**Domino Reactions That Combine an Azido-Schmidt Ring Expansion with the
Diels–Alder Reaction**

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Supporting Information

Experimental details

Copies of ^1H and ^{13}C spectra of listed compounds

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Experimental Details

General Information. The synthesis of the azido diene **1**¹ and azido enone **19**² have been described previously. Azido enones **10** and **11** were prepared via a Horner-Wadsworth-Emmons condensation of the known 3-azido propionaldehyde **A**³ and the corresponding β -keto phosphonates **B**⁴ and **C**.⁵ *Caution! All low molecular weight alkyl azides are potential explosion hazards and should be used with appropriate caution.*

(E)-6-Azido-3-hexen-2-one (10). To a solution of β -keto phosphonate **B**⁴ (1.60 g, 10 mmol) in THF/ H_2O (4:1, 40 mL) was added K_2CO_3 (2.10 g, 15 mmol). The resulting solution was cooled in an ice bath followed by dropwise addition of the 3-azido propionaldehyde **A** (1.0 g, 10 mmol) in THF (4 mL). The mixture was stirred in an ice bath for 1 h and then quenched with saturated aqueous $NaHCO_3$. The reaction mixture was partitioned between water and EtOAc. The organic layer was dried (Na_2SO_4), filtered, and concentrated to give an oil. Chromatography (10% EtOAc/hexane) afforded 940 mg (68%) of **10** as an oil. $R_f = 0.46$ (25% EtOAc/hexane); 1H NMR (400 MHz, $CDCl_3$) δ 2.25 (s, 3H), 2.51 (m, 2H), 3.44 (t, $J = 6.7$ Hz, 2H), 6.15 (d, $J = 16.0$ Hz, 1H), 6.74 (m, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 27.5, 32.2, 50.0, 133.6, 143.4, 198.5; IR (neat) 1709, 1644 cm^{-1} ; MS (CI) m/z 140 ($M^+ + 1$); HRMS calcd for $C_6H_{10}N_3O$ ($M^+ + H$): 140.0824, found 140.0853.

(E)-6-Azido-3-methyl-3-hexen-2-one (11). Azido enone **11** was prepared as described for **10** using β -keto phosphonate **C**⁵ (1.95 g, 10 mmol) to afford 310 mg (20%) of **11** as an oil. R_f = 0.47 (25% EtOAc/hexane); ¹H NMR (400 MHz, CDCl₃) δ 1.82 (s, 3H), 2.35 (s, 3H), 2.57 (m, 2H), 3.46 (t, J = 6.7 Hz, 2H), 6.60 (m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 11.8, 26.0, 29.2, 50.5, 138.4, 140.3, 199.9; IR (neat) 2097, 1668 cm⁻¹; MS (CI) m/z 154 (M^+ +1); HRMS calcd for C₇H₁₂N₃O (M^+ +H): 154.0980, found 154.0966.

(±)-1,2,5,6,6a,7,9a,9b-Octahydro-pyrrolo[3,2,1-*i,j*]quinolin-4-one (2). To a solution of diene **1**¹ (123 mg, 1 mmol) and 2-cyclopentenone (1 equiv) (200 mg, 1.4 mmol) in CH₂Cl₂ (50 mL) was added MeAlCl₂ (1.0 M in hexanes, 2.5 mL) at -78 °C. The resulting mixture was stirred at -78 °C for 2 h and then at room temperature for 12 h. After quenching with aqueous NaHCO₃, the mixture was partitioned between water and CHCl₃. The organic layer was collected, dried (Na₂SO₄), filtered and concentrated to give an oil. Chromatography (10-50% EtOAc/hexane followed by 1-2% MeOH/CHCl₃) afforded 68 mg (38%) of **2** as an oil. R_f = 0.45 (1:10 MeOH/EtOAc); ¹H NMR (400 MHz, CDCl₃) δ 1.70-2.05 (complex, 8H), 2.25 (m, 1H), 2.42 (m, 2H), 2.93 (br, s, 1H), 3.38 (m, 1H), 3.56 (m, 1H), 3.75 (m, 1H), 5.43 (dd, J = 1.2, 10.1 Hz, 1H), 5.72 (m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 23.0, 26.1, 27.7, 28.5, 28.9, 39.4, 44.2, 59.4, 127.1, 127.4, 169.7; IR (neat) 1621 cm⁻¹; MS (CI) m/z 178 (M^+ +1); HRMS calcd for C₁₁H₁₆NO (M^+ +H): 178.1232, found 178.1224.

(*E*)-6-Azido-2-trimethylsilyloxy-1,3-hexadiene (3). To a solution of enone **10** (1.4 g, 10 mmol) in anhydrous ethyl ether (35 mL) under argon was added Et₃N (2.1 g, 20 mmol) at 0 °C followed by the addition of TMSOTf (3.3 g, 15 mmol). The resulting mixture was stirred at 0 °C for 30 min. The organic layer was washed with brine, dried (Na₂SO₄), and evaporated under reduced pressure to give 2.18 g (98%) of **3** as an oil. This diene was used directly for the next step. *R*_f = 0.52 (2:98 EtOAc/hexane); ¹H NMR (400 MHz, CDCl₃) δ 0.26 (s, 9H), 2.42 (m, 2H), 3.36 (t, *J* = 7.0 Hz, 2H), 4.31 (d, *J* = 3.5 Hz, 2H), 5.92 (m, 1H), 6.01 (d, *J* = 15.2 Hz, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ -0.1, 31.5, 50.7, 95.3, 126.4, 130.5, 154.3; IR (neat) 2098 cm⁻¹; MS (CI) *m/z* 212 (M⁺+1); HRMS calcd for C₉H₁₈N₃OSi (M⁺+H): 212.1219, found 212.1196.

