Asymmetric Total Synthesis of Dendrobatid Alkaloid 251F

Aaron Wroblewski, Kiran Sahasrabudhe, and Jeffrey Aubé*

*Department of Medicinal Chemistry
University of Kansas
Lawrence, Kansas 66045-2506*

Supporting Information

Experimental details and characterization data for new compounds (34 pages)

Experimental Section

3-Methyl-bicyclo[2.2.1]hept-5-ene-2-carboxylic acid methoxy-methyl-amide

(A). Acid **4**¹ (4.5 g, 29.6 mmol) was dissolved in 125 mL of CH₂Cl₂ and stirred at room temperature. To this solution was added BOP (13.2 g, 29.6 mmol) and TEA (4.3 mL, 31.1 mmol). The resulting solution was stirred for 0.5 h upon which time HNMe(OMe)•HCl (3.18 g, 32.6 mmol) and TEA (4.3 mL, 31.1 mmol) were added and the reaction was stirred at room temperature for 24 h. The reaction mixture was then quenched with 1 N HCl. The aqueous layer was extracted with CH₂Cl₂, and the combined organic layers were subsequently washed with brine, dried over Na₂SO₄, filtered, concentrated, and chromatography (20% EtOAc/hexanes) yielded the amide (5.48 g, 95% yield) as a clear, colorless oil. [α]_D -148.2 (*c* 1.50, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 1.11 (d, *J* = 7.0 Hz, 3H), 1.39 (m, 1H), 1.57 (d, *J* = 8.5 Hz, 1H), 1.91 (m, 1H), 2.44 (br s, 1H), 2.61 (t, *J* = 3.8 Hz, 1H), 3.06 (br s, 1H), 3.12 (s, 3H), 3.68 (s, 3H), 5.89 (dd, *J* = 2.8, 5.6 Hz, 1H), 6.25 (dd, *J* = 3.1, 5.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 20.7, 32.4, 37.0, 46.5, 46.6, 49.2, 49.7, 61.0, 132.3, 137.9, 175.3; IR (neat) 1670 cm⁻¹; MS (CI) *m/e* 196 (M⁺ + 1); HRMS calcd for C₁₁H₁₈NO₂ (M⁺ + H): 196.1338, found 196.1313.

1-(3-Methyl-bicyclo[2.2.1]hept-5-en-2-yl)-propenone (5). The Weinreb amide **A** (980 mg, 5.03 mmol) was dissolved in 40 mL of THF and cooled to 0 °C. Vinyl magnesium bromide (1.0 M in THF, 15.1 mL, 15.1 mmol) was added dropwise and the solution stirred at 0 °C for 1 h. The reaction was then quenched with 1 N HCl, and the aqueous layer was extracted with Et₂O. The combined organic layers were washed with brine and dried over Na₂SO₄. Chromatography (5% EtOAc/hexanes) yielded **5** (775 mg,

95% yield) as a clear, colorless oil. $[\alpha]_D -97.9$ (c 0.38, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 1.15 (d, $J = 7.0$ Hz, 3H), 1.44-1.46 (m, 1H), 1.63 (d, $J = 8.5$ Hz, 1H), 1.95-1.98 (m, 1H), 2.48 (br s, 1H), 2.67 (t, $J = 4.3$ Hz, 1H), 3.13 (br s, 1H), 5.69 (dd, $J = 1.4, 10.5$ Hz, 1H), 5.83 (dd, $J = 2.8, 5.6$ Hz, 1H), 6.20-6.25 (m, 2H), 6.46 (dd, $J = 10.5, 17.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 20.9, 35.4, 46.3, 46.6, 49.2, 59.0, 127.4, 132.2, 135.4, 138.5, 200.4; IR (neat) 1696 cm^{-1} ; MS (CI) m/e 163 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{11}\text{H}_{15}\text{O}$ ($\text{M}^+ + \text{H}$): 163.1123, found 163.1113.

(5S, 6R)-6-Methyl-5-vinyl-4,5,6,6a-tetrahydro-3a-H-pentalen-1-one (3).

