**Rearrangements of Bicyclic Nitrones to Lactams: Comparison of Photochemical
and Modified Barton Conditions**

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Supporting Information

Experimental Section

Copies of ^1H and ^{13}C spectrum of new compounds

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General Information. General procedures for the preparation of iodoalkyl or bromoalkyl ketone intermediates have been described.¹⁻³ The following lactams: **15-17**, **19-20**, **24**, **27** and the vinylogous amide **35** are known compounds.^{2,3}

General Procedure for preparation of the *N,O*-di(butoxycarbonyl)hydroxyl-amino-alkyl)ketones. Iodoalkyl or bromoalkyl ketone (5-12 mmol) was dissolved in 15-20 mL of DMF, *N,O*-di(butoxycarbonyl)hydroxylamine⁴ (0.83-0.95 equiv) and K_2CO_3 (1.2 equiv) were added at room temperature and the reaction mixture was stirred for 24 h. The reaction 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 77-92% of *N,O*-di(butoxycarbonyl)hydroxylaminoalkyl)ketones isolated as oils.

(±)-2-(4'-*N,O*-Di(butoxycarbonyl)hydroxylaminobutyl)cyclopentanone (1). (±)-2-(4'-Iodobutyl)cyclopentanone² (3.2 g, 12 mmol) afforded 2.9 g (78%) of **1** isolated as an oil. TLC (10:1 hexane/EtOAc): $R_f = 0.32$. 1H NMR (400 MHz, $CDCl_3$) δ 1.20-1.42

(m, 3H), 1.47 (s, 9H), 1.52 (s, 9H), 1.59 (m, 2H), 1.70-2.32 (complex, 8H), 3.57 (br s, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 20.9, 24.8, 27.2, 27.8, 28.3, 29.5, 29.7, 38.3, 49.3, 50.1, 82.4, 84.9, 152.5, 155.1, 221.5; IR (neat) 1796, 1730 cm^{-1} ; MS (ESI) m/z 372 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{19}\text{H}_{34}\text{NO}_6$ ($\text{M}^+ + \text{H}$): 372.2386, found 372.2370.

(±)-2-(4'-N,O-Di(butoxycarbamyl)hydroxylaminopropyl)cyclohexanone (2).

(±)-2-(4'-Iodopropyl)cyclohexanone² (2.0 g, 7 mmol) afforded 1.9 g (79%) of **2** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.35. ^1H NMR (400 MHz, CDCl_3) δ 1.20-1.40 (m, 2H), 1.46 (s, 9H), 1.51 (s, 9H), 1.58-2.38 (complex, 11H), 3.55 (br s, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 25.1, 25.3, 26.8, 28.0, 28.4, 28.5, 34.4, 42.4, 50.6, 50.7, 82.6, 85.1, 152.7, 155.3, 213.4; IR (neat) 1789, 1716 cm^{-1} ; MS (ESI) m/z 372 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{19}\text{H}_{34}\text{NO}_6$ ($\text{M}^+ + \text{H}$): 372.2386, found 372.2380.

(±)-2-(4'-N,O-Di(butoxycarbamyl)hydroxylaminopentyl)cyclohexanone (3).

(±)-2-(4'-Iodobutyl)cyclohexanone² (1.2 g, 4.6 mmol) afforded 1.36 g (80%) of **3** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.36. ^1H NMR (400 MHz, CDCl_3) δ 1.15-1.35 (m, 4H), 1.44 (s, 9H), 1.50 (s, 9H), 1.54-2.37 (complex, 11H), 3.54 (br s, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 24.5, 25.3, 27.5, 28.0, 28.4, 28.5, 29.3, 34.2, 42.4, 50.4, 51.0,

82.5, 85.0, 152.7, 155.3, 213.6; IR (neat) 1781, 1716 cm^{-1} ; MS (ESI) m/z 386 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{20}\text{H}_{36}\text{NO}_6$ ($\text{M}^+ + \text{H}$): 386.2543, found 386.2564.

(±)-2-(4'-N,O-di(butoxycarbonyl)hydroxylaminopentyl)cyclohexanone (4).

(±)-2-(4'-Iodopentyl)cyclohexanone² (1.4 g, 4.7 mmol) afforded 1.44 g (82%) of **4d** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.37. ^1H NMR (400 MHz, CDCl_3) δ 1.10-1.36 (m, 6H), 1.45 (s, 9H), 1.50 (s, 9H), 1.54-2.36 (complex, 11H), 3.53 (br s, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 25.2, 27.1, 27.2, 27.3, 28.0, 28.4, 28.5, 29.6, 34.3, 42.4, 50.4, 51.0, 82.5, 85.0, 152.7, 155.3, 213.8; IR (neat) 1789, 1716 cm^{-1} ; MS (ESI) m/z 400 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{21}\text{H}_{38}\text{NO}_6$ ($\text{M}^+ + \text{H}$): 400.2699, found 400.2696.

(±)-2-(4'-N,O-di(butoxycarbonyl)hydroxylaminobutyl)cycloheptanone (5).

(±)-2-(4'-Iodobutyl)cycloheptanone² (1.23 g, 4.6 mmol) afforded 1.36 g (80%) of **5** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.39. ^1H NMR (400 MHz, CDCl_3) δ 1.25-1.38 (m, 7H), 1.48 (s, 9H), 1.54 (s, 9H), 1.57-2.53 (complex, 10H), 3.56 (br s, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 24.7, 24.9, 27.5, 28.0, 28.5, 28.9, 29.9, 31.6, 32.3, 43.2,

50.4, 52.6, 82.6, 85.1, 152.7, 155.3, 216.7; IR (neat) 1781, 1716 cm^{-1} ; MS (ESI) m/z 400 (M^++1); HRMS calcd for $\text{C}_{21}\text{H}_{38}\text{NO}_6$ (M^++H): 400.2699, found 400.2680.

