Regioselective Single and Double Conjugate Additions to Substituted Cyclohexa-2,5-dienone Monoacetals

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Supporting Information

Experimental Section

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

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Preparation of Substituted Cyclohexa-2,5-dienone Monoacetals.

Methyl 5-ethoxy-2-hydroxy benzoate. Oxalyl chloride (1.3 mL, 15 mmol) was added dropwise to a solution of 5-ethoxysalicylic acid (Aldrich, 1.3 g, 7.1 mmol) in 35 mL of THF. A few drops of DMF were added and the reaction was stirred at room temperature for 30 min at which time it was concentrated to a yellow solid. The residue was dissolved in 10 mL of methanol and the reaction stirred for an additional 15 min, followed by quenching with water (30 mL). The aqueous layer was extracted with ethyl acetate (2 × 50 mL), washed with a solution of saturated aqueous NaHCO₃ (1 × 60 mL), and the combined organic layers were dried (MgSO₄), filtered, and concentrated. Chromatography (5-15% EtOAc/hex) provided the product as a white solid (1.24 g, 89%). *R_f* = 0.60 (40% EtOAc/hex); mp 45-47 °C; IR (neat) 3400 (br), 1680, 1610 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.25 (d, *J* = 3.1 Hz, 1H), 7.04 (dd, *J* = 5.9, 3.1 Hz, 1H), 6.87 (d, *J* = 9.0 Hz, 2H), 3.94 (q, *J* = 7.0 Hz, 2H), 3.90 (s, 3H), 1.37 (t, *J* = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 170.2, 155.9, 151.2, 124.4, 118.3, 112.7, 111.7, 64.0, 52.1, 14.7; MS (FAB) *m/z* (relative intensity) 196 (M⁺, 20), 154 (100); HRMS calcd for C₁₁H₁₂O₄ (MH⁺): 197.0814, found: 197.0810.

Methyl 3,3-diethoxy-6-oxocyclohexa-1,4-dienecarboxylate (1). Prepared according to the procedure developed by Pelter et al.¹ Thus, iodobenzene diacetate (1.96 g, 6.1 mmol) was added to a solution of methyl 5-ethoxy-2-hydroxy benzoate (1.2 g, 6.1 mmol) in 50 mL of EtOH with stirring at room temperature. The reaction was complete (TLC) within 30 min at which time the reaction mixture was concentrated and

chromatographed (10-35% EtOAc/hex) to give a yellow oil (1.18 g, 80%). R_f = 0.30 (20% EtOAc/hex); IR (neat) 3400 (br), 1680, 1440 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.41 (t, J = 3.2 Hz, 1H), 6.82 (td, J = 10.4, 1.9 Hz, 1H), 6.24 (dd, J = 10.4, 3.5 Hz, 1H), 3.82 (d, J = 3.6 Hz, 3H), 3.63 (m, 4H), 1.20 (m, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 180.9, 163.9, 148.1, 142.9, 131.3, 129.6, 91.9, 58.5, 52.4, 15.4; MS (FAB) m/z (relative intensity) 240 (M^+ , 45), 209 (34); HRMS calcd for $\text{C}_{12}\text{H}_{16}\text{O}_5$: 240.0998, found: 240.1000.

5-Ethoxy-2-hydroxy-N-phenylbenzamide.² Prepared according to the procedure developed by Herz et. al.³ 5-Ethoxy-2-hydroxy benzoic acid (1.18 g, 6.5 mmol) was placed in a round bottom flask fitted with a reflux condenser. Toluene was added (10 mL) followed by the addition of triphenyl phosphite (2.0 mL, 7.8 mmol) and aniline (0.71 mL, 7.8 mmol). The mixture was then heated to reflux with stirring for 3 h. After cooling to room temperature, a voluminous solid formed, which was washed with two portions of ice-cold chloroform (15 mL each). The resulting solid was dried (P_2O_5) to give a white solid (1.54 g, 92%). R_f = 0.48 (40% EtOAc/hex); mp 157-158 $^\circ\text{C}$; IR (neat) 3180 (br), 1635 cm^{-1} ; ^1H NMR (500 MHz, acetone- d_6) δ 9.79 (s, 1H), 7.77 (d, J = 8.2 Hz, 2H), 7.54 (d, J = 3.0 Hz, 1H), 7.39 (m, 2H), 7.18 (t, J = 5.0 Hz, 1H), 7.11 (dd, J = 9.0, 3.1 Hz, 1H), 6.92 (d, J = 9.0 Hz, 1H), 4.04 (q, J = 6.9 Hz, 2H), 1.37 (t, J = 6.9 Hz, 3H); ^{13}C NMR (125 MHz, acetone- d_6) δ 167.9, 154.8, 151.4, 137.9, 128.6, 124.4, 121.7, 121.2, 118.4, 115.2, 112.0, 63.9, 14.2; MS (FAB) m/z (relative intensity) 258 (MH^+ , 100), 165 (48); HRMS calcd for $\text{C}_{15}\text{H}_{16}\text{NO}_3$ (MH^+): 258.1135, found: 258.1130.

3,3-Diethoxy-6-oxo-*N*-phenylcyclohexa-1,4-dienecarboxamide (2). Oxidation of 5-ethoxy-2-hydroxy-*N*-phenylbenzamide (0.50 g, 1.9 mmol) according to the procedure developed by Pelter et al.¹ afforded a yellow solid (0.48 g, 83%) after chromatography (20% EtOAc/hex). R_f = 0.63 (40% EtOAc/hex); mp 62-64 °C; IR (neat) 3270, 2975, 1695 cm^{-1} ; ^1H NMR (500 MHz, acetone- d_6) δ 10.65 (s, 1H), 8.03 (d, J = 3.3 Hz, 1H), 7.77 (d, J = 7.6 Hz, 2H), 7.38 (t, J = 7.6 Hz, 2H), 7.17 (m, 2H), 6.34 (d, J = 10.2 Hz, 1H), 3.77 (q, J = 7.0 Hz, 4H), 1.22 (t, J = 7.0 Hz, 6H); ^{13}C NMR (125 MHz, acetone- d_6) δ 185.8, 159.5, 151.4, 145.4, 138.3, 129.4, 128.8, 128.7, 124.2, 120.0, 92.0, 58.2, 15.1; MS (FAB) m/z (relative intensity) 302 (MH^+ , 62), 256 (67), 209 (100); HRMS calcd for $\text{C}_{17}\text{H}_{20}\text{NO}_4$ (MH^+): 302.1392, found: 302.1392.