Enone **5** (200 mg, 1.23 mmol, 0.01M) was dissolved in 120 mL of CH_2Cl_2 and stirred at room temperature. Ethylene was bubbled through the solution for ca. 5 min followed by addition of Grubbs catalyst (50 mg, 0.06 mmol). The reaction flask was flushed with ethylene, and the reaction was allowed to stir for 12 h at room temperature under an ethylene balloon. The reaction was concentrated and chromatographed with 1-3% EtOAc/hexanes to yield **3** (186 mg, 93% yield) as a clear, slightly black tinted oil. $[\alpha]_D +182$ (c 1.13, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 1.15 (d, $J = 6.6$ Hz, 3H), 1.18-1.27 (m, 1H), 1.48-1.55 (m, 1H), 2.11-2.18 (m, 1H), 2.27-2.32 (m, 1H), 2.37 (dd, $J = 7.0, 9.1$, 1H), 3.36-3.42 (m, 1H), 4.95-5.03 (m, 2H), 5.55-5.64 (m, 1H), 5.92 (dd, $J = 1.8, 5.6$, 1H), 7.58 (dd, $J = 2.6, 5.6$, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 17.6, 36.4, 42.2, 46.3, 56.5, 57.1, 115.2, 131.2, 139.7, 166.5, 211.7; IR (neat) 1705 cm^{-1} ; MS (CI) m/e 163 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{11}\text{H}_{15}\text{O}$ ($\text{M}^+ + \text{H}$): 163.1123, found 163.1107.

(4S)-5-Benzyloxy-4-methyl-1-pentene (B). NaH (785 mg, 19.6 mmol, 60% in mineral oil dispersion) was washed with hexanes and then suspended in 25 mL of anhydrous DMF. After cooling the above suspension to $0\text{ }^\circ\text{C}$, a solution of (*S*)-2-methyl-4-penten-1-ol² (1.63 g, 16.3 mmol) in 10 mL DMF was added dropwise. The mixture

was stirred for 15 min at 0 °C at which time benzyl bromide (3.62 g, 21.2 mmol) was added dropwise. The reaction was allowed to slowly warm to ambient temperature and then stirred overnight. The reaction was then quenched with water and extracted with EtOAc. The combined organic layers were washed with brine, dried over Na₂SO₄, filtered, concentrated, and chromatography (1-3% EtOAc/hexanes) yielded **B** (3.09 g, 100% yield) as a clear, colorless liquid. $[\alpha]_D +2.4$ (*c* 2.30, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 0.98 (d, *J* = 6.5 Hz, 3H), 1.89-2.01 (m, 2H), 2.25-2.31 (m, 1H), 3.32 (dd, *J* = 6.2, 9.1 Hz, 1H), 3.38 (dd, *J* = 6.1, 9.1 Hz, 1H), 4.55 (s, 2H), 5.03-5.09 (m, 2H), 5.78-5.88 (m, 1H), 7.31-7.39 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 16.7, 33.3, 38.0, 72.9, 75.3, 115.9, 127.4, 127.5, 128.3, 136.9, 138.7; IR (neat) 2900 cm⁻¹; MS (CI) *m/e* 191 (*M*⁺ + 1); HRMS calcd for C₁₃H₁₉O (*M*⁺ + H): 191.1436, found 191.1446.

(3S)-4-Benzyloxy-3-methyl-butylaldehyde (7). (4S)-5-Benzyloxy-4-methyl-1-pentene (**B**, 3.09 g, 16.3 mmol) was diluted in 100 mL of 1:1 ether/water and stirred at ambient temperature. To this solution was added OsO₄ (125 mg, 0.49 mmol) dissolved in 25 mL of water followed by NMNO (2.86 g, 24.5 mmol). The reaction was stirred for 5 min and NaIO₄ (5.24 g, 24.5 mmol) was then added in one portion. After stirring for 24 h, the reaction was filtered thru Celite, and the aqueous layer was extracted with ether. The combined organic layers were washed with brine, dried over Na₂SO₄, filtered, concentrated, and chromatography (5-10% EtOAc/hexanes) yielded **7** (2.20 g, 70% yield) as a clear, colorless liquid. $[\alpha]_D -11.2$ (*c* 1.70, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 1.01 (d, *J* = 6.8 Hz, 3H), 2.27-2.34 (m, 1H), 2.43-2.48 (m, 1H), 2.55-2.61 (m, 1H), 3.27 (dd, *J* = 7.6, 9.1 Hz, 1H), 3.44 (dd, *J* = 5.1, 9.1 Hz, 1H), 4.51 (s, 2H), 7.31-7.39 (m, 5H), 9.79 (t, *J* = 2.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 17.0, 29.0, 48.4, 73.0, 74.8, 127.50, 127.53, 128.3, 138.2, 202.4; IR (neat) 2750, 1725 cm⁻¹; MS (CI) *m/e* 193 (*M*⁺ + 1); HRMS calcd for C₁₂H₁₇O₂ (*M*⁺ + H): 193.1229, found 193.1241.