(±)-2-(4'-N,O-di(butoxycarbamyl)hydroxylaminopropyl)cyclooctanone (6).

(±)-2-(4'-Iodopropyl)cyclooctanone² (1.2 g, 4.5 mmol) afforded 1.4 g (82%) of **6** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.38. ^1H NMR (400 MHz, CDCl_3) δ 1.16-1.43 (m, 6H), 1.47 (s, 9H), 1.53 (s, 9H), 1.55-2.57 (complex, 11H), 3.54 (br s, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 25.1, 25.4, 25.8, 26.1, 27.8, 28.0, 28.5, 29.7, 33.2, 42.5, 50.3, 50.5, 82.6, 85.1, 152.6, 155.3, 220.3; IR (neat) 1781, 1701 cm^{-1} ; MS (ESI) m/z 400 (M^++1); HRMS calcd for $\text{C}_{21}\text{H}_{38}\text{NO}_6$ (M^++H): 400.2699, found 400.2678.

(±)-2-(4'-N,O-Di(butoxycarbamyl)hydroxylaminobutyl)cyclooctanone (7).

(±)-2-(4'-Iodobutyl)cyclooctanone² (3.4 g, 11 mmol) afforded 3.3 g (85%) of **7** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.42. ^1H NMR (400 MHz, CDCl_3) δ 1.15-1.44 (m, 6H), 1.47 (s, 9H), 1.53 (s, 9H), 1.57-2.60 (complex, 13H), 3.54 (br s, 2H); ^{13}C NMR

(100.6 MHz, CDCl₃) δ 24.9, 25.2, 25.8, 26.1, 27.5, 27.8, 28.0, 28.5, 32.5, 33.2, 42.6, 50.3, 50.9, 82.6, 85.1, 152.7, 155.3, 220.6; IR (neat) 1789, 1716 cm⁻¹; MS (ESI) m/z 414 (M⁺+1); HRMS calcd for C₂₂H₄₀NO₆ (M⁺+H): 414.2865, found 414.2845.

General Procedure for Preparation of Nitrones. *Method A.*¹ *N,O*-DiBoc-hydroxyaminoketone (1-2 mmol) was dissolved in TFA (1.5-2.5 mL) and CH₂Cl₂ to which was added 0.5-0.8 g of 4 Å molecular sieves. The reaction was stirred for 24 h and then concentrated. To the residue was added CH₂Cl₂ (10 mL) and NaHCO₃ (1.7-2.5 g). After stirring for 2 h, the reaction mixture was filtered through Celite and concentrated to give an oil. Flash chromatography using neutral alumina/silica gel (10% EtOAc/hexane, followed by 1-2% MeOH/CHCl₃) afforded nitrones in 75-90% yields.

Method B. *N,O*-DiBoc-hydroxyamino ketone (0.5-2 mmol) and TFA (1.0-2.5 mL) were dissolved in 5-10 mL of CH₂Cl₂. The reaction was stirred for 1 h, the solvent was evaporated and benzene (15 mL) was added. The mixture was refluxed using a Dean-Stark condenser for another 2 h. Concentration followed by chromatography using neutral alumina/silica gel (10% EtOAc/Hexane, followed by 1-2% MeOH/CHCl₃) afforded nitrones in 78-95% yields.

(±)-2,3,4,5,5a,6,7,8-Octahydrocyclopent[b]azepine-N-oxide (8). Compound **1** (400 mg, 1.1 mmol), using method A, gave 140 mg (85%) of nitrone **8** as a white powder. Mp 28-30 °C; TLC (10:1 MeOH/CHCl₃): *R_f* = 0.38. ¹H NMR (400 MHz, CDCl₃) δ 1.26-2.11 (complex, 10H), 2.70 (m, 3H), 3.98 (m, 2H); ¹³C NMR (100.6 MHz, CDCl₃) δ 23.4, 25.7, 29.4, 31.9, 32.2, 35.3, 42.8, 66.6, 159.3; IR (neat) 1650 cm⁻¹; MS (EI) *m/z* 154 (*M*⁺+1); HRMS calcd for C₉H₁₆NO (*M*⁺+1): 154.1232, found 154.1251.

(±)-2,3,4,4a,5,6,7,8-Octahydroquinoline-N-oxide (9). Compound **2** (300 mg, 0.8 mmol), using method A, gave 110 mg (90%) of **9** as an oil. TLC (10:1 MeOH/CHCl₃): *R_f* = 0.37. ¹H NMR (400 MHz, CDCl₃) δ 1.20-1.46 (complex, 4H), 1.76-1.92 (complex, 7H), 2.35 (m, 1H), 3.69 (m, 1H), 3.80-3.82 (m, 2H); ¹³C NMR (100.6 MHz, CDCl₃) δ 22.1, 25.2, 25.3, 26.4, 27.1, 34.1, 38.5, 58.9, 151.7; IR (neat) 1643 cm⁻¹; MS (CI) *m/z* 154 (*M*⁺+1); HRMS calcd for C₉H₁₆NO (*M*⁺+1): 154.1232, found 154.1232.