4,4-Dimethoxy-2-(2'-nitrophenyl)cyclohexa-2,5-dienone (3). Iodobenzene diacetate (3.1 g, 9.6 mmol) was added to a solution of 2-hydroxy-2'-nitrobiphenyl³ (1.08 g, 4.70 mmol) in 50 mL of dry methanol.¹ The reaction was stirred at room temperature for 3 h then concentrated to an oil, which was chromatographed (10-40% EtOAc/hex) to give a yellow solid (0.75 g, 74%). R_f = 0.35 (40% EtOAc/hex); mp 97-100 °C; IR (neat) 1680, 1660, 1350 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 8.14 (dd, J = 3.1, 1.0 Hz, 1H), 7.69 (dt, J = 5.0, 1.3 Hz, 1H), 7.59 (dt, J = 8.3, 1.5 Hz, 1H), 7.37 (dd, J = 7.6, 1.4 Hz, 1H), 6.92 (dd, J = 10.4, 3.1 Hz, 1H), 6.84 (d, J = 3.17 Hz, 1H), 6.42 (d, J = 10.3 Hz, 1H), 3.49 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 182.4, 148.6, 143.5, 140.3, 139.5, 133.6, 131.6, 130.3, 130.2, 129.7, 124.4, 93.1, 50.7; MS (FAB) m/z (relative intensity)

276 (MH^+ , 68), 244 (100); HRMS calcd for $\text{C}_{14}\text{H}_{14}\text{NO}_5$ (MH^+): 276.0872, found: 276.0872.

2,5-Dihydroxy-*N*-phenylbenzamide.⁴ 2,5-Dihydroxybenzoic acid (8.0 g, 52 mmol) was dissolved in toluene (45 mL), followed by the addition of triphenyl phosphite (16.4 mL, 62 mmol) and aniline (5.7 mL, 62 mmol) according to the procedure developed by Herz et. al.³ The mixture was heated to reflux for 3 h. After cooling to room temperature, a voluminous solid formed, which was washed with ice-cold chloroform (2 $\times$ 35 mL). The resulting solid was dried (P_2O_5) to give a white solid (11.5 g, 96%). R_f = 0.64 (60% EtOAc/hex); mp 160-163 $^\circ\text{C}$; IR (neat) 3050, 1640, 1590 cm^{-1} ; ^1H NMR (500 MHz, DMSO-d_6) δ 9.10 (s, 1H), 7.70 (d, J = 9.4 Hz, 2H), 7.37 (m, 3H), 7.13 (t, J = 7.4 Hz, 1H), 6.90 (dd, J = 8.8, 2.19 Hz, 1H), 6.83 (d, J = 8.8 Hz, 1H), 3.37 (s, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 166.2, 150.9, 150.1, 138.7, 129.1, 124.4, 121.5, 121.0, 118.4, 118.2, 115.0; MS (FAB) m/z (relative intensity) 230 (MH^+ , 47), 192 (100); HRMS calcd for $\text{C}_{13}\text{H}_{12}\text{NO}_3$ (MH^+): 230.0817, found: 230.0823.

2-Ethoxy-5-hydroxy-*N*-phenylbenzamide. 2,5-Dihydroxy-*N*-phenylbenzamide (1.5 g, 6.5 mmol) was placed in a round-bottom flask fitted with a reflux condenser. Acetone (40 mL) was added, followed by potassium carbonate (0.9 g, 6.5 mmol) and ethyl iodide (0.5 mL, 6.5 mmol). The mixture was heated to reflux and stirred for 15 h, then cooled to room temperature and quenched with a saturated solution of NH_4Cl (40 mL) and extracted with ethyl acetate (4 $\times$ 40 mL). The organic extracts were combined, dried (MgSO_4), filtered and concentrated to give yellow crystals which were

recrystallized from boiling acetone to give light yellow crystals (1.15 g, 68%). R_f = 0.33 (40% EtOAc/hex); mp 180-182 °C; IR (neat) 3315 (br), 1650 cm^{-1} ; ^1H NMR (500 MHz, acetone- d_6) δ 10.26 (s, 1H), 8.27 (s, 1H), 7.79 (d, J = 9.6 Hz, 2H), 7.68 (d, J = 3.9 Hz, 1H), 7.37 (t, J = 10.4 Hz, 2H), 7.10 (m, 2H), 7.01 (dd, J = 11.1, 3.9 Hz, 1H), 4.28 (q, J = 8.7, 2H), 1.59 (t, J = 8.7 Hz, 3H); ^{13}C NMR (125 MHz, acetone- d_6) δ 162.7, 151.5, 150.2, 139.2, 128.9, 123.6, 122.9, 119.7, 119.6, 117.4, 114.6, 65.4, 14.3; MS (FAB) m/z (relative intensity) 258 (MH^+ , 100); HRMS calcd for $\text{C}_{15}\text{H}_{16}\text{NO}_3$ (MH^+): 258.1130, found: 258.1116.

2,2-Diethoxy-5-oxo-N-phenylcyclohexa-1,3-dienecarboxamide (4). Oxidation of 2-Ethoxy-5-hydroxy-*N*-phenylbenzamide (0.64 g, 2.5 mmol) according to the procedure developed by Pelter et al.² afforded a yellow oil (0.73 g, 97%) after chromatography (5% EtOAc/hex). R_f = 0.53 (60% EtOAc/hex); IR (neat) 3331 (br), 2975, 1665 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 9.48 (s, 1H), 7.62 (d, J = 8.0 Hz, 2H), 7.35 (t, J = 7.8 Hz, 2H), 7.23 (d, J = 2.1 Hz, 1H), 7.14 (t, J = 7.5 Hz, 1H), 6.82 (d, J = 10.3 Hz, 1H), 6.47 (dd, J = 10.3, 2.2 Hz, 1H), 3.64 (m, 2H), 3.48 (m, 2H), 1.25 (t, J = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 185.4, 160.8, 146.7, 144.5, 137.9, 134.9, 132.2, 129.6, 125.3, 120.5, 95.5, 60.5, 15.9; MS (FAB) m/z (relative intensity) 301 (M^+ , 10), 256 (93), 228 (100); HRMS calcd for $\text{C}_{17}\text{H}_{20}\text{NO}_4$ (MH^+): 302.1392, found: 302.1407.

General Procedure for the Conjugate Additions of Substituted Cyclohexa-2,5-dienone Monoacetals with Nucleophiles. The substituted cyclohexa-2,5-dienone monoacetal (1.0 equiv) was dissolved in THF (5 mL) followed by the addition of the indicated nucleophile (1.0 equiv) and potassium *tert*-butoxide (0.15 equiv). The reaction mixture was stirred at room temperature for 10 h at which time it was quenched with saturated aqueous solution of NH_4Cl (1 mL) and diluted with H_2O (20 mL). The aqueous

layer was then extracted with ethyl acetate (3×20 mL) and the organic extracts were combined and concentrated to an oil which was chromatographed to afford the indicated products.