(3*S*, 3'*S*, 5*S*, 6*R*)-(E)-2-(4'-Benzyloxy-3'-methylbutylidene)-3,6-dimethyl-5-vinyl-hexahydro-penten-1-one (8). CuI (295 mg, 1.54 mmol) was suspended in 20 mL of THF. The mixture was cooled to 0 °C, and MeLi (1.4 M in Et₂O, 2.20 mL, 3.09 mmol) was added dropwise, causing the solution to initially form a heterogeneous yellow solution that ultimately turned clear with complete addition of MeLi. Enone **3** (200 mg, 1.23 mmol), diluted in 1 mL of THF, was added to the above cuprate solution dropwise, again forming a yellow, heterogeneous solution. The reaction mixture was stirred for 20 min at 0 °C followed by addition of aldehyde **7** (235 mg, 1.23 mmol) in 1 mL of THF. The resulting solution was stirred for 15 min at 0 °C, 15 min at room temperature, and then quenched with saturated NH₄Cl. The reaction was stirred at room temperature for ca. 1 h. The aqueous layer was extracted with Et₂O, and the combined organic layers were washed with brine, dried over Na₂SO₄, filtered, concentrated, and chromatography (1-3% EtOAc/hexanes) afforded **8** (325 mg, 60% yield) as a clear, colorless oil. [α]_D +60.0 (*c* 0.850, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 0.96 (d, *J* = 6.6 Hz, 3H), 0.98-1.08 (m, 1H), 1.12 (d, *J* = 7.2 Hz, 3H), 1.18 (d, *J* = 6.8 Hz, 3H), 1.77-1.80 (m, 1H), 1.99-2.11 (m, 4H), 2.35-2.47 (m, 3H), 2.75 (q, *J* = 7.1 Hz, 1H), 3.31-3.39 (m, 2H), 4.52 (s, 2H), 4.93-5.02 (m, 2H), 5.59-5.67 (m, 1H), 6.61 (dt, *J* = 1.5, 6.5 Hz, 1H), 7.28-7.37 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 16.8, 19.9, 21.6, 33.2, 33.6, 36.6, 40.4, 44.1, 46.1, 54.1, 58.3, 73.0, 75.1, 114.4, 127.5, 128.3, 136.8, 138.4, 140.6, 143.3, 209.8; IR (neat) 1709 cm⁻¹; MS (CI) *m/e* 353 (M⁺ + 1); HRMS calcd for C₂₄H₃₃O₂ (M⁺ + H): 353.2481, found 353.2467.

(2*R*, 3*S*, 3'*S*, 5*S*, 6*R*)-2-(4'-Hydroxy-3'-methylbutyl)-3,6-dimethyl-5-vinyl-hexahydro-penten-1-one (C). Ammonia (15 mL) was condensed into a 3-necked round

bottom flask fitted with a cold-finger condensor. To the flask was added 15 mL of THF and absolute ethanol (0.28 mL, 4.73 mmol), and the solution was stirred at -78 °C. Pieces of sodium were added to this solution until a dark blue color was maintained for 5 min. Enone **8** (330 mg, 0.95 mmol) in 3 mL of THF was added to the above solution dropwise. If the dark blue color dissipated, more sodium was added until the reaction mixture was again dark blue. The addition of enone was continued, adding more sodium when necessary. Upon complete addition of **8**, the dark blue reaction was allowed to stir for an additional 5 min at which time it was quenched with saturated NH₄Cl. The ammonia was allowed to evaporate, and the aqueous layer was extracted with Et₂O. The combined organic layers were washed with brine, dried over Na₂SO₄, filtered, concentrated, and chromatographed (5-10% EtOAc/hexanes) to yield **C** (134 mg, 54% yield) as a clear, colorless oil as an ca. 4:1 mixture of diastereomers. Major isomer **C**: ¹H NMR (400 MHz, CDCl₃) δ 0.91 (d, *J* = 6.7 Hz, 3H), 1.06 (d, *J* = 6.4 Hz, 3H), 1.10 (d, *J* = 6.4 Hz, 3H), 1.24-1.28 (m, 2H), 1.49-1.63 (m, 6H), 1.84-1.87 (m, 2H), 2.11-2.28 (m, 4H), 3.43-3.52 (m, 2H), 4.94-5.03 (m, 2H), 5.58-5.67 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 16.5, 17.1, 18.6, 24.0, 29.8, 35.8, 38.0, 41.8, 44.1, 46.3, 55.2, 58.8, 59.6, 67.6, 114.8, 140.7, 220.3; IR (neat) 3420, 1726 cm⁻¹; MS (CI) *m/e* 265 (M⁺ + 1); HRMS calcd for C₁₇H₂₉O₂ (M⁺ + H): 265.2168, found 265.2178. Minor diastereomer ¹³C NMR (100 MHz, CDCl₃) δ 16.9, 18.5, 21.2, 22.6, 31.0, 31.5, 35.7, 39.4, 45.8, 45.9, 49.4, 54.6, 56.9, 67.7, 115.0, 140.0, 221.6.