(±)-2,3,4,5,5a,6,7,8,9-Nonahydrocyclohex[b]azepine-N-oxide (10). Compound **3** (526 mg, 1.36 mmol), using method A, gave 183 mg (80%) of **10** as an oil. TLC (10:1 MeOH/CHCl₃): R_f = 0.35. ¹H NMR (400 MHz, CDCl₃) δ 1.46-1.60 (complex, 6H), 1.65-1.80 (complex, 5H), 1.94 (m, 1H), 2.38 (m, 1H), 2.79 (m, 1H), 3.00-3.06 (dt, J = 5.6, 19.5 Hz, 1H), 3.98 (dd, J = 5.5, 15.8 Hz, 1H), 4.30-4.36 (t, J = 11.9 Hz, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 20.7, 21.9, 24.6, 25.5, 29.3, 32.1, 32.2, 38.4, 64.1, 152.6; IR (neat) 1650 cm⁻¹; MS (CI) m/z 168 (M^+ +1); HRMS calcd for C₁₀H₁₈NO (M^+ +1): 168.1388, found 168.1381.

(±)-2,3,4,5,6,6a,7,8,9,10-Decahydrocyclohex[b]azocine-N-oxide (11). Compound **4** (500 mg, 1.25 mmol), using method A, gave 200 mg (88%) of **11** as an oil. TLC (10:1 MeOH/CHCl₃): R_f = 0.36. ¹H NMR (400 MHz, CDCl₃) δ 1.14-2.10 (complex, 15H), 3.01 (m, 1H), 3.53 (m, 1H), 3.80 (dt, J = 3.7, 12.8 Hz, 1H), 4.37 (ddd, J = 2.2, 12.8 Hz, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 20.1, 23.9, 24.3, 25.7, 26.4, 30.0, 30.9, 36.1, 36.7, 60.4,

152.4; IR (neat) 1658 cm^{-1} ; MS (EI) m/z 182 ($M^+ + 1$); HRMS calcd for $\text{C}_{11}\text{H}_{20}\text{NO}$ ($M^+ + 1$): 182.1545, found 182.1523.

(±)-2,3,4,5,5a,6,7,8,9,10-Decahydrocyclohept[b]azepine-N-oxide (12). Compound **5** (400 mg, 1.0 mmol), using method B, gave 172 mg (95%) of **12** as an oil. TLC (10:1 MeOH/ CHCl_3): R_f = 0.38. ^1H NMR (400 MHz, CDCl_3) δ 1.14-2.10 (complex, 15H), 2.62 (m, 1H), 3.41 (m, 1H), 3.98 (dt, J = 4, 12.8 Hz, 1H), 4.33 (t, J = 11.6 Hz, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 25.1, 26.8, 27.5, 28.4, 29.3, 30.9, 31.4, 34.9, 44.3, 64.6, 156.5; IR (neat) 1654 cm^{-1} ; MS (EI) m/z 182 ($M^+ + 1$); HRMS calcd for $\text{C}_{11}\text{H}_{20}\text{NO}$ ($M^+ + 1$): 182.1545, found 182.1550.

(±)-2,3,4,4a,5,6,7,8,9,10-Decahydrocycloocta[b]pyridine-N-oxide (13). Compound **6** (700 mg, 1.76 mmol), using method A, afforded 248 mg (78%) of **13** as an oil. TLC (10:1 MeOH/ CHCl_3): R_f = 0.38. ^1H NMR (400 MHz, CDCl_3) δ 0.95-2.15 (complex, 15H), 2.59(m, 1H), 3.11 (m, 1H), 3.78 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 22.4, 24.2,

24.7, 25.4, 25.6, 26.2, 28.6, 30.3, 38.7, 58.8, 154.3; IR (neat) 1649 cm^{-1} ; MS (CI) m/z 182 (M^++1); HRMS calcd for $\text{C}_{11}\text{H}_{20}\text{NO}$ (M^++1): 182.1545, found 182.1554.

(±)-2,3,4,5,5a,6,7,8,9,10,11-Undecahydrocyclooct[b]azepine-N-oxide (14).

Compound **7** (410 mg, 1.0 mmol), using method B, gave 170 mg (87%) of **14** as an oil. TLC (10:1 MeOH/ CHCl_3): R_f = 0.48. ^1H NMR (400 MHz, CDCl_3) δ 1.09-2.20 (complex, 17H), 2.48 (m, 1H), 2.89 (m, 1H), 3.90 (m, 1H), 4.30 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 24.8, 25.1, 25.7, 25.9, 27.6, 28.2, 29.9, 32.1, 32.2, 42.4, 64.0, 156.1; IR (neat) 1687 cm^{-1} ; MS (CI) m/z 196 (M^++1); HRMS calcd for $\text{C}_{12}\text{H}_{22}\text{NO}$ (M^++1): 196.1701, found 196.1709.

4-N,O-Di(butoxycarbonyl)hydroxylaminobutylphenylketone (22). 4-Iodo-butylphenyl-ketone² (3.4 g, 11 mmol) using the general method described above afforded 3.5 g (89%) of **22** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.33. ^1H NMR (400 MHz, CDCl_3) δ 1.46 (s, 9H), 1.51(s, 9H), 1.69 (m, 2H), 1.80 (m, 2H), 2.98 (t, J = 10.8 Hz, 2H), 3.62 (br s, 2H), 7.42-7.94 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 21.7, 27.1, 27.8, 28.0, 28.2, 28.5, 28.8, 38.4, 50.2, 82.6, 85.1, 128.4, 129.0, 133.3, 137.3, 152.7, 155.3,

200.2; IR (neat) 1782, 1710, 1690 cm^{-1} ; MS (ESI) m/z 394 (M^++1); HRMS calcd for $\text{C}_{21}\text{H}_{32}\text{NO}_6$ ($M^++\text{H}$): 394.2230, found 394.2210.