Methyl 2-(diethyl malonyl)-3-ethoxy-6-hydroxybenzoate (5). The crude ^1H NMR showed mostly the aromatized product indicated along with a trace amount of unstable conjugate addition adduct. Thus, the residue was dissolved in benzene (5 mL) and *p*-toluenesulfonic acid (1.2 equiv) was added and the mixture heated to reflux according to the method described by Parker and Kang.⁵ After 30 min, the reaction was cooled and quenched with a saturated aqueous solution of NaHCO_3 (25 mL) and extracted with ethyl acetate (3×20 mL). The organic extracts were combined, dried over MgSO_4 , filtered and concentrated to an oil, which was chromatographed (10-20% EtOAc/hex) to afford a clear oil (71%). $R_f = 0.26$ (20% EtOAc/hex); IR (neat) 2975, 1740, 1620 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 10.27 (s, 1H), 7.14 (d, $J = 9.1$ Hz, 1H), 6.98 (d, $J = 9.1$ Hz, 1H), 5.53 (s, 1H), 4.21 (q, $J = 7.2$ Hz, 4H), 4.02 (q, $J = 7.0$ Hz, 2H), 3.85 (s, 3H), 1.35 (t, $J = 7.0$ Hz, 3H), 1.26 (t, $J = 7.1$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.4, 160.1, 155.8, 150.3, 123.5, 121.6, 118.3, 113.4, 66.5, 61.4, 51.9, 50.8, 14.8, 14.1; MS (FAB) m/z (relative intensity) 354 (M^+ , 100), 323 (60); HRMS calcd for $\text{C}_{17}\text{H}_{23}\text{O}_8$ (MH^+): 355.1393, found: 355.1398.

Methyl 5,5-diethoxy-6-(3-ethoxy-1-nitro-3-oxopropyl)-2-hydroxycyclohexa-1,3-dienecarboxylate (6). For this reaction, 1.2 equiv ethyl 3-nitropropionate and 1.0 equiv potassium *tert*-butoxide were used. A diastereomeric ratio of ca. 10:1 was estimated from ^1H NMR analysis of the crude reaction mixture. Yellow oil (100% crude) following workup. Crystallization from Et_2O /hexane (1:1) at 0 °C gave crystals suitable for X-ray crystallographic analysis. $R_f = 0.45$ (40% EtOAc /hex). **Major isomer:** ^1H NMR (400 MHz, CDCl_3) δ 6.34 (d, $J = 10.2$ Hz, 1H), 6.12 (d, $J = 10.1$ Hz, 1H), 4.84 (td, $J = 10.5, 2.6$ Hz, 1H), 4.37 (m, 1H), 4.15 (m, 4H), 3.56 (m, 3H), 3.46 (m, 3H), 3.09 (dd, $J = 17.4, 10.5$ Hz, 1H), 2.51 (dd, $J = 16.2, 2.5$ Hz, 1H), 1.28 (m, 6H), 1.14 (q, $J = 6.9$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.3, 166.4, 135.8, 125.6, 97.9, 94.0, 81.8, 61.4, 56.8, 52.3, 40.1, 32.7, 31.1, 15.1, 14.4, 14.0. **Minor isomer** (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 6.32 (d, $J = 10.1$ Hz, 1H), 5.05 (td, $J = 10.5, 2.6$ Hz, 1H), 3.95 (m, 1H), 3.66 (s, 3H), 2.60 (dd, $J = 17.4, 7.2$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.6, 165.9, 139.7, 126.8.

Methyl 3-ethoxy-2-(1'-nitro-2'-carboethoxyethyl)-6-hydroxybenzoate. The unstable adducts **6** were treated with *p*-toluenesulfonic acid according to the method described by Parker and Kang⁶ to give an orange oil (41% over two steps). $R_f = 0.40$ (40% EtOAc /hex); IR (neat) 2975, 1730, 1560 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 10.18 (s, 1H), 7.12 (d, $J = 9.2$ Hz, 1H), 7.05 (d, $J = 9.2$ Hz, 1H), 6.80 (dd, $J = 9.2, 2.8$ Hz, 1H), 4.25 (m, 2H), 4.06 (m, 1H), 3.98 (s, 3H), 3.87 (m, 1H), 3.79 (dd, $J = 17.5, 8.3$ Hz,

1H), 2.74 (dd, $J = 17.5, 2.8$ Hz, 1H), 1.32 (q, $J = 6.9$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.2, 169.6, 155.4, 150.0, 123.6, 120.4, 119.7, 112.7, 80.1, 65.3, 61.3, 52.9, 36.9, 30.9, 14.3, 14.2; MS (FAB) m/z (relative intensity) 341 (M^+ , 100); HRMS calcd for $\text{C}_{15}\text{H}_{19}\text{NO}_8$: 341.1111, found: 341.1097.

4-Ethyl 6-methyl 9,9-diethoxy-3-methyl-7-oxo-2-oxa-bicyclo[3.3.1]non-3-ene-4,6-dicarboxylate (7). Isolated as a mixture of keto and enol tautomers. Clear oil (84%). $R_f = 0.30$ (20% EtOAc/hex); IR (neat) 2975, 1740, 1620, cm^{-1} ; MS (FAB) m/z (relative intensity) 370 (M^+ , 15), 325 (100); HRMS calcd for $\text{C}_{18}\text{H}_{27}\text{O}_8$ (MH^+): 371.1706, found 371.1697. **Keto tautomer:** ^1H NMR (400 MHz, CDCl_3) δ 4.56 (q, $J = 3.7$ Hz, 1H), 4.19-4.11 (m, 2H), 4.01 (d, $J = 2.9$ Hz, 1H), 3.74 (s, 3H), 3.66-3.43 (m, 4H), 3.00 (dd, $J = 16.6, 4.1$ Hz, 1H), 2.66 (td, $J = 16.5, 2.4$ Hz, 1H), 2.59 (dd, $J = 18.1, 1.3$ Hz, 1H), 2.16 (s, 3H), 1.30 (t, $J = 7.1$ Hz, 3H), 1.20-1.10 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 203.3, 171.6, 169.3, 167.6, 166.2, 101.7, 96.4, 74.9, 59.8, 56.8, 56.6, 52.1, 37.8, 33.0, 19.7, 15.3, 15.0, 14.2. **Enol tautomer** (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ (s, 12.04, 1H), 4.45 (m, 1H), 3.85 (m, 1H), 3.40 (t, $J = 2.1$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.6, 161.5, 106.4.