General procedure for the intermolecular Diels–Alder/intramolecular Schmidt reaction involving diene 3. To a cooled (-78 °C) solution of enone (1.0–1.5 mmol) in CH₂Cl₂ (15–20 mL) under argon was added SnCl₄ (1 equiv) followed by the addition of diene (1.8 equiv). The resulting mixture was stirred at -78 °C for 2 h. After slow addition of another portion of SnCl₄ (1.5 equiv), the mixture was stirred at room temperature for 12 h, and then quenched with aqueous NaHCO₃. The mixture was partitioned between water and CHCl₃. The organic layer was collected, dried (Na₂SO₄), filtered, and concentrated to give an oil. Chromatography (10-50% EtOAc/hexane followed by 1-2% MeOH/CHCl₃) afforded the desired lactams.

(±)-1,2,5,6,6a,7,9a,9b-Octahydro-pyrrolo[3,2,1-*i,j*]quinolin-4,8-dione (4). 2-

Cyclopentenone (90 mg, 1.1 mmol) afforded 110 mg (52%) of **4** as an oil. R_f = 0.28 (1:10 MeOH/EtOAc); ^1H NMR (400 MHz, CDCl_3) δ 1.70-1.82 (complex, 2H), 2.01-2.18 (complex, 4H), 2.28-2.73 (complex, 5H), 3.62 (t, J = 10.2 Hz, 1H), 3.73 (t, J = 4.8 Hz, 1H), 3.80 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 25.8, 27.2, 28.5, 33.6, 39.2, 40.7, 41.0, 43.1, 59.4, 169.0, 210.6; IR (neat) 1714, 1614 cm^{-1} ; MS (CI) m/z 194 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{11}\text{H}_{16}\text{NO}_2$ ($\text{M}^+ + \text{H}$): 194.1181, found 194.1181.

(±)-5-Octahydro-azipino[3,2,1-*h,i*]indol-4,9-dione (5). 2-Cyclohexenone (100

mg, 1.0 mmol) afforded 178 mg (82%) of **5**, an oil, as a ca. 3.4: 1 (**5a** : **5b**) mixture of diastereomers. The mixture solidified upon standing. R_f = 0.32 (1:10 MeOH/EtOAc); IR (neat) 1709, 1624 cm^{-1} ; MS (CI) m/z 208 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{12}\text{H}_{18}\text{NO}_2$ ($\text{M}^+ + \text{H}$): 208.1338, found 208.1357. A pure sample of the major diastereomer was obtained after recrystallization from $\text{CH}_3\text{Cl}/\text{EtOAc}$. Mp 131-133 °C; ^1H NMR (500 MHz, CDCl_3) δ 1.54-1.84 (complex, 6H), 2.00 (m, 1H), 2.11 (d, J = 15.7 Hz, 1H), 2.24 (dd, J = 4.0, 15.9 Hz, 1H), 2.46 (m, 3H), 2.65 (m, 2H), 2.99 (m, 1H), 3.24 (two sets of t, J = 6.56, 11.9 Hz, 1H), 3.91 (m, 1H), 4.13 (d, 1H, J = 6.9 Hz); ^{13}C NMR (125.8 MHz, CDCl_3) δ 18.5, 31.2, 33.6, 35.7, 38.0, 39.3, 39.7, 43.3, 45.8, 59.7, 174.3, 211.2. Minor diastereomer

(diagnostic peaks only, from the mixture): ^1H NMR (400 MHz, CDCl_3) δ 3.51 (m), 3.75 (m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 22.1, 26.6, 28.4, 31.9, 39.7, 43.2, 45.6, 47.3, 62.6, 171.6, 211.5.

(±)-1-Acetyl-*cis*(3a, 7a)-octahydro-indol-5-one (6). Methyl vinyl ketone (100 mg, 1.43 mmol) afforded 155 mg (60%) of **6**, an oil, as a ca. 2.6:1 mixture of rotamers. R_f = 0.30 (1:10 MeOH/EtOAc); IR (neat) 1708, 1623 cm^{-1} ; MS (CI) m/z 182 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{10}\text{H}_{16}\text{NO}_2$ ($\text{M}^+ + \text{H}$): 182.1181, found 182.1199. Major rotamer: ^1H NMR (500 MHz, CDCl_3) δ 1.76 (m, 3H), 2.06 (s, 3H), 2.13 (m, 1H), 2.30-2.51 (complex, 4H), 2.61-2.80 (m, 2H), 3.46-3.60 (m, 2H), 4.37 (m, 1H); ^{13}C NMR (125.8 MHz, CDCl_3) δ 22.5, 25.5, 30.7, 36.9, 37.0, 41.6, 46.6, 54.7, 169.4, 210.9. Minor rotamer (diagnostic peaks only): ^1H NMR (500 MHz, CDCl_3) δ 2.15 (s), 2.25 (m), 2.80 (m), 4.12 (m); ^{13}C NMR (125.8 MHz, CDCl_3) δ 21.9, 27.5, 28.7, 37.2, 39.1, 41.4, 44.6, 56.3, 168.7, 209.4.

(±)-1-Acetyl-7-methyl-*cis*(3a, 7a)-octahydro-indol-5-one (7). 3-Penten-2-one (100 mg, 1.2 mmol) afforded 137 mg (59%) of **7**, an oil, as a ca. 2.4:1 mixture of rotamers. R_f = 0.34 (1:10 MeOH/EtOAc). IR (neat) 1712, 1627 cm^{-1} ; MS (CI) m/z 196 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{11}\text{H}_{18}\text{NO}_2$ ($\text{M}^+ + \text{H}$): 196.1338, found 196.1338. Major

rotamer: ^1H NMR (500 MHz, CDCl_3) δ 1.09 (d, $J = 6.7$ Hz, 3H), 1.76 (m, 2H), 1.86 (m, 1H), 2.08 (s, 3H), 2.05-2.41 (complex, 3H), 2.68 (m, 2H), 3.56 (m, 2H), 4.30 (m, 1H); ^{13}C NMR (125.8 MHz, CDCl_3) δ 19.9, 22.4, 30.0, 34.4, 38.1, 41.4, 46.0, 46.2, 60.0, 170.2, 210.0. Minor rotamer (diagnostic peaks only): ^1H NMR (500 MHz, CDCl_3) δ 1.12 (d, $J = 6.6$ Hz), 2.17 (s), 3.40 (m), 3.79 (m); ^{13}C NMR (125.8 MHz, CDCl_3) δ 19.7, 22.5, 28.1, 34.0, 38.1, 39.7, 41.5, 43.7, 46.0, 63.5.