2-Phenyl-,3,4,5,6-tetrahydropyridine-N-oxide (23). Compound **22** (780 mg, 2 mmol), using method B, gave 300 mg (85%) of **23** as an oil. TLC (10:1 MeOH/ CHCl_3): R_f = 0.40. ^1H NMR (400 MHz, CDCl_3) δ 1.87-1.90 (t, J = 5.8 Hz, 2H), 2.01-2.03 (t, J = 5.0 Hz, 2H), 2.79-2.82 (m, 2H), 3.99-4.02 (m, 2H), 7.35-7.41 (m, 3H), 8.01-8.03 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.7, 23.6, 29.7, 60.8, 128.5, 128.7, 129.8, 134.4, 142.6; IR (neat) 1685 cm^{-1} ; MS (CI) m/z 176 (M^++1); HRMS calcd for $\text{C}_{11}\text{H}_{14}\text{NO}$ (M^++1): 176.1075, found 176.1073.

(±)-6-(N,O-Di(butoxycarbonyl)hydroxylamino)-6-phenyl-2-hexanone (25). (±)-6-bromo-6-phenyl-2-hexanone³ (1.8 g, 7 mmol) using the general method described above afforded 2 g (77%) of **25** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.22. ^1H NMR (400 MHz, CDCl_3 ; mixture of rotamers) δ 1.22-2.03 (complex, 23H), 2.07 (s, 3H), 2.42 (m, 2H), 5.14 (m, 1H), 7.19-7.34 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3 ; mixture of rotamers) δ 14.5, 17.2, 20.8, 21.1, 21.2, 23.0, 27.7, 27.9, 28.4, 30.1, 30.4, 31.4, 31.9, 35.0,

43.5, 62.2, 63.6, 77.3, 77.6, 77.9, 82.6, 82.9, 84.5, 84.7, 119.6, 128.2, 128.4, 128.7, 128.9, 137.9, 139.6, 151.8, 152.8, 154.6, 155.4, 208.6; IR (neat) 1789, 1709 cm^{-1} ; MS (ESI) m/z 408 (M^++1); HRMS calcd for $\text{C}_{22}\text{H}_{34}\text{NO}_6$ ($M^++\text{H}$): 408.2386, found 408.2377.

(±)-2-Phenyl-6-methyl-2,3,4,5-tetrahydropyridine-N-oxide (26). Compound **25** (800 mg, 2.0 mmol), using method A, gave 290 mg (77%) **26** as an oil. TLC (20:1 MeOH/ CHCl_3): R_f = 0.09. ^1H NMR (400 MHz, CDCl_3) δ 1.59 (m, 2H), 2.06 (m, 1H), 2.25 (m, 4H), 2.54 (m, 2H), 5.06 (br s, 1H), 7.13-7.33 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 15.0, 19.2, 31.2, 31.6, 71.1, 126.7, 127.8, 129.0, 139.6, 147.8; IR (neat) 1629 cm^{-1} ; MS (CI) m/z 190 (M^++1); HRMS calcd for $\text{C}_{12}\text{H}_{16}\text{NO}$ (M^++1): 190.1232, found 190.1223.

3-(*S)-(4'-*N,O*-Di(butoxycarbonyl)hydroxylaminobutyl)-(1*R**,4*S**)-norcamphor (28).** 2-(*S**)-(4'-Iodobutyl)-(1*R**,4*S**)-norcamphor⁵ (1.50 g, 5.1 mmol) using the general method described above afforded 1.80 g (92%) of **28** isolated as an oil. TLC (10:1 hexane/EtOAc): R_f = 0.33. ^1H NMR (500 MHz, CDCl_3) δ 1.19-1.88 (complex, 13H), 1.44 (s, 9H), 1.50 (s, 9H), 2.39 (m, 1H), 2.49 (m, 1H), 3.54 (br s, 2H); ^{13}C NMR (100.6

MHz, CDCl₃) δ 24.4, 25.7, 27.3, 28.0, 28.3, 28.5, 29.0, 35.2, 39.4, 49.9, 50.2, 54.3, 82.5, 85.1, 152.7, 155.3, 220.6; IR (neat) 1775, 1720, 1710 cm⁻¹; MS (ESI) m/z 398 (M⁺+1); HRMS calcd for C₂₁H₃₆NO₆ (M⁺+H): 398.2543, found 398.2514.

(6a*S,7*S**,10*R**)-(3-Aza-tricyclo[7.2.1.0^{127,233}]*dodec-2-ene-N-oxide* (29).**

Compound **28** (400 mg, 1.0 mmol), using method B, gave 165 mg (92%) of nitron **29** as a white powder. Mp 142-144 °C; TLC (20:1 MeOH/CHCl₃): R_f = 0.05. ¹H NMR (400 MHz, CDCl₃) δ 1.22 (m, 3H), 1.46 (m, 2H), 1.55-1.72 (complex, 6H), 1.95 (m, 1H), 2.10 (m, 2H), 3.40 (br s, 1H), 3.80 (m, 1H), 4.01 (m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 25.5, 26.3, 28.5, 29.4, 30.1, 36.5, 40.9, 44.0, 50.4, 67.0, 159.4; IR (neat) 1636 cm⁻¹; MS (CI) m/z 180 (M⁺+1); HRMS calcd for C₁₁H₁₈NO (M⁺+1): 180.1388, found 180.1385.