(2*R*, 3*S*, 3'*S*, 5*S*, 6*R*)-2-(4'-Azido-3'-methylbutyl)-3,6-dimethyl-5-vinyl-hexahydro-penten-1-one (9). Compound **C** (226 mg, 0.86 mmol) was diluted in 25 mL of anhydrous toluene and stirred at room temperature. To this solution was added Zn(N₃)₂•2 pyr (396 mg, 1.28 mmol) and triphenylphosphine (913 mg, 3.42 mmol). To the above heterogeneous solution was added DEAD (0.565 mL, 3.42 mmol), and the reaction was allowed to stir for 12 h at room temperature. The reaction was quenched with water, and the aqueous layer was extracted with Et₂O. The combined organic layers

were washed with brine, dried over Na₂SO₄, filtered, concentrated, and chromatographed (5% EtOAc/hexanes) to yield **9** (245 mg, 100% yield) as a clear, colorless oil. Major isomer: ¹H NMR (500 MHz, CDCl₃) δ 1.00 (d, *J* = 6.4 Hz, 3H), 1.10 (d, *J* = 6.4 Hz, 3H), 1.14 (d, *J* = 6.3 Hz, 3H), 1.28-1.39 (m, 3H), 1.51-1.59 (m, 4H), 1.69-1.74 (m, 1H), 1.87-1.91 (m, 1H), 2.15-2.34 (m, 4H), 3.15 (dd, *J* = 6.8, 11.9 Hz, 1H), 3.25 (dd, *J* = 5.9, 12.0 Hz, 1H), 4.99-5.06 (m, 2H), 5.63-5.70 (m, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 17.2, 17.6, 18.7, 24.6, 31.1, 33.9, 39.5, 42.0, 44.3, 46.4, 55.2, 57.6, 58.5, 59.7, 114.8, 140.7, 219.6; IR (neat) 2095, 1726 cm⁻¹; MS (CI) *m/e* 262 (*M*⁺ + 1 - 28). Minor diastereomer ¹³C NMR (125 MHz, CDCl₃) δ 16.9, 17.7, 18.5, 21.5, 32.1, 33.8, 35.6, 39.5, 45.9, 46.0, 49.3, 54.6, 56.9, 57.4, 115.0, 140.0, 221.0.

(1R, 2S, 3'S, 4S, 5R)-5-(4'-Azido-3'-methylbutyl)-1,4-dimethyl-6-oxooctahydro-pentalene-2-carbaldehyde (D). Azide **9** (130 mg, 0.45 mmol) was diluted in 4 mL of anhydrous CH₂Cl₂ and cooled to -78 °C. Ozone was bubbled through the solution until a blue tint was observed (ca. 3 min). To the resulting blue solution was added 3 mL of dimethyl sulfide. The reaction was allowed to warm to ambient temperature and stirred for 16-24 h at which point it was concentrated and chromatographed (5% EtOAc/hexanes) to yield **D** (92 mg, 70% yield) as a clear colorless oil. Major isomer: ¹H NMR (500 MHz, CDCl₃) δ 0.96 (d, *J* = 6.2 Hz, 3H), 1.14 (d, *J* = 6.4 Hz, 3H), 1.20 (d, *J* = 6.6 Hz, 3H), 1.47-1.56 (m, 4H), 1.66-1.79 (m, 3H), 1.85-1.89 (m, 1H), 2.15-2.33 (m, 3H), 2.38 (t, *J* = 9.4 Hz, 1H), 2.50-2.56 (m, 1H), 3.13 (dd, *J* = 6.8, 12.0 Hz, 1H), 3.21 (dd, *J* = 5.9, 12.0 Hz, 1H), 9.61 (d, *J* = 2.8 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 17.5, 18.4, 18.6, 24.6, 30.9, 31.0, 33.8, 37.7, 42.8, 47.1, 57.4, 58.1, 59.5, 61.4, 202.4, 218.1; IR (neat) 2720, 2090, 1721 cm⁻¹; MS (CI) *m/e* 264 (*M*⁺ + 1 - 28). Minor diastereomer ¹³C NMR (125 MHz, CDCl₃) δ 16.6, 17.6, 19.7, 21.6, 31.9, 33.2, 33.7, 35.6, 41.0, 46.4, 49.7, 56.8, 57.3, 61.6, 202.3, 220.0.