(±)-cis(4a, 8a)-Hexahydroisoquinolin-1,6-dione (8) and **(±)-1-Formyl-cis(3a, 7a)-octahydro-indol-5-one (9)**. Acrolein (90%, 125 mg, 2.0 mmol) was reacted using MeAlCl_2 as Lewis acid in place of SnCl_4 to afford 120 mg (36%) of **8**, as an oil. $R_f = 0.21$ (1:10 MeOH/EtOAc); ^1H NMR (400 MHz, CDCl_3) δ 1.76 (m, 2H), 2.06 (m, 2H), 2.34-2.58 (complex, 5H), 2.74 (m, 1H), 3.42 (m, 2H), 6.19 (s br, 1H, D_2O exchangeable); ^{13}C NMR (100.6 MHz, CDCl_3) δ 25.9, 26.8, 35.3, 39.5, 40.2, 41.0, 44.1, 173.3, 210.9; IR (neat) 1708, 1649 cm^{-1} ; MS (FAB^+) m/z 168 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_9\text{H}_{14}\text{NO}_2$ ($\text{M}^+ + \text{H}$): 168.1025, found 168.1000.

The same reaction also afforded 70 mg (21%) of **9**, an oil, as a ca. 1.3:1 mixture of rotamers. $R_f = 0.25$ (1:10 MeOH/EtOAc). IR (neat) 1712, 1658 cm^{-1} ; MS (FAB^+) m/z 168 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_9\text{H}_{14}\text{NO}_2$ ($\text{M}^+ + \text{H}$): 168.1025, found 168.0983. Major rotamer: ^1H NMR (400 MHz, CDCl_3) δ 1.76 (m, 2H), 2.13 (m, 2H), 2.37 (complex, 4H),

2.63 (m, 2H), 3.60 (m, 2H), 4.12 (m, 1H), 8.28 (s, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 25.4, 28.9, 36.8, 38.5, 41.9, 43.0, 55.4, 161.0, 209.9. Minor rotamer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 1.90 (m), 2.21 (m), 4.33 (m), 8.35 (s); ^{13}C NMR (100.6 MHz, CDCl_3) δ 29.5, 30.8, 36.6, 37.0, 42.1, 45.4, 53.6, 161.5.

(±)-1-Acetyl-*trans*(3a, 7a)-2,3,3a,4,7,7a-hexahydro-1H-indole (12). Butadiene was bubbled through a solution of enone **10** (200 mg, 1.4 mmol) in CH_2Cl_2 (50 mL) at 0 °C for 5 min followed by dropwise addition of MeAlCl_2 (1.0 M in hexanes, 4.3 mL). The resulting mixture was stirred at 0 °C for 2 h and then at room temperature for 26 h. After dilution with CH_2Cl_2 (100 mL), aqueous NaHCO_3 was added. The organic layer was separated, washed with brine, and dried (MgSO_4). The organic layer was filtered, and concentrated to give 128 mg (54%) of **12**, a yellow oil, as a ca. 2.5:1 mixture of rotamers. R_f = 0.5 (EtOAc); IR (neat) 1418, 1443, 1633 cm^{-1} ; MS (FAB $^+$) m/z 166 ($\text{M}^+ + 1$); HRMS calcd. for $\text{C}_{10}\text{H}_{16}\text{NO}$ ($\text{M}^+ + \text{H}$) 166.1232, found 166.1221. Major rotamer: ^1H NMR (400 MHz, CDCl_3) δ 1.46-1.49 (m, 2H), 1.89-1.95 (m, 1H), 1.98 (s, 3H), 2.27-2.28 (m, 2H), 3.14-3.18 (m, 2H), 3.51 (t, J = 9.4, 1H), 5.60 (s, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 23.7, 30.5, 31.0, 33.0, 42.4, 48.9, 60.6, 126.5, 126.9, 170.7. Minor rotamer: ^1H NMR (400 MHz, CDCl_3) (diagnostic peaks only) δ 2.01 (s), 3.36-3.43 (m), 3.83-3.88 (dd, J = 8.2 and 11.8 Hz); ^{13}C NMR (100.6 MHz, CDCl_3) δ 23.4, 29.2, 34.2, 44.1, 47.8, 125.3, 127.9, 170.8.

General Procedure for the Diels–Alder/Schmidt reaction using enones **10, **11****

or **19**. To a solution of azido enone (**10**, **11** or **19**; 0.4–2.1 mmol) and diene (3 equiv) in CH₂Cl₂ (15–20 mL) at 0 °C was added AlMeCl₂ (3 equiv). The resulting mixture was stirred at 0 °C for 2 h and then at room temperature for 12 h, and then quenched with aqueous NaHCO₃. The mixture was partitioned between water and CHCl₃. The organic layer was collected, dried (Na₂SO₄), filtered, and concentrated to give an oil. Chromatography (10% EtOAc/hexane followed by 1–2% MeOH/CHCl₃) afforded the desired lactams.

(±)-1-Acetyl-5-methyl-*trans*(3*a*,7*a*)-2,3,3*a*,4,7,7*a*-hexahydro-1*H*-indole (13**).**

Enone **10** (292 mg, 2.1 mmol) afforded 275 mg (73%) of **13**, a yellow oil, as a ca. 2.2:1 mixture of rotamers. R_f = 0.25 (EtOAc); IR (neat) 1417, 1650 cm⁻¹; MS (CI) m/z 180 (M^+ +1); HRMS calcd for C₁₁H₁₇NO (M^+ +H) 180.1388, found 180.1385. Major rotamer: ¹H NMR (400 MHz, CDCl₃) δ 1.62 (s, 3H), 1.73–1.95 (m, 5H), 2.06–2.11 (m, 2H), 3.42 (dt, J = 6.21, 17.1 Hz, 2H), 3.52 (t, J = 9.6 Hz, 1H), 5.29 (s, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 23.6, 23.7, 30.4, 32.5, 35.7, 43.0, 49.2, 60.8, 121.0, 133.9, 170.7. Minor rotamer (diagnostic peaks only): ¹H NMR (400 MHz, CDCl₃) δ 1.65 (s), 2.00 (s), 3.24 (dt, J = 5.7, 17.7 Hz), 3.86 (dd, J = 8.2, 11.8 Hz); ¹³C NMR (100.6 MHz, CDCl₃) δ 23.3, 23.6, 29.2, 33.8, 35.8, 44.6, 48.1, 60.6, 119.4, 135.5, 170.8.