Methyl 9,9-diethoxy-7-methyl-6-nitro-3-oxo-8-oxa-bicyclo[3.3.1]non-6-ene-4-carboxylate (8). White solid (52%). $R_f = 0.51$ (40% EtOAc/hex); mp 122-123 $^{\circ}\text{C}$. IR

(neat) 2975, 1660 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 12.17 (s, 1H), 4.59-4.58 (m, 1H), 4.52 (d, $J = 2.7$, 1H), 3.80 (s, 3H), 3.63-3.55 (m, 3H), 3.46-3.42 (m, 1H), 2.92 (dd, $J = 19.7$, 5.6 Hz, 1H), 2.65 (d, $J = 18.7$ Hz, 1H), 2.36 (s, 3H), 1.16 (hex, $J = 3.4$, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.6, 171.5, 162.3, 133.3, 100.0, 96.0, 71.4, 57.3, 56.9, 51.9, 37.3, 34.5, 20.7, 15.2, 15.1; MS (FAB) m/z (relative intensity) 344 (MH^+ , 22), 307 (37), 298 (100); HRMS calcd for $\text{C}_{15}\text{H}_{21}\text{NO}_8\text{Na}$ ($\text{M}^+ + \text{Na}$): 366.1165, found: 366.1165.

Methyl 5,5-diethoxy-6-(3-ethoxy-1-nitro-3-oxopropyl)-1-methyl-2-oxocyclohex-3-enecarboxylate (10). Quinone monoacetal **1** (59 mg), ethyl 3-nitropropionate (36 mg), and potassium *tert*-butoxide (28 mg) were dissolved in THF (3 mL) at room temperature. After 20 min, MeI (178 mg, 80 μL) was added. After 10 h, the reaction was subjected to the usual quench, workup and purification protocol, which provided a clear oil (45%). Crystallization from hexane/ Et_2O (3:1) gave crystals suitable for X-ray crystallographic analysis. A diastereomeric ratio of greater than 10:1 was estimated from ^1H NMR analysis of the crude reaction mixture. $R_f = 0.53$ (40% EtOAc/hex); IR (neat) 2975, 1730, 1690, cm^{-1} ; MS (TOF) m/z (relative intensity) 424 ($\text{M}^+ + \text{Na}$, 100); HRMS calcd for $\text{C}_{18}\text{H}_{27}\text{N}_1\text{O}_8\text{Na}$ ($\text{M}^+ + \text{Na}$): 424.1584, found 424.1583. **Major isomer:** ^1H NMR (400 MHz, CDCl_3) δ 7.00 (d, $J = 10.6$ Hz, 1H), 6.24 (d, $J = 10.6$ Hz, 1H), 5.30 (d, $J = 10.8$ Hz, 1H), 4.20 (m, 2H), 3.79 (dd, $J = 8.64$, 1.5 Hz, 1H), 3.65 (s, 3H), 3.58 (d, $J = 1.5$ Hz, 1H), 3.45 (m, 3H), 3.27 (broad d, $J = 17.9$ Hz, 1H), 3.18 (dd, $J = 7.0$, 1.8 Hz, 1H), 1.71 (s, 3H), 1.28 (t, $J = 7.0$ Hz, 3H), 1.15 (q, $J = 7.0$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 194.3, 172.2, 171.3, 144.8, 130.7, 97.6, 82.3, 61.0, 59.1, 58.7, 55.7, 52.5, 51.5, 34.1, 20.6, 15.2, 14.9, 14.1.

Ethyl 5-ethoxy-8-hydroxy-1,3-dioxo-2-phenyl-1,2,3,4-tetrahydroisoquinoline-4-carboxylate (11). The unstable adduct was treated with *p*-toluenesulfonic acid according to the method described by Parker and Kang⁶ to provide a yellow semi-solid (77%). $R_f = 0.75$ (40% EtOAc/hex); IR (neat) 2970, 1720, 1660 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 10.97 (s, 1H), 7.53 (m, 3H), 7.23 (m, 3H), 7.04 (dd, $J = 9.2, 0.5$ Hz, 1H), 5.19 (d, $J = 0.5$ Hz, 1H), 4.28 (q, $J = 7.1$ Hz, 2H), 4.09 (m, 2H), 1.44 (t, $J = 7.0$ Hz, 3H), 1.33 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.3, 166.5, 166.5, 155.7, 148.0, 133.8, 129.5, 129.2, 128.4, 120.5, 120.4, 118.0, 109.1, 64.8, 62.6, 50.5, 14.7, 14.1; MS (FAB) m/z (relative intensity) 370 (MH^+ , 100); HRMS calcd for $\text{C}_{20}\text{H}_{19}\text{NO}_6$: 369.1212, found: 369.1212.

3-Ethoxy-6-hydroxy-2-(1'-nitro-2'-carboethoxyethyl)-N-phenylbenzamide (12). *tert*-Butanol was used. The unstable adduct was treated with *p*-toluenesulfonic acid according to the method described by Parker and Kang⁶ which provided white crystals (57%) following chromatography. $R_f = 0.23$ (40% EtOAc/hex); mp 154-156 $^{\circ}\text{C}$; IR (neat) 3310 (br), 1730, 1550 cm^{-1} ; ^1H NMR (400 MHz, acetone- d_6) δ 9.58 (d, 1H), 8.74 (s, 1H), 7.85 (d, $J = 7.8$ Hz, 2H), 7.38 (t, $J = 7.7$ Hz, 2H), 7.14 (t, $J = 7.4$ Hz, 1H), 7.04 (s, 2H), 6.24 (dd, $J = 10.1, 2.5$ Hz, 1H), 4.18 (q, $J = 7.3$ Hz, 2H), 4.10 (m, 1H), 3.93 (m, 1H), 3.77 (dd, $J = 17.5, 10.2$ Hz, 1H), 2.91 (m, 1H) 1.30 (t, $J = 7.0$ Hz, 3H), 1.24 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (100 MHz, acetone- d_6) δ 169.7, 164.3, 150.1, 147.8, 139.2, 128.7, 126.5, 123.9, 122.8, 119.7, 117.6, 115.0, 80.7, 64.6, 60.6, 36.4, 13.8, 13.5; MS (FAB)

m/z (relative intensity) 403 (MH^+ , 25), 356 (100); HRMS calcd for $\text{C}_{20}\text{H}_{23}\text{N}_2\text{O}_7$ (MH^+): 403.1505, found: 403.1495.