(2R , 3S , 3'S, 5S , 6R)-2-(4'-Azido-3'-methylbutyl)-3,6-dimethyl-5-hydroxymethyl-hexahydro-penten-1-one (2): Compound **D** (92 mg, 0.32 mmol) was diluted in 5 mL of EtOH and cooled to 0 °C. Sodium borohydride (12 mg, 0.32 mmol) was dissolved in ca. 1 mL of EtOH and added dropwise to the above solution. TLC analysis of the reaction mixture immediately after borohydride addition indicated consumption of all starting material. The reaction was quenched with 1N HCl, stirred for 10 min, and concentrated. The aqueous layer was extracted with CH₂Cl₂, and the combined organic layers were washed with brine, dried over Na₂SO₄, filtered, concentrated and chromatographed (3-10% EtOAc/hexanes) to yield **2** (72 mg, 78% yield) as a clear, colorless oil. [α]_D +11.9 (*c* 2.10, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 0.96 (d, *J* = 6.7 Hz, 3H), 1.12 (t, *J* = 6.8 Hz, 6H), 1.30-1.36 (m, 1H), 1.47-1.57 (m, 4H), 1.63-1.86 (m, 6H), 2.15-2.30 (m, 3H), 3.12 (dd, *J* = 6.8, 12.0 Hz, 1H), 3.21 (dd, *J* = 5.9, 12.0 Hz, 1H), 3.53 (dd, *J* = 6.4, 10.7 Hz, 1H), 3.70 (dd, *J* = 4.4, 10.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 17.5, 18.1, 18.6, 24.7, 31.0, 33.8, 34.2, 38.4, 43.5, 46.2, 51.9, 57.5, 58.1, 60.1, 64.6, 219.9; IR (neat) 3430, 2095, 1726 cm⁻¹; MS (CI) *m/e* 266 (M⁺ + 1 - 28).

6-Oxo-251F (10). Azide **2** (90 mg, 0.31 mmol) was diluted in 10 mL of CH₂Cl₂ and cooled to 0 °C. Triflic acid (0.135 mL, 1.54 mmol) was added dropwise and stirred at 0 °C until TLC indicated the consumption of all starting material (ca. 6 h). The reaction was then quenched with saturated NaHCO₃. The aqueous layer was extracted with CH₂Cl₂, and the combined organic layers were washed with brine, dried over

Na₂SO₄, filtered, concentrated and chromatographed (30-100% EtOAc/hexanes) to yield **10** (64 mg, 79% yield) as a clear, oily solid. Crystallization from Et₂O at 0 °C gave crystals suitable for X-ray analysis. $[\alpha]_D +28.9$ (*c* 1.00, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 0.87 (d, *J* = 6.6 Hz, 3H), 0.95 (d, *J* = 6.4 Hz, 3H), 0.98-1.07 (m, 2H), 1.20 (d, *J* = 6.2 Hz, 3H), 1.22-1.26 (m, 1H), 1.38-1.42 (m, 2H), 1.71-1.84 (m, 4H), 1.93 (t, *J* = 12.3 Hz, 1H), 2.00-2.08 (m, 2H), 2.18-2.23 (m, 2H), 2.66 (dt, *J* = 2.5, 9.7 Hz, 1H), 3.47 (dd, *J* = 6.6, 10.5 Hz, 1H), 3.68 (dd, *J* = 4.3, 10.6 Hz, 1H), 4.64-4.69 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 16.5, 18.6, 19.1, 30.5, 32.5, 32.7 (2C), 38.1, 40.3, 40.8, 48.7, 48.8, 52.3, 61.8, 65.2, 171.3; IR (neat) 3455, 1615 cm⁻¹; MS (CI) *m/e* 266 (*M*⁺ + 1); HRMS calcd for C₁₆H₂₈NO₂ (*M*⁺ + H): 266.2120, found 266.2146.