(±)-1-Acetyl-5,6-dimethyl-*trans*(3*a*, 7*a*)-2,3,3*a*,4,7,7*a*-hexahydro-1*H*-indole

(14). Enone **10** (200 mg, 1.4 mmol) afforded 208 mg (75%) of **14**, a yellow oil, as a ca. 2.4:1 mixture of rotamers. $R_f = 0.28$ (EtOAc); IR (neat) 1444, 1650 cm^{-1} ; MS (FAB⁺) m/z 194 ($M^+ + 1$); HRMS Calcd for $\text{C}_{12}\text{H}_{20}\text{NO}$ ($M^+ + \text{H}$) 194.1545, found 194.1551. Major rotamer: ^1H NMR (400 MHz, CDCl_3) δ 1.58 (s, 6H), 1.85-1.97 (m, 5H), 1.99 (s, 3H), 2.07-2.11 (m, 2H), 3.42 (dt, $J = 6.3, 17.4$ Hz, 1H), 3.52 (t, $J = 9.9$ Hz, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.4, 19.7, 23.7, 29.7, 36.9, 38.8, 43.1, 49.2, 61.2, 125.3, 125.8, 170.7. Minor rotamer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 1.61 (s), 2.03 (s); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.9, 23.4, 29.2, 37.0, 40.1, 44.7, 48.1, 61.0, 124.2, 126.9, 170.8.

(±)-1-Acetyl-*trans*(3*a*, 7*a*)-octahydro-1*H*-indol-5-one (15). Enone **10** (275 mg, 2.0 mmol) afforded 220 mg (71%) of **15**, a yellow oil, as a ca. 1.4:1 mixture of rotamers. $R_f = 0.18$ (EtOAc); IR (neat) 1647, 1712 cm^{-1} ; MS (CI) m/z 182 ($M^+ + \text{H}$); HRMS calcd for $\text{C}_{10}\text{H}_{16}\text{NO}_2$ ($M^+ + \text{H}$) 182.1181, found 182.1161. Major rotamer: ^1H NMR (400 MHz, CDCl_3) δ 1.31-1.45 (m, 2H), 1.91 (s, 3H), 2.19-2.34 (m, 3H), 2.46 (m, 1H), 3.28-3.45 (m, 1H), 3.39-3.46 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 23.6, 28.9, 30.2, 39.4, 45.3,

45.5, 49.3, 62.3, 171.3, 209.4. Minor rotamer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 1.49-1.57 (m), 1.89 (s), 2.50 (m), 3.00-3.06 (m), 3.51-3.56 (m); ^{13}C NMR (400 MHz, CDCl_3) δ 23.3, 29.2, 30.4, 39.0, 45.2, 47.1, 48.83, 62.0, 170.9, 208.1.

(±)-1-Acetyl-7a-methyl-*trans*(3a, 7a)-octahydro-1H-indol-5-one (16). Enone **11** (110 mg, 0.7 mmol) afforded 85 mg (61%) of **16** isolated as an oil. TLC (10:1 $\text{CHCl}_3/\text{MeOH}$): R_f = 0.50. ^1H NMR (400 MHz, CDCl_3) δ 1.33 (s, 3H), 1.82 (m, 2H), 1.92 (m, 1H), 2.02 (s, 3H), 2.16-2.53 (complex, 5H), 3.13 (m, 1H), 3.58 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 14.8, 24.1, 26.8, 35.2, 38.1, 41.8, 47.2, 48.4, 62.1, 170.1, 210.2; IR (neat) 1709, 1644 cm^{-1} ; MS (CI) m/z 196 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{11}\text{H}_{18}\text{NO}_2$ ($\text{M}^+ + \text{H}$): 196.1338, found 196.1332.

(±)-6-Acetyl-5-(2-azidoethyl)-bicyclo[2.2.2]oct-2-ene (17). Enone **10** (200 mg, 1.4 mmol) afforded 247 mg (79%) of **17** as a dark yellow oil. R_f = 0.64 (1:4 EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 1.56-1.70 (m, 8H), 2.17 (s, 3H), 2.42-2.45 (m, 1H), 2.86-2.88 (m, 1H), 3.20-3.33 (m, 2H), 6.06 (t, J = 7.0 Hz, 1H), 6.41 (t, J = 7.1 Hz, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 18.9, 26.6, 28.8, 33.6, 33.9, 34.1, 36.1, 50.7, 59.6,

130.6, 137.2, 209.6; IR (neat) 1708, 2096 cm^{-1} . MS (CI) m/z 220 ($\text{M}^+ + \text{H}$); HRMS calcd for $\text{C}_{12}\text{H}_{18}\text{N}_3\text{O}$ ($\text{M}^+ + \text{H}$) 220.1450, found 220.1453.

(±)-6-Methyl-6-acetyl-5-(2-azidoethyl)-bicyclo[2.2.2]oct-2-ene (18). Enone **11** (110 mg, 0.72 mmol) afforded 120 mg (72%) of **18** as a dark yellow oil. R_f = 0.67 (1:4 EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 1.04-1.19 (m, 2H), 1.14 (s, 3H), 1.59-1.82 (m, 5H), 2.13 (s, 3H), 2.20 (m, 1H), 2.40 (s br, 1H), 2.66 (s br, 1H), 3.33 (m, 2H), 6.25 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 17.9, 19.8, 20.6, 25.4, 28.4, 33.5, 37.1, 38.8, 51.1, 53.1, 133.7, 136.2, 212.9; IR (neat) 1702, 2094 cm^{-1} . MS (CI) m/z 234 ($\text{M}^+ + \text{H}$); HRMS calcd for $\text{C}_{13}\text{H}_{20}\text{N}_3\text{O}$ ($\text{M}^+ + \text{H}$) 234.1606, found 234.1594.