Ethyl 9,9-diethoxy-3-methyl-7-oxo-6-(phenylcarbamoyl)-2-oxabicyclo[3.3.1]non-3-ene-4-carboxylate (13). Light yellow crystals (42%) suitable for X-ray crystallographic analysis were obtained from recrystallization in hexanes/ CHCl_3 (3:1). R_f = 0.58 (40% EtOAc/hex); mp 125-127 °C; IR (KBr pellet) 2975, 1650 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 14.78 (s, 1H), 10.09 (s, 1H), 7.69 (d, J = 8.1 Hz, 2H), 7.35 (t, J = 7.8 Hz, 2H), 7.11 (t, J = 7.4 Hz, 1H), 4.58 (m, 1H), 4.34-4.30 (m, 2H), 3.80 (d, J = 2.5 Hz, 1H), 3.62-3.47 (m, 4H), 2.91 (dd, J = 19.2, 5.2 Hz, 1H), 2.67 (d, J = 19.2 Hz, 1H), 2.24 (s, 3H), 1.36 (t, J = 7.1 Hz, 3H), 1.18 (q, J = 6.8 Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 170.8, 170.6, 170.5, 165.3, 138.4, 128.7, 123.8, 120.4, 105.3, 101.7, 96.5, 71.0, 61.3, 57.0, 56.4, 38.0, 33.2, 21.0, 15.3, 15.1, 14.3; MS (FAB) m/z (relative intensity) 432 (MH^+ , 65), 386 (100); HRMS calcd for $\text{C}_{23}\text{H}_{30}\text{NO}_7$ (MH^+): 432.2022, found: 432.2005.

5-(Diethylmalonyl)-4,4-dimethoxy-2-(2'-nitrophenyl)-2-cyclohexenone (14). Yellow oil (ca. 100%). R_f = 0.34 (40% EtOAc/hex); IR (neat) 2930, 1730, 1530 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 8.11 (dd, J = 8.1, 0.9 Hz, 1H), 7.67 (dt, J = 7.5, 1.0 Hz, 1H), 7.56 (dt, J = 7.9, 1.3 Hz, 1H), 7.33 (dd, J = 7.4, 1.3 Hz, 1H), 6.85 (d, J = 1.0 Hz, 1H), 4.22 (m, 4H), 3.82 (d, J = 7.6 Hz, 1H), 3.77 (t, J = 6.4 Hz, 2H), 3.49 (t, J = 7.0 Hz, 1H),

3.40 (s, 3H), 3.36 (s, 3H), 2.98 (dd, $J = 17.5, 4.9$ Hz, 1H) 2.78 (dd, $J = 17.5, 5.7$ Hz, 1H), 1.87 (p, $J = 3.3$ Hz, 2H), 1.30 (t; $J = 7.1$ Hz, 3H), 1.22 (t; $J = 7.1$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 194.3, 168.7, 168.5, 148.9, 142.2, 142.0, 134.0, 131.9, 130.8, 130.1, 124.8, 98.8, 62.0, 62.0, 51.7, 50.9, 50.0, 40.8, 38.7, 14.4, 14.3; MS (FAB) m/z (relative intensity) 435 (M^+ , 20), 330 (100); HRMS calcd for $\text{C}_{21}\text{H}_{29}\text{N}_2\text{O}_9$ ($\text{M}^+ + \text{NH}_4$): 453.1873, found: 453.1864.

Ethyl 9,9-dimethoxy-6-(2-nitrophenyl)-3,7-dioxobicyclo[3.3.1]nonane-2-carboxylate (15). Light yellow crystals (71%) suitable for X-ray crystallographic analysis were obtained from recrystallization in hexanes/ CHCl_3 (5:1). $R_f = 0.63$ (30% EtOAc/hex); IR (KBr pellet) 2955, 1720, 1655 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.91 (dd, $J = 8.2, 0.9$ Hz, 1H), 7.65 (dt, $J = 7.7, 1.3$ Hz, 1H), 7.46 (dt, $J = 7.7, 1.3$ Hz, 1H), 7.28 (m, 1H), 4.73 (d, $J = 5.6$ Hz, 1H), 3.51 (s, 3H), 3.29 (s, 3H), 3.09 (m, 1H), 2.94 (dd, $J = 15.5, 4.5$ Hz, 1H), 2.61-2.52 (m, 2H), 2.30 (d, $J = 18.4$ Hz, 3H), 1.33 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 207.0, 171.1, 170.7, 150.2, 134.0, 132.0, 129.8, 128.2, 124.8, 99.3, 98.8, 60.8, 51.6, 48.6, 48.2, 43.7, 37.0, 34.8, 29.9, 14.2; MS (CI) m/z (relative intensity) 423 ($\text{M}^+ + \text{NH}_4$, 65), 374 (100); HRMS calcd for $\text{C}_{20}\text{H}_{27}\text{N}_2\text{O}_8$ ($\text{M}^+ + \text{NH}_4$): 423.1767, found: 423.1794.

6,6-Diethoxy-5-diethyl malonyl-3-oxo-N-phenyl-1-cyclohexenecarboxamide (16). Clear oil (58%). $R_f = 0.53$ (40% EtOAc/hex); IR (neat) 2975, 1675 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 9.72 (s, 1H), 7.63 (d, $J = 7.6$ Hz, 2H), 7.37 (t, $J = 7.6$ Hz, 2H), 7.16 (t, $J = 7.4$ Hz, 1H), 6.76 (s, 1H), 4.29 (m, 1H), 4.18-4.00 (m, 3H), 3.80 (m, 1H), 3.68 (d, $J = 4.8$ Hz, 1H), 3.65-3.50 (m, 4H), 3.06 (dd, $J = 18.8, 6.0$ Hz, 1H), 2.81 (d, $J = 18.8$ Hz, 1H), 1.39 (t, $J = 7.0$ Hz, 3H), 1.24 (t, $J = 7.1$ Hz, 3H), 1.18 (dt, $J = 7.1, 2.4$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 196.7, 168.6, 168.1, 162.3, 149.0, 137.9, 134.1, 129.2, 124.7, 119.8, 99.9, 62.1, 59.5, 56.7, 41.3, 37.6, 15.0, 14.9, 13.9, 13.6; MS (FAB) m/z (relative intensity) 461 (M^+ , 31), 416 (100); HRMS calcd for $\text{C}_{24}\text{H}_{32}\text{NO}_8$ (MH^+): 462.2128, found: 462.2122.