General Procedure for the Rearrangement of Nitrones: 10a*R-Octahydro-7,10-methanopyrido[1,2-*a*]azepin-6(2*H*)-one (30). Method A.¹** A degassed mixture of nitron **29** (95 mg, 0.5 mmol) in benzene (0.025 M) was photolyzed in a capped quartz tube (254 nm, Merry-Go-Round apparatus) for 2h. Concentration followed by chromatography (10% EtOAc/hexane, followed by 1-2% MeOH/CHCl₃) afforded 60 mg

(63%) of **30** isolated as an oil. TLC (20:1 MeOH/CHCl₃): R_f = 0.23. ¹H NMR (400 MHz, CDCl₃) δ 1.21-1.53 (complex, 7H), 1.72-1.94 (complex, 5H), 2.10 (m, 1H), 2.27 (m, 1H), 2.63 (m, 1H), 2.88 (br d, J = 10.6 Hz, 1H), 4.53 (dt, J = 2.1, 13.0 Hz, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 25.5, 25.9, 30.1, 30.4, 30.6, 32.6, 39.6, 42.8, 44.2, 65.4, 173.6; IR (neat) 2930, 1643 cm⁻¹; MS (EI) m/z 179 (M^+); HRMS calcd for C₁₁H₁₇NO (M^+ +1): 180.1376, found 180.1388.

Method B. Nitron **29** (280 mg, 1.6 mmol) was added to a solution of triflic anhydride (1.8 equiv) in 10 mL of CH₂Cl₂ and stirred for 2 h. The solvent was evaporated and 25 mL of 15% KOH was added and the reaction was allowed to stir for another 24 h. The reaction mixture was extracted with CH₂Cl₂. The combined organic layers were dried over Na₂SO₄, filtered, and concentrated to give an oil. Chromatography (10% EtOAc/hexane, followed by 1-2% MeOH/CHCl₃) afforded 265 mg (95%) of **30** isolated as an oil.

(±)-Decahydro-azapino[1,2-a]azepin-5-one (18). **Method A.** Nitron **11** (100 mg, 0.56 mmol) in benzene (20 mL), using method A, afforded 35 mg (35%) of lactam **18** as an oil. TLC (10:1 MeOH/CHCl₃): R_f = 0.72. ¹H NMR (400 MHz, CDCl₃) δ 1.27-1.91 (complex, 15H), 2.48-2.53 (m, 1H), 2.68-2.78 (m, 2H), 3.64-3.69 (m, 1H), 4.15-4.20(m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 23.6, 27.3, 28.8, 29.6, 30.4, 34.1, 36.0,

37.0, 41.5, 60.2, 176.1; IR (neat) 2930, 1636 cm^{-1} ; MS (CI) m/z 182 (M^++1); HRMS calcd for $\text{C}_{11}\text{H}_{20}\text{NO}$ (M^++1): 182.1545, found 182.1547.

Method B. Nitron **11** (80 mg, 0.44 mmol), using method B, afforded 32 mg (40%) lactam **18** as an oil.

(±)-Undecahydro-6H-pyridin[1,2-a]azonin-6-one (21). **Method A.** Nitron **14** (100 mg, 0.51 mmol) in benzene (20 mL), using method A, afforded 45 mg (45%) of lactam **21** as an oil. TLC (10:1 MeOH/ CHCl_3): R_f = 0.76. ^1H NMR (400 MHz, CDCl_3) δ 1.01-1.97 (complex, 15H), 1.98-2.10 (m, 1H), 2.15-2.30 (m, 1H), 2.40-2.52 (m, 3H), 4.0-4.23 (m, 1H), 4.40-4.43 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.1, 20.1, 25.1, 25.5, 27.9, 29.5, 29.7, 29.9, 36.6, 37.0, 52.5, 175.5; IR (neat) 2930, 1629 cm^{-1} ; MS (CI) m/z 196 (M^++1); HRMS calcd for $\text{C}_{12}\text{H}_{22}\text{NO}$ (M^++1): 196.1701, found 196.1707.

Method B. Nitron **14** (100 mg, 0.51 mmol), using method B, afforded 35 mg (35%) lactam **21** as an oil.

1-Acetoxy-2-(3'-N,O-di(butoxycarbonyl)hydroxylaminopropyl)-1-cyclohexene (A). 1-Acetoxy-2-(3'-iodopropyl)-1-cyclohexene (1.4 g, 4.50 mmol, prepared from the corresponding chloride as previously described²) afforded 1.8 g (97%) of compound **A** as

an oil. TLC (5:1 hexane/EtOAc): R_f = 0.39. ^1H NMR (400 MHz, CDCl_3) δ 1.49 (s, 9H), 1.51 (s, 9H), 1.61-1.70 (complex, 6H), 1.98 (m, 2H), 2.05 (br s, 2H), 2.13 (br s, 2H), 2.14 (s, 3H), 3.54 (br s, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 21.3, 22.8, 23.4, 25.1, 27.5, 27.6, 28.0, 28.4, 28.5, 50.4, 82.6, 85.1, 123.6, 143.0, 152.7, 155.3, 169.8; IR (neat) 1785, 1752, 1716 cm^{-1} ; MS (ESI) m/z 414 (M^+); HRMS calcd for $\text{C}_{21}\text{H}_{39}\text{N}_2\text{O}_7$ (M^+): 431.2757, found 431.2748.