(±)-12-Methyl-5-aza-tricyclo[8.4.0.0^{1,5}]tetradec-12-en-6-one (20). Enone **19**² (180 mg, 1.0 mmol) afforded 185 mg (84%) of **20** as an oil. R_f = 0.47 (10:1 $\text{CHCl}_3/\text{MeOH}$); ^1H NMR (400 MHz, CDCl_3) δ 1.51-1.79 (complex, 12H), 1.96 (m, 1H), 2.16 (m, 1H), 2.47 (m, 3H), 2.61 (m, 1H), 3.36 (m, 1H), 3.90 (m, 1H), 5.25 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 20.7, 23.8 (two peaks), 32.0, 34.1, 39.2, 39.4, 40.8, 43.5,

49.6, 64.6, 119.1, 132.4, 174.2; IR (neat) 1626 cm^{-1} ; MS (CI) m/z 220 ($M^+ + 1$); HRMS calcd for $\text{C}_{14}\text{H}_{22}\text{NO}$ ($M^+ + \text{H}$): 220.1701, found 220.1701.

(±)-12, 13-Dimethyl-5-aza-tricyclo[8.4.0.0^{1,5}]tetradec-12-en-6-one (21).

Enone **19** (180 mg, 1.0 mmol) afforded 198 mg (85%) of **21** as an oil. $R_f = 0.48$ (10:1 $\text{CHCl}_3/\text{MeOH}$); ^1H NMR (400 MHz, CDCl_3) δ 1.51-1.90 (complex, 16H), 2.12 (m, 1H), 2.47 (m, 3H), 2.63 (dd, $J = 8.3, 14.6$ Hz, 1H), 3.38 (m, 1H), 3.90 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.2, 19.4, 20.7, 23.8, 32.0, 34.1, 38.1, 39.2, 40.9, 41.0, 43.8, 49.6, 65.1, 123.4, 123.8, 174.3; IR (neat) 1624 cm^{-1} ; MS (CI) m/z 234 ($M^+ + 1$); HRMS calcd for $\text{C}_{15}\text{H}_{24}\text{NO}$ ($M^+ + \text{H}$): 234.1858, found 234.1850.

(±)-5-Aza-tricyclo[8.4.0.0^{1,5}]tetradecane-6,12-dione (22). Enone **19** (70 mg, 0.4 mmol) afforded 80 mg (91%) of **22** as an oil which solidified upon standing. The structure **22** was determined by X-ray crystallography after recrystallization from MeOH/EtOAc . Mp 136-138 $^{\circ}\text{C}$; $R_f = 0.41$ (10:1 $\text{CHCl}_3/\text{MeOH}$); ^1H NMR (400 MHz, CDCl_3) δ 1.54-1.64 (m, 2H), 1.80-2.06 (complex, 7H), 2.25 (m, 1H), 2.421-2.52 (m, 4H), 2.64 (m, 2H), 2.76 (dd, $J = 5.1, 13.2$ Hz, 1H), 3.49 (m, 1H), 3.93 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 20.9, 22.3, 28.4, 33.9, 38.4, 38.6, 40.3, 45.8, 48.2, 49.6, 64.9,

173.9, 209.2; IR (neat) 1705, 1613 cm^{-1} ; MS (CI) m/z 222 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{13}\text{H}_{20}\text{NO}_2$ ($\text{M}^+ + \text{H}$): 222.1494, found 222.1484.

References

- (1) Grieco, P. A.; Galatsis, P.; Spohn, R. F. *Tetrahedron* **1986**, *42*, 2847–2853.
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- (3) Davies, A. J.; Donald, A. S. R.; Marks, R. E. *J. Chem. Soc. Sect. C* **1967**, 2109–2112.
- (4) Corbel, B.; Medinger, L.; Haelters, J. P.; Sturtz, G. *Synthesis* **1985**, 1048–1051.
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SI-15

Curr
NAME
EXPNO
PROCNO

2

F2 - Acquisition Parameters
Date_ 20031022
Time 17.30
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 181
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
FPMCH 0.59846 ppm/cm
HZCM 239.46359 Hz/cm

Current Data Parameters
NAME III-38-3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031022
Time 17.35
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 449
DS 2
SWH 23148.148 Hz
FIDRES 0.351213 Hz
AQ 1.4156276 sec
RG 32768
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

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

F2 - Processing parameters
SI 32768
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
FIP 215.000 ppm
F1 21631.74 Hz
F2P -503.06 Hz
F2 -503.06 Hz
FPMCH 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

SI-16

Date: 20040723
Time: 12:25
INSTRUM: spect
PROBHD: 5 mm BBO BB-1H
PULPROG: zg30
TD: 32768
SOLVENT: CDCl₃
NS: 16
DS: 2
SWH: 6009.615 Hz
FIDRES: 0.18399 Hz
AQ: 2.7264305 sec
RG: 114
DW: 83.200 usec
DE: 6.00 usec
TE: 297.3 K
D1: 0.03000000 sec
HOREST: 0.00000000 sec
HCHRG: 0.01500000 sec

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

F1 - Acquisition parameters
AQ: 2
TD: 256
SFO1: 500.1320 MHz
FIDRES: 23.475050 Hz
SW: 12.018 ppm
FMODE: undefined

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

F1 - Processing parameters
SI: 1024
MC2: OF
SF: 500.1300000 MHz
WDW: 051MC
SSB: 0
LB: 0.30 Hz
GB: 0

1D NMR plot parameters
CX: 20.00 cm
CY: 0.00 cm
F1P: 10.000 ppm
F1: 5005.33 Hz
F2P: -2.000 ppm
F2: -1004.25 Hz
PPHCK: 0.60081 ppm/cm
HZCK: 300.48077 Hz/cm