Ethyl 9,9-diethoxy-3,7-dioxo-5-(phenylcarbamoyl)bicyclo[3.3.1]nonane-2-carboxylate (17). White crystals (69%) suitable for X-ray crystallographic analysis were obtained from recrystallization in hexanes/ CHCl_3 (5:1). $R_f = 0.45$ (40% EtOAc/hex); mp 152-154 $^\circ\text{C}$; IR (neat) 3330, 2975, 1720, 1660 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 9.59 (s, 1H), 7.51 (dd, $J = 8.7, 1.1$ Hz, 2H), 7.36 (t, $J = 7.5$, 2H), 7.14 (t, $J = 3.4$ Hz, 1H), 4.34-4.18 (m, 2H), 3.80-3.71 (m, 3H), 3.62-3.57 (m, 3H), 3.41 (q, $J = 2.7$ Hz, 1H), 3.1 (dd, $J = 19.3, 2.0$ Hz, 1H), 3.02 (dd, $J = 16.2, 2.0$ Hz, 1H), 2.91 (dd, $J = 15.0, 4.6$ Hz, 1H), 2.62 (d, $J = 2.9$ Hz, 1H) 2.58 (d, $J = 2.5$ Hz, 1H) 2.41 (td, $J = 15.0, 2.6$ Hz, 1H)

1.37-1.30 (m, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 208.0, 172.1, 170.6, 169.6, 138.0, 129.2, 124.3, 119.4, 101.0, 98.0, 61.0, 58.0, 57.5, 50.8, 49.2, 43.2, 40.1, 37.4, 15.5, 15.3, 14.3; MS (FAB) m/z (relative intensity) 432 (MH^+ , 20), 385 (39), 340 (100); HRMS calcd for $\text{C}_{23}\text{H}_{30}\text{NO}_7$ (MH^+): 432.2022, found: 432.2029.

References:

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2. Herz, J. E.; Mantecon, R. E. *Organic Preparations and Procedures International* **1972**, *4*, 123-8.
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4. Lappin, G. R.; Moore, L. D., Jr. U.S. Patent 2,835,648, 1958.
5. Parker, K. A.; Kang, S.-K. *J. Org. Chem.* **1980**, *45*, 1218-24.

Current Data Parameters
NAME sg11_179
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040420
Time 9.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8009.615 Hz
FIDRES 0.183358 Hz
AQ 2.7264309 sec
RG 32
DN 83.200 usec
DE 6.00 usec
TE 298.2 K
D1 0.0300000 sec
HOREST 0.0000000 sec
HOMO 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320000 MHz

F1 - Acquisition parameters
AQ 2
TD 65536
SFO1 500.1320000 MHz
FIDRES 23.475060 Hz
SW 12.616 ppm
FREQZ 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 1624
AC2 OF
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CA 20.00 cm
CY 10.00 cm
FIP 11.000 ppm
F1 5501.43 Hz
F2 -1.000 ppm
F2 -500.13 Hz
PRCH 0.60000 ppm/cm
NCH 300.07800 Hz/cm

SI-16

Current Data Parameters
NAME sg11_179
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040420
Time 9.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 150
DS 4
SWH 31440.541 Hz
FIDRES 0.478000 Hz
AQ 1.0400000 sec
RG 18380.4
DN 13.500 usec
DE 6.00 usec
TE 298.2 K
D1 0.1500000 sec
D11 0.0300000 sec
DELTA 0.0500000 sec
HOREST 0.0000000 sec
HOMO 0.0150000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.50 usec
PL1 5.00 dB
SFO1 125.7673000 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
AC2 DM
PCPDZ 30.00 usec
PL2 -1.00 dB
PL12 15.00 dB
PL13 30.00 dB
SFO2 500.1375000 MHz

F1 - Acquisition parameters
AQ 2
TD 256
SFO1 500.1320000 MHz
FIDRES 23.475060 Hz
SW 12.616 ppm
FREQZ 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 1624
AC2 OF
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CA 20.00 cm
CY 10.00 cm
FIP 240.517 ppm
F1 20498.43 Hz
F2 -1.330 ppm
F2 -125.7673 Hz
PRCH 1577.37900 Hz/cm

1

Current Data Parameters
NAME 99_111_88a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041008
Time 12.02
INSTRUM spect
PROBHD 5 mm CPDQX 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 9
DN 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec
MCREST 0.00000000 sec
MCNMR 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 15.00 usec
PL1 -5.10 dB
SFO1 500.1320005 MHz

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

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.008 ppm
F1 5005.33 Hz
F2P -2.008 ppm
F2 -1004.28 Hz
PPMCH 0.60081 ppm/cm
HZCH 300.48077 Hz/cm

SI-17

Current Data Parameters
NAME 99_111_88a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041008
Time 12.04
INSTRUM spect
PROBHD 5 mm CPDQX 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 31446.541 Hz
FIDRES 8.479830 Hz
AQ 1.842083 sec
RG 11588.2
DN 15.288 usec
DE 5.00 usec
TE 300.0 K
D1 0.15000000 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCNMR 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.00 usec
PL1 -5.20 dB
SFO1 125.767800 MHz

***** CHANNEL f2 *****
CPUPROG waltz16
NUC2 1H
P2 10.00 usec
PL2 -5.10 dB
PL12 10.00 dB
PL13 10.00 dB
SFO2 500.1320000 MHz

F1 - Acquisition parameters
NS 2
TD 256
SF01 500.1320 MHz
FIDRES 23.479860 Hz
SW 31446.541 Hz
FREQ01 uncalibrated

F2 - Processing parameters
SI 65536
SF 125.757800 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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

1D NMR plot parameters
CX 20.00 cm
CY 0.80 cm
F1P 300.000 ppm
F1 29151.50 Hz
F2P -5.000 ppm
F2 -600.710 Hz
PPMCH 10.25000 ppm/cm
HZCH 1289.81750 Hz/cm

Current Data Parameters
 NAME sg_111_17
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040819
 Time 9.41
 INSTRUM spect
 PROBRD 5 mm BBO BB-H
 PULPROG zgpg30
 TD 32768
 SOLVENT Aceton
 NS 16
 DS 2
 SWH 6009.515 Hz
 FIDRES 0.183399 Hz
 AQ 2.7284309 sec
 RG 128
 DW 83.200 usec
 DE 6.00 usec
 TE 297.2 K
 D1 0.03000000 sec
 HOREST 0.00000000 sec
 MCNRM 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.70 usec
 PL1 -4.00 dB
 SFO1 500.132005 MHz

F1 - Acquisition Parameters
 MD 2
 TD 256
 SFO1 500.1325 MHz
 FIDRES 23.475000 Hz
 SW 12.018 ppm
 FWHM undefined

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

F1 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 NQW 0
 SSB 0
 LB 0.30 Hz
 GB 0

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 10.000 ppm
 F1 500.13 Hz
 F2P -2.000 ppm
 F2 -1004.29 Hz
 PRMCH 0.00001 ppm/c
 HZCM 300.48080 Hz/cm