2-(3'-N,O-di(butoxycarbamyl)hydroxylaminopropyl)-2-(phenylselenenyl)-cyclohexanone (B). To silver trifluoroacetate (0.81 g, 3.60 mmol) in 15 mL benzene was added the di-Boc-hydroxylamine prepared above (1.37 g, 3.30 mmol) followed by the addition of phenylselenenyl chloride in 2 mL of benzene (0.70 g, 3.60 mmol). The solution was stirred for 30 min and then quenched with NaHCO_3 (4.0 mmol in 1 mL of H_2O). The reaction mixture was filtered, dried (Na_2SO_4), and concentrated to give an oil. Flash chromatography using silica gel (2-5% EtOAc/hexane) afforded compound **B** 1.4 g (80%) as an oil. TLC (5:1 hexane/EtOAc): R_f = 0.33. ^1H NMR (400 MHz, CDCl_3) δ 1.26 (br s, 1H), 1.47 (s, 9H), 1.54 (s, 9H), 1.59-2.10 (complex, 9H), 2.23 (br d, J = 14.1 Hz, 1H), 2.33 (br d, J = 15.1 Hz, 1H), 3.38-3.47 (m, 3H), 7.27-7.43 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 22.5, 22.6, 26.8, 28.0, 28.5, 32.6, 36.9, 37.7, 50.8, 60.3, 82.6, 85.1, 126.7, 129.4, 129.8, 137.8, 152.7, 155.1, 206.7; IR (neat) 1784, 1715, 1693 cm^{-1} ; MS (ESI) m/z 528 (M^+); HRMS calcd for $\text{C}_{25}\text{H}_{41}\text{N}_2\text{O}_6\text{Se}$ (M^+): 545.2130, found 545.2148.

2-(3'-N,O-di(butoxycarbonyl)hydroxylaminopropyl)cyclohex-2-enone (31).

To a solution of selenide as prepared above (1.0 g, 1.90 mmol) in CH₂Cl₂ (25 mL) was added Et₃N (1 mL, 6.93 mmol) and 35% H₂O₂ (8 mL). The resulting solution was refluxed for 1 h. Workup (CHCl₃/H₂O) followed by chromatography (5-10% EtOAc/hexane) afforded 0.64 g (91%) of **31** as an oil. TLC (5:1 hexane/EtOAc): R_f = 0.30. ¹H NMR (400 MHz, CDCl₃) δ 1.47 (s, 9H), 1.54 (s, 9H), 1.70 (m, 2H), 1.98 (m, 2H), 2.24 (m, 2H), 2.34-2.43 (complex, 4H), 3.56 (br s, 2H), 6.77 (t, *J* = 3.8 Hz, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 23.5, 26.2, 26.4, 27.1, 28.0, 28.5, 38.9, 50.0, 82.6, 85.1, 139.1, 146.3, 152.7, 155.3, 199.8; IR (neat) 1774, 1709, 1665 cm⁻¹; MS (ESI) *m/z* 370 (M⁺+1); HRMS calcd for C₁₉H₃₅N₂O₆(M⁺+NH₄): 387.2495, found 387.2503.

2,3,4,6,7,8-Hexahydroquinoline-N-oxide (32). Compound **31** (120 mg, 0.33 mmol), using method B, gave 46 mg (91%) of **32** as an oil. TLC (10:1 MeOH/CHCl₃): R_f = 0.39. ¹H NMR (400 MHz, CDCl₃) δ 1.78 (p, *J* = 6.6 Hz, 2H), 1.96 (p, *J* = 4.2 Hz, 2H), 2.16 (m, 2H), 2.37 (m, 2H), 2.77 (t, *J* = 6.7 Hz, 2H), 3.89 (t, *J* = 5.9 Hz, 2H), 5.82 (t, *J* = 4.5 Hz, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 21.4, 22.5, 24.6, 25.3, 27.2, 59.2,

128.1, 130.5, 145.7; IR (neat) 1650 cm^{-1} ; MS (CI) m/z 152 ($M^+ + 1$); HRMS calcd for $\text{C}_9\text{H}_{14}\text{NO}$ ($M^+ + 1$): 152.1075, found 152.1070.

2,3,4,6,7,8-Hexahydro-oxazirino[3,2-j]quinoline (33). Nitron **32** (50 mg, 0.33 mmol) in benzene (15 mL), using method A, afforded 23 mg (45%) of oxaziridine **33** as an oil. TLC (10:1 MeOH/ CHCl_3): R_f = 0.85. ^1H NMR (400 MHz, CDCl_3) δ 1.37 (m, 1H), 1.50 (m, 1H), 1.83-2.40 (complex, 8H), 3.23 (m, 1H), 3.50 (m, 1H), 5.99 (t, J = 1.7 Hz, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.1, 21.0, 25.9, 26.2, 33.4, 51.6, 78.9, 130.2, 133.8; IR (neat) 2937, 2862, 1437 cm^{-1} ; MS (CI) m/z 152 ($M^+ + 1$); HRMS calcd for $\text{C}_9\text{H}_{14}\text{NO}$ ($M^+ + 1$): 152.1075, found 152.1073.

1,2,3,6,7,8-Hexahydro-pyrrolo[1,2-a]azepin-5-one (33). Method A. Nitron **32** (50 mg, 0.33 mmol) in benzene (15 mL), using method A, afforded 10 mg (20%) of lactam **34** as an oil. TLC (10:1 MeOH/ CHCl_3): R_f = 0.65. ^1H NMR (400 MHz, CDCl_3) δ 1.83 (m, 4H), 2.24 (m, 2H), 2.52-2.62 (complex, 4H), 3.70 (t, J = 7.0 Hz, 2H), 4.91 (t, J = 4.2 Hz, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 21.6, 21.7, 29.4, 34.9, 38.2, 49.4, 104.7, 137.5, 173.4; IR (neat) 1647 cm^{-1} ; MS (CI) m/z 152 ($M^+ + 1$); HRMS calcd for $\text{C}_9\text{H}_{14}\text{NO}$ ($M^+ + 1$): 152.1075, found 152.1075.