Current Data Parameters
NAME: 1H-005-2
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20040723
Time: 12:21
INSTRUM: spect
PROBHD: 5 mm BBO BB-1H
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl₃
NS: 325
DS: 4
SWH: 31449.541 Hz
FIDRES: 0.479036 Hz
AQ: 1.8400003 sec
RG: 256
DW: 15.900 usec
DE: 6.00 usec
TE: 298.3 K
D1: 0.15000001 sec
d11: 0.05000000 sec
DELTA: 0.05000000 sec
HOREST: 0.00000000 sec
HCHRG: 0.01500000 sec

***** CHANNEL f1 *****
NUC1: 13C
P1: 6.50 usec
PL1: 5.00 dB
SFO1: 125.767500 MHz

***** CHANNEL f2 *****
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 95.00 usec
PL2: -4.00 dB
PL12: 19.00 dB
PL13: 30.00 dB
SFO2: 500.132000 MHz

F1 - Acquisition parameters
AQ: 2
TD: 256
SFO1: 500.1320 MHz
FIDRES: 23.475050 Hz
SW: 12.018 ppm
FMODE: undefined

F2 - Processing parameters
SI: 65536
SF: 125.7578000 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

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

1D NMR plot parameters
CX: 20.00 cm
CY: 0.00 cm
F1P: 219.234 ppm
F1: 27658.41 Hz
F2P: -30.100 ppm
F2: -1786.13 Hz
PPHCK: 12.50282 ppm/cm
HZCK: 1572.32703 Hz/cm

SI-17

F2 - Acquisition Parameters
 Date_ 20040719
 Time 17.15
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 128
 DW 104.400 usec
 DE 5.50 usec
 TE 294.2 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

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

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

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 239.46362 Hz/cm

Current Data Parameters
 NAME IV-46
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040719
 Time 17.20
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 247
 DS 4
 SMH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.356452 sec
 RG 32768
 DW 20.700 usec
 DE 5.50 usec
 TE 294.2 K
 D1 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 12.30 usec
 PL1 2.00 dB
 SFO1 100.6248025 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 65536
 SP 100.6127290 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 240.037 ppm
 F1 24150.82 Hz
 F2P -0.037 ppm
 F2 -3.77 Hz
 PPMCM 12.00374 ppm/cm
 HZCM 1207.72949 Hz/cm

SI-18

5a

F2 - Acquisition Parameters
 Date_ 20040726
 Time 15.16
 INSTRUM spect
 PROBO 5 mm BBO BB-1H
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6009.615 Hz
 FIDRES 0.183399 Hz
 AQ 2.7264309 sec
 RG 322.5
 DM 83.200 usec
 DE 6.00 usec
 TE 297.4 K
 D1 0.03000000 sec
 MCREST 0.00000000 sec
 MCNWK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.70 usec
 PL1 -4.00 dB
 SF01 500.132000 MHz

F1 - Acquisition Parameters
 MD 2
 TO 256
 SF01 500.1325 MHz
 FIDRES 23.475000 Hz
 SW 12.016 ppm
 FWHDE undefined

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

F1 - Processing parameters
 SI 1024
 NUC2 OF
 SF 500.1300000 MHz
 MDW OSINE
 SSB 0
 LB 0.30 Hz
 GB 0

1D NMR plot parameters
 CX 20.00 cm
 CY 0.00 cm
 FIP 10.008 ppm
 FI 5005.33 Hz
 FIP -2.008 ppm
 F2 -1004.20 Hz
 PRNCK 0.65081 ppm/cm
 HZCK 300.48077 Hz/cm

Current Data Parameters
 NAME IV-45-2b
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040726
 Time 15.19
 INSTRUM spect
 PROBO 5 mm BBO BB-1H
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 226
 DS 4
 SWH 31445.541 Hz
 FIDRES 0.479036 Hz
 AQ 1.0420003 sec
 RG 256
 DM 15.900 usec
 DE 6.00 usec
 TE 297.4 K
 D1 0.15000001 sec
 D11 0.03000000 sec
 DELTA 0.05000000 sec
 MCREST 0.00000000 sec
 MCNWK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 6.50 usec
 PL1 5.00 dB
 SF01 125.772000 MHz

***** CHANNEL f2 *****
 CHNPG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 -4.00 dB
 PL12 10.00 dB
 PL13 30.00 dB
 SF02 500.1325000 MHz

F1 - Acquisition parameters
 MD 2
 TO 256
 SF01 500.1325 MHz
 FIDRES 23.475000 Hz
 SW 12.016 ppm
 FWHDE undefined

F2 - Processing parameters
 SI 65536
 SF 125.7577361 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

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

1D NMR plot parameters
 CX 20.00 cm
 CY 0.00 cm
 FIP 212.517 ppm
 FI 30490.41 Hz
 FIP -7.539 ppm
 F2 -548.11 Hz
 PRNCK 12.50202 ppm/cm
 HZCK 1572.32617 Hz/cm

SI-19

F2 - Acquisition Parameters

Date_ 20040717

Time 15.45

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 114

DM 104.400 usec

DE 5.50 usec

TE 294.2 K

D1 1.00000000 sec

MCREST 0.00000000 sec

MCWRR 0.01500000 sec

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

NUC1 1H

P1 7.70 usec

PL1 -6.00 dB

SFO1 400.1320007 MHz

F2 - Processing parameters

SI 32768

SF 400.1300000 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

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

PPMCH 0.59846 ppm/cm

HZCM 239.46362 Hz/cm

Current Data Parameters

NAME IV-42-4

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20040717

Time 15.52

INSTRUM drx400

PROBHD 5 mm Multinucl

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 551

DS 4

SWH 24154.590 Hz

FIDRES 0.348570 Hz

AQ 1.3586452 sec

RG 32768

DM 20.700 usec

DE 5.50 usec

TE 294.2 K

D1 0.15000001 sec

d11 0.03000000 sec

DELTA 0.05000000 sec

MCREST 0.00000000 sec

MCWRR 0.01500000 sec

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

NUC1 13C

P1 12.10 usec

PL1 2.00 dB

SFO1 100.6248025 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 65536

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 240.037 ppm

F1 24150.82 Hz

F2P -0.037 ppm

F2 -3.77 Hz

PPMCH 12.00374 ppm/cm

HZCM 1207.72949 Hz/cm

SI-20

Date_ 20040722
Time 16.09
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.515 Hz
FIDRES 0.183399 Hz
AQ 2.7254309 sec
RG 203.2
DN 83.200 usec
DE 5.00 usec
TE 298.2 K
O1 0.03000000 sec
MCRES1 0.00000000 sec
MCNMR 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.138005 MHz