SI-18

Current Data Parameters
 NAME sg_111_17
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040819
 Time 16.18
 INSTRUM spect
 PROBRD 5 mm BBO BB-H
 PULPROG zgpg30
 TD 65536
 SOLVENT Aceton
 NS 148
 DS 4
 SWH 31446.541 Hz
 FIDRES 0.479836 Hz
 AQ 1.0420803 sec
 RG 10390.4
 DW 15.300 usec
 DE 6.00 usec
 TE 297.2 K
 D1 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 HOREST 0.00000000 sec
 MCNRM 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 6.50 usec
 PL1 5.00 dB
 SFO1 125.7697360 MHz

***** CHANNEL f2 *****
 CPOPRG2 waltz16
 NUC2 1H
 PPRG2 85.00 usec
 PL2 -4.00 dB
 PL12 19.00 dB
 PL13 30.00 dB
 SFO2 500.1325000 MHz

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

1D NMR plot parameters
 CX 20.00 cm
 CY 12.00 cm
 F1P 242.517 ppm
 F1 30498.43 Hz
 F2P -7.530 ppm
 F2 -948.11 Hz
 PRMCH 12.50282 ppm/c
 HZCM 1572.32703 Hz/cm

2

Current Data Parameters
NAME khong
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041112
Time 12.03
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 114
OW 104.400 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
MCPRST 0.00000000 sec
MCWRK 0.01500000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 10.985 ppm
F1 4395.28 Hz
F2 -0.985 ppm
F2 -393.59 Hz
PRMCA 0.58946 ppm/cm
HZCH 239.46359 Hz/cm

SI-19

Current Data Parameters
NAME khong
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040818
Time 18.28
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 128
DS 4
SWH 31448.541 Hz
FIDRES 0.478928 Hz
AQ 1.0400000 sec
RG 16290.4
OW 10.500 usec
DE 6.00 usec
TE 296.6 K
D1 0.15000000 sec
MCPRST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 8.50 usec
PL1 1.50 dB
SFO1 125.7687500 MHz

***** CHANNEL f2 *****
NUC2 1H
P2 99.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 30.00 dB
SFO2 500.1325000 MHz

F1 - Acquisition parameters
NO 2
TD 256
SF01 500.1325000 MHz
FIDRES 22.4725000 Hz
SW 12.016 ppm
FWDK 1 usec

F2 - Processing parameters
SI 1024
SF 125.7578000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 242.517 ppm
F1 30490.43 Hz
F2 -17.336 ppm
F2 -948.11 Hz
PRMCA 12.50082 ppm/cm
HZCH 1072.32762 Hz/cm

Current Data Parameters
NAME 98_11_217
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040517
Time 17.42
INSTRUM spect
PROBHD 5 mm BBO BB-H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.515 Hz
FIDRES 0.182395 Hz
AQ 7.7264329 sec
RG 203.2
DW 83.200 usec
DE 6.00 usec
TE 298.2 K
D1 0.03000000 sec
HOREST 0.00000000 sec
HCHW 0.01500000 sec

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

F1 - Acquisition parameters
NUC 1
TD 256
SFO1 500.1325 MHz
FIDRES 23.473060 Hz
SW 12.016 ppm
FNUC1 undefined

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

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

10 MHz plot parameters
C1 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 5000.33 Hz
F2P -2.000 ppm
F2 -1004.29 Hz
PPHCH 0.60081 ppm/cm
H2CH 300.46880 Hz/cm

SI-20

Current Data Parameters
NAME 98_11_217
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040517
Time 17.47
INSTRUM spect
PROBHD 5 mm BBO BB-H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 372
DS 4
SWH 21446.541 Hz
FIDRES 0.479636 Hz
AQ 1.0409843 sec
RG 2084.3
DW 15.500 usec
DE 8.50 usec
TE 298.2 K
D1 0.15000001 sec
D11 0.03000000 sec
DELTA 0.05000000 sec
HOREST 0.00000000 sec
HCHW 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
SFO1 125.7607360 MHz

===== CHANNEL f2 =====
CPROG2 waltz16
NUC2 1H
P1P2 35.00 usec
PL2 -4.00 dB
PL12 12.00 dB
PL13 20.00 dB
SFO2 500.1325000 MHz

F1 - Acquisition parameters
NUC 13C
TD 256
SFO1 500.1325 MHz
FIDRES 22.473060 Hz
SW 12.016 ppm
FNUC2 undefined

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

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

10 MHz plot parameters
C1 20.00 cm
CY 12.00 cm
F1P 215.834 ppm
F1 27056.42 Hz
F2P -20.100 ppm
F2 -1004.29 Hz
PPHCH 12.50242 ppm/cm
H2CH 1332.32782 Hz/cm

Current Data Parameters
NAME 99_11_1426
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040420
Time 9.50
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 1930
FIDRES 32768
SOLVENT DMSO
NS 16
DS 2
SWH 6009.815 Hz
FIDRES 0.182379 Hz
AQ 2.7264301 sec
RG 228.1
DM 83.200 usec
DE 6.00 usec
TE 298.2 K
D1 0.03000000 sec
HOREST 0.00000000 sec
HOREN 0.01000000 sec

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

F1 - Acquisition parameters
AQ 2
TD 256
SFO1 500.1325 MHz
FIDRES 23.4750000 Hz
SW 12.016 ppm
FREQDE 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 EM
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 10.000 ppm
F1 5005.33 Hz
F2 -2.008 ppm
F2 -1004.25 Hz
F2 0.50051 ppm/cm
PCHN 300.40000 Hz/cm
H2CN

SI-21

Current Data Parameters
NAME 99_11_1426
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040420
Time 9.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 1930
FIDRES 32768
SOLVENT DMSO
NS 16
DS 2
SWH 31448.541 Hz
FIDRES 8.479830 Hz
AQ 1.0400000 sec
RG 1098.1
DM 15.500 usec
DE 6.00 usec
TE 298.2 K
D1 0.15000000 sec
HOREST 0.03000000 sec
HOREN 0.05000000 sec
HOREN 0.00000000 sec
HOREN 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 0.00 dB
SFO1 125.7570000 MHz

===== CHANNEL f2 =====
NUC2 1H
P2 55.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 30.00 dB
SFO2 500.1325000 MHz

F1 - Acquisition parameters
AQ 2
TD 256
SFO1 500.1325 MHz
FIDRES 23.4750000 Hz
SW 12.016 ppm
FREQDE undefined

F2 - Processing parameters
SI 32768
SF 125.7570000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 242.511 ppm
F1 30498.43 Hz
F2 -1.326 ppm
F2 -948.11 Hz
PCHN 12.30700 ppm/cm
H2CN 1512.34703 Hz/cm