251F (1): Lactam **10** (111 mg, 0.42 mmol) was dissolved in 10 mL of anhydrous Et₂O and cooled to 0 °C. To this solution was added LAH (1.0 M in Et₂O, 0.840 mL, 0.84 mmol) dropwise. After 20 min at 0 °C, TLC indicated no trace of lactam starting material. The reaction was quenched with 30 μL water, followed by 30 μL of 15% KOH, and finally an additional 90 μL of water. The solution was stirred for 10 min at 0 °C, at which time MgSO₄ was added and stirring was continued for another 10 min. The entire solution was filtered through a cotton plug and washed with EtOAc. The eluant was concentrated to yield 251F (90 mg, 86% yield) as a white solid. $[\alpha]_D -11.1$ (*c* 0.960, CHCl₃); mp = 110-112 °C; ¹H NMR (400 MHz, D₂O) δ 0.79 (d, *J* = 6.3 Hz, 6H), 0.87 (d, *J* = 6.0 Hz, 3H), 0.95-1.23 (m, 4H), 1.51-1.60 (m, 2H), 1.68-1.77 (m, 4H), 1.95-2.00 (m, 1H), 2.10 (d, *J* = 14.5 Hz, 1H), 2.49 (t, *J* = 12.4 Hz, 1H), 2.54 (t, *J* = 11.0 Hz, 1H), 3.09 (dd, *J* = 3.3, 13.5 Hz, 1H), 3.17 (d, *J* = 12.3 Hz, 1H), 3.30 (d, *J* = 13.5 Hz, 1H), 3.37 (dd, *J* = 6.9, 10.7 Hz, 1H), 3.54 (dd, *J* = 4.4, 10.9 Hz, 1H); ¹³C NMR (100 MHz, D₂O) δ 14.7, 15.6, 17.5, 27.1, 28.3, 30.2, 31.4, 35.3, 37.6, 41.2, 44.3, 47.3, 53.1, 61.3, 64.8, 67.3; IR (neat) 3367, 2800, 2756 cm⁻¹; MS (CI) *m/e* 252 (*M*⁺ + 1); HRMS calcd for C₁₆H₃₀NO (*M*⁺ + H): 252.2327, found 252.2317.

References for Supporting Information

- (1) Evans, D. A.; Chapman, K. T.; Bisaha, J. *J. Am. Chem. Soc.* **1988**, *110*, 1238-1256.
- (2) Evans, D. A.; Bender, S. L.; Morris, J. *J. Am. Chem. Soc.* **1988**, *110*, 2506-2526.

Current Data Parameters
 NAME adw-IV-017
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011130
 Time 14.15
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 6009.615 Hz
 FIDRES 0.183399 Hz
 AQ 2.7264309 sec
 RG 32
 DM 83.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.03000000 sec

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

F1 - Acquisition parameters
 NDO 2
 TD 256
 SF01 500.1325 MHz
 FIDRES 23.475060 Hz
 SN 12.016 ppm

F2 - Processing parameters
 SI 32768
 SF 500.1300132 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

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

1D NMR plot parameters
 CX 20.00 cm
 CY 11.73 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

Current Data Parameters
 NAME adv-IV-017
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 2001130
 Time 14.20
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TO 6536
 SOLVENT CDCl3
 NS 505
 DS 4
 SWH 31446.541 Hz
 FIDRES 0.479836 Hz
 AQ 1.0420863 sec
 RG 3643.1
 DM 15.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec
 d12 0.00020000 sec

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

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 17.50 dB
 PL12 19.00 dB
 PL13 30.00 dB
 SF02 500.1325000 MHz

F1 - Acquisition parameters
 N00 2
 T0 256
 SF01 500.1325 MHz
 FIDRES 23.475060 Hz
 SW 12.016 ppm

F2 - Processing parameters
 SI 65536
 SF 125.7578038 MHz
 EQ 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 NUC2 1H
 OF 0
 SF 500.1300000 MHz
 N00 0
 SSB 0
 LB 0.30 Hz
 GB 0

10 NMR plot parameters
 CX 20.00 cm
 CY 9.41 cm
 FIP 220.000 ppm
 F1 27666.71 Hz
 F2 -10.000 ppm
 F2P -1257.58 Hz
 PPMCH 11.50000 ppm/cm
 HZCN 1446.21472 Hz/cm