F1 - Acquisition parameters
NUC 2
TD 256
SFO1 500.1325 MHz
FIDRES 23.475060 Hz
SW 12.016 ppm
PRMODE undefined

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

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

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 10.008 ppm
F1 5005.33 Hz
F2P -2.008 ppm
F2 -1004.29 Hz
PPHCH 0.60081 ppm/cm
H2CH 300.48977 Hz/cm

Current Data Parameters
NAME 1H-13C-20
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040722
Time 16.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 320
DS 4
SWH 31446.543 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 256
DN 15.800 usec
DE 6.00 usec
TE 298.2 K
O1 0.15000001 sec
O11 0.03000000 sec
DELTA 0.05000000 sec
MCRES1 0.00000000 sec
MCNMR 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.7735687 MHz

***** CHANNEL f2 *****
CPROG2 waltz16
NUC2 1H
PCPD2 25.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 36.00 dB
SFO2 500.1325000 MHz

F1 - Acquisition parameters
NUC 2
TD 256
SFO1 500.1325 MHz
FIDRES 23.475060 Hz
SW 12.016 ppm
PRMODE undefined

F2 - Processing parameters
S1 65536
SF 125.7735688 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 242.517 ppm
F1 30498.43 Hz
F2P -7.533 ppm
F2 -948.11 Hz
PPHCH 12.50282 ppm/cm
H2CH 1572.3789 Hz/cm

SI-21

Date_ 20040722
 Time 16.24
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5009.615 Hz
 FIDRES 0.183359 Hz
 AQ 2.7264309 sec
 RG 256
 DM 83.208 usec
 DE 6.00 usec
 TE 299.0 K
 D1 0.03000000 sec
 HCREST 0.00000000 sec
 HCNMR 0.01500000 sec

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

F1 - Acquisition parameters
 MD 2
 TO 256
 SFO1 500.1325 MHz
 FIDRES 23.475660 Hz
 SW 12.016 GHz
 FWHDE undefined

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

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

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

Current Data Parameters
 NAME 17-45-2
 EXPNO 2
 PROCNO 1

F2 - Acquisition parameters
 Date_ 20040722
 Time 16.30
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 484
 DS 4
 SWH 31446.541 Hz
 FIDRES 0.478836 Hz
 AQ 1.040083 sec
 RG 256
 DM 15.900 usec
 DE 6.00 usec
 TE 299.0 K
 D1 0.15000001 sec
 D11 0.03000000 sec
 DELTA 0.05000000 sec
 HCREST 0.00000000 sec
 HCNMR 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 6.50 usec
 PL1 5.00 dB
 SFO1 125.772500 MHz

***** CHANNEL f2 *****
 NUC2 1H
 P2 95.00 usec
 PL2 -4.00 dB
 PL12 19.00 dB
 PL13 20.00 dB
 SFO2 500.132500 MHz

F1 - Acquisition parameters
 MD 2
 TO 256
 SFO1 500.1325 MHz
 FIDRES 23.475660 Hz
 SW 12.016 GHz
 FWHDE undefined

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

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

1D NMR plot parameters
 CX 20.00 cm
 CY 0.00 cm
 FIP 242.517 ppm
 F1 30498.43 Hz
 F2P -7.533 ppm
 F2 -948.11 Hz
 PPMCH 12.50282 ppm/cm
 HZCH 1572.32703 Hz/cm

SI-22

F2 - Acquisition Parameters
Date_ 20040911
Time 11.42
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 143.7
OW 104.400 usec
DE 5.50 usec
TE 293.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

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

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 239.46362 Hz/cm

Current Data Parameters
NAME Iv-105-2b
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040911
Time 11.46
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 205
DS 2
SWH 24154.590 Hz
FIDRES 0.160570 Hz
AQ 1.3566452 sec
RG 32768
DM 20.700 usec
DE 5.50 usec
TE 293.2 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 12.10 usec
PL1 2.00 dB
SFO1 100.6248025 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 65536
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-23

F2 - Acquisition Parameters
 Date_ 20040918
 Time 11.15
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 181
 DM 104.400 usec
 DE 5.50 usec
 TE 297.2 K
 DI 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

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

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

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 239.46362 Hz/cm

Current Data Parameters
 NAME Iv-115-1b
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040918
 Time 15.22
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SMH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.3586452 sec
 RG 32768
 DM 20.700 usec
 DE 5.50 usec
 TE 297.2 K
 DI 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 12.10 usec
 PL1 2.00 dB
 SFO1 100.6248025 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 65536
 SF 100.6127280 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 240.037 ppm
 F1 24150.82 Hz
 F2P -0.037 ppm
 F2 -3.77 Hz
 PPMCM 12.00174 ppm/cm
 HZCM 1207.72949 Hz/cm

SI-24

F2 - Acquisition Parameters
 Date_ 20040530
 Time 18.35
 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
 DM 104.400 usec
 DE 5.50 usec
 TE 679.6 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCNRK 0.01500000 sec

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

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

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
 PPMCH 0.59846 ppm/cm
 HZCH 239.46362 Hz/cm

Current Data Parameters
 NAME 11-292
 EXNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040530
 Time 18.38
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 95
 DS 4
 SWH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.3566452 sec
 RG 32768
 DM 20.700 usec
 DE 5.50 usec
 TE 679.6 K
 D1 0.15000000 sec
 d11 0.01000000 sec
 DELTA 0.05000000 sec
 MCREST 0.00000000 sec
 MCNRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 12.30 usec
 PL1 2.00 dB
 SFO1 100.6248025 MHz

