.32
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 812.7
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME KF4.0830.Rt15.
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 980406
Time 22.25
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1290.2
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1315005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232833 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127713 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-99

37b

PROCNO 1

F2 - Acquisition Parameters
 Date_ 980403
 Time 17.18
 INSTRUM drx400
 PROBN 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 1149.4
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300088 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCN 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME KF4.0830.Rt13.
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980406
 Time 23.01
 INSTRUM drx400
 PROBN 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 1149.4
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 D11 0.0300000 sec
 D12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPOPRG2 waltz16
 PCPO2 100.00 usec
 SF02 400.1318005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127734 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCN 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

SI-100

38a

NS 2
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 181
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 15384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME kf4.150.9.7min
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 980627
Time 2.04
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPOPRG2 wait16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127706 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

SI-101

38b

min

DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 128
 DM 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300085 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME kf4.150.7.6min
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980627
 Time 2.49
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 2048
 DM 21.500 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 O1 0.05000000 sec
 CPOPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1316005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127713 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 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

SI-102

39 (and isomers)

AD 3.4210291 sec
RG 512
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.47500 ppm/cm
HZCM 190.06175 Hz/cm

Current Data Parameters
NAME bs2o.80-fr-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000510
Time 9.01
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 186
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1024
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.0500000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL12 18.00 dB
P1 5.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127748 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 190.000 ppm
F1 19116.43 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.50000 ppm/cm
HZCM 955.82141 Hz/cm

SI-
103

Current Data Parameters
NAME bs2p.92-ir-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000518
Time 16.15
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 181
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.47500 ppm/cm
HZCM 190.06175 Hz/cm

Current Data Parameters
NAME bs2p.92-ir-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000519
Time 9.28
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 228
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1024
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 7.0300000 sec
d12 0.0000000 sec
PL13 18.00 dB
D1 0.0500000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127227 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 190.000 ppm
F1 19116.43 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.50000 ppm/cm
HZCM 955.82129 Hz/cm

SI-104

Current Data Parameters
NAME bs2p.92-fr-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000518
Time 16.05
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16394
SF 400.1300077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.47500 ppm/cm
HZCM 190.06175 Hz/cm

Current Data Parameters
NAME bs2p.92-fr-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000518
Time 16.11
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 162
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 1H
PL1 -6.00 dB

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
F1P 190.000 ppm
F1 19116.42 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.50000 ppm/cm
HZCM 955.82092 Hz/cm

SI-105

40 a,b

Current Data Parameters
NAME bs2c.92
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000513
Time 9.38
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 8
DW 104.400 usec
DE -4.50 usec
TE 300.0 K
D1 1.0000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.132007 MHz
NUC1 1H
PL1 -6.00 dB

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
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.47500 ppm/cm
HZCM 190.06175 Hz/cm

Current Data Parameters
NAME bs2p.95
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000514
Time 8.40
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 376
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DW 21.600 usec
DE -4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.0500000 sec
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127777 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 190.000 ppm
F1 19116.43 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.50000 ppm/cm
HZCM 955.82141 Hz/cm

SI-106