Method B. Nitron **32** (50 mg, 0.33 mmol), using method B, afforded 5 mg (10%) lactam **34** as an oil.

References:

- (1) Smith, B. T.; Wendt, J. A.; Aubé, J. *Org. Lett.* **2002**, *4*, 2577-2579.
- (2) Milligan, G. L.; Mossman, C. J.; Aubé, J. *J. Am. Chem. Soc.* **1995**, *117*, 10449-59.
- (3) Wroblewski, A.; Aubé, J. *J. Org. Chem.* **2001**, *66*, 886-889.
- (4) Staszak, M. A.; Doecke, C. W. *Tetrahedron Lett.* **1993**, *34*, 7043-4.
- (5) Wendt, J. A.; Aubé, J. *Tetrahedron Lett.* **1996**, *37*, 1531-4.

S20

F2 - Acquisition Parameters
 Date_ 20020805
 Time 12.41
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146152 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 400.1300052 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 10.972 ppm
 F1 4390.10 Hz
 F2P -0.998 ppm
 F2 -399.17 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME 127
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020805
 Time 12.53
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 177
 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 0.00020000 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 100.6127494 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 FIP 240.037 ppm
 F1 24150.82 Hz
 F2P -0.037 ppm
 F2 -3.77 Hz
 PPMCM 12.00374 ppm/cm
 HZCM 1207.72961 Hz/cm

F2 - Acquisition Parameters
Date_ 20020819
Time 10.05
INSTRUM dtx400
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 400.1300052 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 104-d
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020819
Time 10.10
INSTRUM dtx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 211
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 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 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

S22

F2 - Acquisition Parameters
 Date_ 20030204
 Time 17.23
 INSTRUM drx400
 PROBRHD 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 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 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 76-cdcl3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030204
 Time 17.27
 INSTRUM drx400
 PROBRHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 129
 DS 2
 SWH 24154.590 Hz
 FIDRES 0.168570 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 -0.037 ppm
 F2 -3.77 Hz
 PPMCM 12.00374 ppm/cm
 HZCM 1207.72949 Hz/cm

S23

F2 - Acquisition Parameters
 Date_ 20020808
 Time 15.11
 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 50.8
 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 400.1300052 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 10.972 ppm
 FI 4390.10 Hz
 F2P -0.998 ppm
 F2 -399.17 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME 143-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020808
 Time 15.17
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 199
 DS 2
 SWH 24154.590 Hz
 FIDRES 0.160570 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
 FIP 215.000 ppm
 FI 21631.74 Hz
 F2P -5.000 ppm
 F2 -503.06 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.73999 Hz/cm

S24

F2 - Acquisition Parameters
 Date_ 20030207
 Time 17.34
 INSTRUM drx400
 PROBRD 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 1.0000000 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 108-cdcl3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030207
 Time 17.43
 INSTRUM drx400
 PROBRD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 311
 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 0.0500000 sec
 d11 0.0300000 sec
 d12 0.0000200 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 EN
 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

S25

F2 - Acquisition Parameters

Date_	20030212
Time	14.37
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	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	11-48-3
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20030212
Time	14.45
INSTRUM	drx400
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	291
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	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

S26

F2 - Acquisition parameters
Date_ 20020815
Time 9.06
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 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.984 ppm
F1 3594.67 Hz
F2P -0.210 ppm
F2 -83.84 Hz
PPMCM 0.45966 ppm/cm
HZCM 183.92534 Hz/cm

Current Data Parameters
NAME 122c
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020815
Time 9.18
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 203
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
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

S27

F2 - Acquisition Parameters
 Date_ 20020729
 Time 15.54
 INSTRUM drx400
 PROBHND 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210297 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 400.1300052 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.972 ppm
 F1 4390.10 Hz
 F2P -0.998 ppm
 F2 -399.17 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME 129d
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020729
 Time 15.59
 INSTRUM drx400
 PROBHND 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 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

S28

F2 - Acquisition Parameters

Date_	20020823
Time	17.37
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	101.6
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
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	154-c
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020823
Time	17.42
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	159
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	0.05000000 sec
d12	0.03000000 sec
d12	0.00020000 sec

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

NUC1	13C
PL1	12.30 usec
PL1	2.00 dB
SFO1	100.6232933 MHz

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

CPDPRG2	waltz16
NUC2	1H
PL2	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	100.6127390 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 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

S29

10

F2 - Acquisition Parameters
Date_ 20030206
Time 10.29
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 101.6
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

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

F2 - Processing parameters
SI 16384
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 II-38-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030206
Time 10.34
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 471
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

S30

11

F2 - Acquisition Parameters
 Date_ 20020812
 Time 18.16
 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 90.5
 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 400.1300005 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.983 ppm
 F1 4394.78 Hz
 F2P -0.986 ppm
 F2 -394.50 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME 147
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020812
 Time 18.22
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 233
 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 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

S31

F2 - Acquisition Parameters
 Date_ 20030118
 Time 12.42
 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 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 11-28
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030118
 Time 12.44
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 121
 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 -593.06 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.73999 Hz/cm