***** CHANNEL f2 *****
 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 65536
 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 240.037 ppm
 F1 24150.82 Hz
 F2P -0.037 ppm
 F2 -3.77 Hz
 PPMCH 12.00374 ppm/cm
 HZCH 1267.72943 Hz/cm

SI-25

11

F2 - Acquisition Parameters
Date_ 20031114
Time 12.00
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 128
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCH 0.59846 ppm/cm
HZCM 239.46359 Hz/cm

Current Data Parameters
NAME ITI-67
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031114
Time 12.11
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 252
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 724.1
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
SFO1 100.6232931 MHz

===== CHANNEL f2 =====
CFDPRG2 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 -503.06 Hz
F2 -503.06 Hz
PPMCH 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

SI-26

F2 - Acquisition Parameters
 Date_ 20030901
 Time 20.29
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 32
 OW 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
 CY 18.00 cm
 F1P 10.985 ppm
 F1 4395.28 Hz
 F2P -0.985 ppm
 F2 -393.99 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46362 Hz/cm

Current Data Parameters
 NAME dsr-III-36c
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030901
 Time 20.32
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 137
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 OW 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 100.6127290 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 22138.41 Hz
 F2P -10.036 ppm
 F2 -1009.74 Hz
 PPMCM 11.50359 ppm/cm
 HZCM 1157.40747 Hz/cm

SI-27

F2 - Acquisition Parameters
 Date_ 20030922
 Time 14.35
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 OS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 32
 DM 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 DI 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
 PPMCH 0.59846 ppm/cm
 HZCH 239.46362 Hz/cm

Current Data Parameters
 NAME en-1-12
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030922
 Time 14.42
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 321
 OS 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
 DI 0.05000000 sec
 DI1 0.03000000 sec
 DI2 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 22138.41 Hz
 F2P -10.036 ppm
 F2 -1009.74 Hz
 PPMCH 11.50359 ppm/cm
 HZCH 1157.40747 Hz/cm

SI-28

F2 - Acquisition Parameters
 Date_ 20030926
 Time 20.44
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SMH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 35.9
 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
 PPHCN 0.59846 ppm/cm
 HZCN 239.46362 Hz/cm

Current Data Parameters
 NAME dsr-111-48
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030926
 Time 20.48
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 85536
 SOLVENT CDCl3
 NS 123
 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
 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
 PPRG2 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 22138.41 Hz
 F2P -10.036 ppm
 F2 -1009.74 Hz
 PPHCN 11.50359 ppm/cm
 HZCN 1157.40747 Hz/cm

SI-29

F2 - Acquisition Parameters
 Date_ 20031104
 Time 8.45
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 25.4
 DH 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
 MDW 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
 PPHCM 0.59846 ppm/cm
 HZCM 239.46362 Hz/cm

Current Data Parameters
 NAME eh-I-25
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031105
 Time 8.13
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 653
 DS 2
 SMH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 1448.2
 DH 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.05000000 sec
 d11 0.03000000 sec
 d12 0.0002000 sec

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

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

F2 - Processing parameters
 SI 32768
 SF 100.6127290 MHz
 MDW 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 22138.41 Hz
 F2P -10.036 ppm
 F2 -1009.74 Hz
 PPHCM 11.50359 ppm/cm
 HZCM 1157.40747 Hz/cm

SI-30

16

F2 - Acquisition Parameters
 Date_ 20031208
 Time 12.23
 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 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 III-93
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031208
 Time 12.29
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 362
 DS 2
 SWH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.3566452 sec
 RG 32768
 DW 20.700 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 12.30 usec
 PL1 2.00 dB
 SFO1 100.6248025 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 -501.06 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.73999 Hz/cm

SI-31

F2 - Acquisition Parameters
 Date_ 20030918
 Time 23.43
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 4
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 DM 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.70 usec
 PL1 -5.00 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 239.46362 Hz/cm

Current Data Parameters
 NAME dsr-111-42
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030918
 Time 23.46
 INSTRUM drx400
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 203
 OS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 DM 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.05000000 sec
 d11 0.03000000 sec
 d12 0.0002000 sec

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

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1315005 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 22138.41 Hz
 F2P -10.036 ppm
 F2 -1009.74 Hz
 PPMCM 11.50359 ppm/cm
 HZCM 1157.40747 Hz/cm

SI-32

F2 - Acquisition Parameters
Date_ 20031207
Time 16.26
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SMH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 114
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
H2CM 239.46359 Hz/cm

Current Data Parameters
NAME III-92-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031207
Time 16.30
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 289
DS 2
SMH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 32768
DW 20.700 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 12.30 usec
PL1 2.00 dB
SFO1 100.6248025 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 240.037 ppm
F1 24150.82 Hz
F2P -3.77 Hz
F2 -12.00374 ppm/cm
PPMCM 12.00374 ppm/cm
H2CM 1207.72949 Hz/cm

SI-33

F2 - Acquisition Parameters

Date_	20040202
Time	17.31
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	45.3
DW	104.400 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
PL1	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	III-145-2b
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20040202
Time	17.34
INSTRUM	drx400
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	169
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
PL1	12.39 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

Cu
NA
EX
PR

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F2 - Acquisition Parameters
Date_ 20040206
Time 14.54
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
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPHCH 0.59846 ppm/cm
HZCH 239.46359 Hz/cm

Current Data Parameters
NAME 111-149-2b
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040206
Time 14.57
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 163
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

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

F2 - Processing parameters
SI 32768
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
PPHCH 11.50359 ppm/cm
HZCH 1157.40747 Hz/cm

SI-35

22

Cur
NAM
EXT
PRC

F2 - Acquisition Parameters
Date_ 20040205
Time 17.25
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 114
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
PPMCH 0.59846 ppm/cm
HZCM 239.46359 Hz/cm

Current Data Parameters
NAME III-107-Jc
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040205
Time 17.29
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 262
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
PPMCH 11.50359 ppm/cm
HZCM 1157.40747 Hz/cm