SI-13

Current Data Parameters
NAME adw-III-042
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010608
Time 10.53
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
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 400.1300095 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

Integral

ppm

Current Data Parameters
NAME adw-III-042
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010608
Time 10.56
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 213
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 8192
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SF01 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
SF02 400.1316005 MHz

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
FIP 220.000 ppm
F1 22134.81 Hz
F2 -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

Current Data Parameters
 NAME adw-IV-021
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011206
 Time 14.03
 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
 SF01 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
 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 adw-IV-021
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20011206
Time 14.10
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 293
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 3649.1
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SF01 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
SF02 400.1316005 MHz

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 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

91-12

Current Data Parameters
 NAME adw-III-110
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010719
 Time 12.28
 INSTRUM drx400
 PROBD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 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
 SF01 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

F2 - Acquisition Parameters
 Date_ 20010719
 Time 12.28
 INSTRUM drx400
 PROBD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 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

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

Current Data Parameters
NAME adw-III-110
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010719
Time 12.31
INSTRUM dirx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 217
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
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

ID NMR plot parameters
F1 228.100 ppm
F2 22134.81 Hz
F2 10.000 ppm
PCMC 11.50608 ppm/cm
HZCM 1157.04700 Hz/cm

Current Data Parameters
NAME adw-III-279
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20011120
Time 16.51
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 8
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
SF01 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
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

4.51298
3.46244
3.44958
3.43964
3.42679
3.29761
3.27849
3.27490
3.25574
2.59448
2.58883
2.56999
2.56436
2.55427
2.54863
2.33698
2.33179
2.31974
2.31457
2.29679
2.29159
1.02017
1.00322

9.79122
9.78583
9.78045
7.39547
7.39245
7.38875
7.37702
7.37456
7.37152
7.36310
7.35692
7.34231
7.33568
7.32566
7.31396
7.30731
7.29035
7.28271

3.1769

1.0912
1.0872
1.0771

1.0810
1.0891

2.1023

5.3536

1.0000
Integral

Current Data Parameters
 NAME adw-III-279
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20011120
 Time 16.55
 INSTRUM drx400
 PROBD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 202
 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
 D1 0.05000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.30 usec
 PL1 2.00 dB
 SF01 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 100.6127791 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 1157.04700 Hz/cm

Current Data Parameters
 NAME adw-III-207
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010919
 Time 9.17
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 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
 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

Date		Time		Acquisition Parameters	
20010919	9.21				
INSTRUM	drx400				
PROBHD	5 mm Multinu				
PULPROG	zgpg30				
TD	65536				
SOLVENT	CDCl3				
NS	716				
DS	2				
SWH	23148.148 Hz				
FTRES	0.353213 Hz				
AQ	1.4156276 sec				
RG	8192				
DW	21.600 usec				
DE	4.50 usec				
TE	300.0 K				
D1	0.05000000 sec				
d11	0.03000000 sec				
d12	0.00020000 sec				

```
===== CHANNEL f2 =====
CPDRG2      waltz16
NUC2        1H
PCPD2       100.00 usec
PL2         0.00 dB
PL12        18.00 dB
PL13        18.00 dB
SF02       400.1316005 MHz
```

F2 - Processing parameters	
SI	32768
SF	100.6127749 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

Current Data Parameters
 NAME adw-IV-030
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011220
 Time 10.14
 INSTRUM drx400
 PROBD 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
 SF01 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300089 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	adw-IV-030
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date	20011220
Time	10.19
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	867
DS	2
SWH	23148.148 Hz
FIDRES	0.353213 Hz
AQ	1.4152676 sec
RG	4597.6
WDW	21.600 usec
DE	4.50 usec
TE	300.0 K
d1	0.0500000 sec
d11	0.0300000 sec
d12	0.0000200 sec

```
===== CHANNEL f1 =====
```

NUC1	13C
PI	12.30 usec
PL1	2.00 dB
SFO1	100.6237964 MHz

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

CPDPRG2	waitz16	1H
NUC2	100.00	usec
PCPD2	0.00	dB
PL2	18.00	dB
PL12	18.00	dB
PL13	18.00	dB
SFO2	400.1316005	MHz

F2 - Processing parameters

SI	32768	
SF	100.6127742	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

1D NMR plot parameters	
CX	20.00 cm
F1F1P	225.000 ppm
F1	22637.88 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	11.75000 ppm/cm
HZCM	1182.20020 Hz/cm