S32

13

F2 - Acquisition Parameters

Date_ 20030214

Time 17.26

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

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 II-50c

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20030214

Time 17.32

INSTRUM drx400

PROBHD 5 mm Multinucl

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 735

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 -503.06 Hz

F2 -503.06 Hz

PPMCM 11.00000 ppm/cm

HZCM 1106.73999 Hz/cm

S33

14

F2 - Acquisition Parameters
 Date_ 20021211
 Time 17.50
 INSTRUM dx400
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 40.3
 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 400.1300072 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 10.967 ppm
 F1 4388.05 Hz
 F2P -1.003 ppm
 F2 -401.22 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME II-3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021211
 Time 17.53
 INSTRUM dx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 159
 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

S34

F2 - Acquisition Parameters
 Date_ 20020815
 Time 13.47
 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 101.6
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec

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

F2 - Processing parameters
 SI 16384
 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 10.985 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 150-2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020815
 Time 13.58
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 618
 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 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

S35

F2 - Acquisition Parameters
 Date_ 20020816
 Time 18.24
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 15
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 143.7
 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 10.985 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 151d
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020816
 Time 18.32
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 305
 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 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

F2 - Acquisition Parameters

Date_	20020907
Time	17.24
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	400.1300052 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.972 ppm
F1	4390.10 Hz
F2P	-0.998 ppm
F2	-399.17 Hz
PPMCM	0.59846 ppm/cm
HZCM	239.46359 Hz/cm

Current Data Parameters

NAME	175
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020907
Time	17.30
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	305
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
FL2	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	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

S37

F2 - Acquisition Parameters
 Date_ 20020909
 Time 9.49
 INSTRUM dx400
 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 228.1
 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 400.1300003 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 10.984 ppm
 F1 4394.34 Hz
 F2P -0.986 ppm
 F2 -394.33 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME 178
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020908
 Time 18.00
 INSTRUM dx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 195
 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
 FIP 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

S38

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020926

Time 9.36

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 16

DW 104.400 usec

DE 4.50 usec

TE 300.0 K

D1 1.0000000 sec

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

NUC1 1H

P1 7.70 usec

PL1 -6.00 dB

SFO1 400.1320007 MHz

F2 - Processing parameters

SI 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 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 20ab

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20020926

Time 9.45

INSTRUM drx400

PROBHD 5 mm Multinu

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 493

DS 2

SWH 24154.590 Hz

FIDRES 0.168570 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

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

S39

26

F4 - Acquisition Parameters
 Date_ 20020927
 Time 17.50
 INSTRUM dx400
 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

===== 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 10.985 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 208
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020927
 Time 17.55
 INSTRUM dx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 175
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.153213 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 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

S40

F2 - Acquisition Parameters
 Date_ 20021205
 Time 17.52
 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 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 400.1300049 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 11.000 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 282-2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021205
 Time 17.56
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 135
 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 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

S41

29

F2 - Acquisition Parameters
 Date_ 20021204
 Time 9.05
 INSTRUM drx400
 PROBRD 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 40.3
 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 400.1300069 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 10.967 ppm
 F1 4388.35 Hz
 F2P -1.002 ppm
 F2 -400.93 Hz
 PPMCM 0.59846 ppm/cm
 HZCM 239.46359 Hz/cm

Current Data Parameters
 NAME 285-2
 EXFNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021204
 Time 9.11
 INSTRUM drx400
 PROBRD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 223
 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

S42

30

```

===== 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
FLP        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       287
EXPNO      2
PROCNO     1

```

```

F2 - Acquisition Parameters
Date_      20021205
Time       17.45
INSTRUM    drx400
PROBHD     5 mm Multinucl
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS          139
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
FLP        215.000 ppm
F1         21631.74 Hz
F2P        -503.06 Hz
F2         -503.06 Hz
PPMCM      11.00000 ppm/cm
HZCM       1106.73999 Hz/cm

```

F2 - Acquisition Parameters
 Date_ 20030608
 Time 11.34
 INSTRUM drx400
 PROBED 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 101.6
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec

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

F2 - Processing parameters
 SI 16384
 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 17-205-2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030608
 Time 11.38
 INSTRUM drx400
 PROBED 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 386
 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

S44

F2 - Acquisition Parameters
 Date_ 20030608
 Time 11.47
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 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

===== 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 11-206
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030608
 Time 11.51
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 153
 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 -503.06 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.73999 Hz/cm

S45

F2 - Acquisition Parameters
 Date_ 20030609
 Time 18.08
 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 II-207b
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030609
 Time 18.13
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 226
 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 0.00020000 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

S46

32

Cur
NAM
EXP
PRO

F2
Date_ 20030610
Time 14.37
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
FPCMC 0.59846 ppm/cm
HZCM 239.46359 Hz/cm

Current Data Parameters
NAME II-211-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030610
Time 14.39
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 126
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
FPCMC 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

S47

Current
NAME
EXPNO
PROCNO

33

F2 - Acquisition Parameters
Date_ 20030611
Time 17.14
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
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 II-212-3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030611
Time 18.39
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 996
DS 2
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 574.7
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 1207.72949 Hz/cm
HZCM 1207.72949 Hz/cm

S48

34

F2 - Acquisition Parameters

Date_	20030611
Time	15.56
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	203.2
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	9.016 ppm
F1	3607.57 Hz
F2P	-0.197 ppm
F2	-78.90 Hz
PPMCM	0.46066 ppm/cm
HZCM	184.32396 Hz/cm

Current Data Parameters

NAME	II-215-2
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20030611
Time	17.02
INSTRUM	drx400
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	717
DS	2
SWH	24154.590 Hz
FIDRES	0.368570 Hz
AQ	1.3566452 sec
RG	322.5
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	0.00 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