Current Data Parameters
 NAME adk-IV-051
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020325
 Time 12.41
 INSTRUM spect
 PROBO 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 6009.615 Hz
 FIDRES 0.183399 Hz
 AQ 2.7264309 sec
 RG 256
 DM 83.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.03000000 sec

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

F1 - Acquisition parameters
 NDO 2
 TD 256
 SF01 500.1325 MHz
 FIDRES 23.475060 Hz
 SW 12.016 ppm

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

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

1D NMR plot parameters
 CX 20.00 cm
 CY 11.74 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

Current Data Parameters
 NAME adw-IV-047
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020220
 Time 11:32
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6009.615 Hz
 FIDRES 0.183399 Hz
 AQ 2.7264309 sec
 RG 32
 DM 83.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.03000000 sec

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

F1 - Acquisition parameters
 NDO 2
 TO 256
 SF01 500.1325 MHz
 FIDRES 23.475060 Hz
 SW 12.016 ppm

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

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

10 NMR plot parameters
 CX 20.00 cm
 CY 9.28 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

```

Current Data Parameters
NAME          aov-1047
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
Date_         20020220
Time          11.39
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            745
DS            4
SWH            31446.541 Hz
FIDRES        0.479836 sec
AQ            1.0420883 sec
RG            13004
DM            15,900 USEC
DE            6.00 USEC
TE            300.0 K
O1            0.1000000 sec
O11           0.0300000 sec
O12           0.0002000 sec

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

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

F1 - Acquisition parameters
N0            2
TD            256
SF01          500.1325 MHz
FIDRES        23.475060 Hz
SN            12.016 ppm

F2 - Processing parameters
SI            65536
SF            125.7577985 MHz
WDW            EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

F1 - Processing parameters
SI            1024
MC2           OF
WDW            no
SSB           0
LB            0.30 Hz
GB            0

10 NMR plot parameters
CX            20.00 cm
CY            6.32 cm
F1P           28295.50 Hz
F2P           -10.000 ppm
F2            -1257.58 Hz
PCHNCH       171.75000 ppm/cm
HZCN         1477.65417 Hz/cm

```

Current Data Parameters
 NAME adw-IV-048
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20020221
 Time 11:31
 INSTRUM drx400
 PROBD 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 1.00000000 sec

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

NUC1 1H
 P1 7.70 usec
 PL1 -6.00 dB
 SF01 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
 FLIP 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

219.854 ppm

Current Data Parameters
NAME adw-IV-048
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020221
Time 11.36
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 461
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
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 100.6237964 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 100.6127771 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 225.000 ppm
F1 22637.88 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.75000 ppm/cm
HZCM 1182.20020 Hz/cm

Current Data Parameters
 NAME adw-III-198
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20010910
 Time 10.46
 INSTRUM drx400
 PROBD 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
 SF01 400.1320007 MHz

F2 - Processing parameters

SI 16384
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

ID 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

171.337

77.315
76.997
76.678
65.150
61.837
52.307
48.751
48.686
40.808
40.261
38.121
32.709
32.529
30.498
19.117
18.589
16.491

Current Data Parameters
NAME adw-III-198
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010910
Time 10.50
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 246
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 8192
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
SF01 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
SF02 400.1316005 MHz

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
FIP 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

Current Data Parameters
 NAME adw-III-202-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011204
 Time 14.33
 INSTRUM drx400
 PROBDH 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
 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
 SF01 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300003 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

14.7186
 15.6334
 17.5010
 27.0618
 28.3158
 30.1527
 31.3826
 35.3482
 37.5755
 41.1720
 44.2922
 47.2812
 53.1045
 61.2994
 64.7964
 67.3032

Current Data Parameters
 NAME adw-III-200-3
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20011205
 Time 15.55
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 4598
 DS 4
 SWH 31446.541 Hz
 FIDRES 0.479836 Hz
 AQ 1.0420883 sec
 RG 3649.1
 DW 15.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

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

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 17.50 dB
 PL12 19.00 dB
 PL13 30.00 dB
 SF02 500.1325000 MHz

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

1D NMR plot parameters
 CX 20.00 cm
 CY 4.84 cm
 F1P 220.000 ppm
 F1 27666.71 Hz
 F2P -10.000 ppm
 F2 -1257.58 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1446.21472 Hz/cm

ppm

72.124