Current Data Parameters
NAME sg_III_102
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041210
Time 14.54
INSTRUM spect
PROBHD 5 mm QNP 1H/15
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 6.00 usec
TE 297.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCPRK 0.01500000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCH 0.59846 ppm/cm
HZCH 239.46359 Hz/cm

SI-22

Current Data Parameters
NAME sg_III_102
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041213
Time 8.29
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 198
DS 4
SWH 23560.814 Hz
FIDRES 0.365918 Hz
AQ 1.3654756 sec
RG 2048
DM 20.850 usec
DE 6.00 usec
TE 295.5 K
D1 0.15000000 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCPRK 0.01500000 sec

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

===== CHANNEL f2 =====
CHARGE well115
NUC2 1H
PCPD2 88.40 usec
PL2 -3.00 dB
PL12 19.56 dB
PL13 120.00 dB
SFO2 400.1316605 MHz

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

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P -3.000 ppm
F2 -503.06 Hz
PPMCH 11.00000 ppm/cm
HZCH 1106.74048 Hz/cm

4

Current Data Parameters
NAME sg_11_201a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040504
Time 9.47
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 5.50 usec
TE 1809.2 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 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 10.00 cm
FLP 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCM 0.59846 ppm/cm
HZCM 239.46362 Hz/cm

SI-23

Current Data Parameters
NAME sg_11_201a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040504
Time 9.52
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 125
DS 4
SWH 23149.148 Hz
FIDRES 0.332113 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 5.50 usec
TE 1809.2 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 12.30 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 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 12.00 cm
FLP 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

5

Current Data Parameters
NAME sg_111_77a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041112
Time 12.46
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.145157 Hz
AQ 3.4210281 sec
RG 101.6
DN 104.400 usec
DE 6.00 usec
TE 295.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWPM 0.01500000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 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

SI-24

Current Data Parameters
NAME sg_111_77a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041112
Time 12.55
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 196
DS 4
SWH 22980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DN 25.850 usec
DE 8.00 usec
TE 297.0 K
D1 0.15000001 sec
D11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWPM 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CROSSP2 waltz16
NUC2 1H
PCPD2 96.40 usec
PL2 -3.00 dB
PL12 19.96 dB
PL13 120.00 dB
SFO2 400.1315003 MHz

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 215.900 ppm
F1 21631.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCH 11.00000 ppm/cm
HZCM 1106.74048 Hz/cm

Current Data Parameters
NAME adw-VI-021
EXTNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20030222
Time 14.47
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
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 11.67 cm
FIP 10.000 ppm
P1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCH 0.55000 ppm/cm
HZCH 220.07150 Hz/cm

SI-25

Current Data Parameters
NAME adw-VI-021
EXTNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20030222
Time 14.52
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 543
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 645.1
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 100.6232933 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 100.6127763 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.62 cm
FIP 220.000 ppm
P1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCH 11.50000 ppm/cm
HZCH 1157.04700 Hz/cm

Current Data Parameters
NAME sg_III_98a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041130
Time 9.49
INSTRUM spect
PROBHD 5 mm QNP 1H/15
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 6.00 usec
TE 296.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCHW 0.01500000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.985 ppm
F2P 4395.28 Hz
F2 -393.90 Hz
PPMCH 0.59846 ppm/cm
HZCH 239.46359 Hz/cm

SI-26

Current Data Parameters
NAME sg_III_98a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041130
Time 9.55
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 262
DS 4
SWH 23396.814 Hz
FIDRES 0.363918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 296.3 K
D1 0.15000001 sec
D11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCHW 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.40 usec
PL2 -3.00 dB
PL12 15.36 dB
PL13 120.00 dB
SFO2 400.1316002 MHz

F2 - Processing parameters
SI 32768
SF 100.6127650 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 216.000 ppm
F2P -5.000 ppm
F2 -503.06 Hz
PPMCH 11.00000 ppm/cm
HZCH 1106.74048 Hz/cm

keto

enol

7

Current Data Parameters
NAME sg_III_81ae
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041119
Time 13.04
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6377.551 Hz
FIDRES 0.194627 Hz
AQ 2.5690513 sec
RG 32
DN 78.400 usec
DE 6.00 usec
TE 297.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 7.05 usec
PL1 -3.00 dB
SFO1 400.1324008 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 10.00 cm
F1P 13.959 ppm
F1 5589.56 Hz
F2P -1.969 ppm
F2 -787.99 Hz
PPOW 0.78653 ppm/cm
HZCH 318.67753 Hz/cm

SI-27

Current Data Parameters
NAME sg_III_76a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041112
Time 12.32
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 377
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DN 20.850 usec
DE 6.00 usec
TE 296.9 K
D1 0.15000001 sec
D11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

----- CHANNEL f2 -----
CPOPRG2 waltz16
NUC2 1H
PCPD2 99.40 usec
PL2 -3.00 dB
PL12 19.98 dB
PL13 120.00 dB
SFO2 400.1316005 MHz

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

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 215.000 ppm
F1 21831.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPOW 11.00000 ppm/cm
HZCH 1106.74048 Hz/cm

8

Current Data Parameters
NAME sg_III_120
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041222
Time 13.43
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5582.841 Hz
FIDRES 0.170880 Hz
AQ 2.9235092 sec
RG 104
DM 89.400 usec
DE 6.00 usec
TE 297.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCHMK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 7.05 usec
PL1 -3.00 dB
SFO1 400.1324008 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 10.00 cm
FIP 12.989 ppm
F1 5197.20 Hz
F2P -0.989 ppm
F2 -395.64 Hz
PPMCH 0.69888 ppm/cm
HZCH 279.64206 Hz/cm

SI-28

Current Data Parameters
NAME sg_III_120
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041222
Time 13.36
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 195
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3654796 sec
RG 2048
DM 20.650 usec
DE 6.00 usec
TE 297.0 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCHMK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6262598 MHz

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

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

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
FIP 215.000 ppm
F1 21631.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCH 11.00000 ppm/cm
HZCH 1106.74048 Hz/cm

ca 10:1

10

Current Data Parameters
NAME sg_III_164a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050215
Time 10.01
INSTRUM spect
PROBHD 5 mm GNP 1H/15
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 71.8
DM 104.400 usec
DE 6.00 usec
TE 296.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWPK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.05 usec
PL1 -3.00 dB
SF01 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 10.00 cm
F1P 10.965 ppm
F1 4395.28 Hz
F2P -0.585 ppm
F2 -393.99 Hz
PPHCH 0.59846 ppm/cm
HZCH 239.46359 Hz/cm

SI-29

Current Data Parameters
NAME sg_III_164a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050215
Time 10.07
INSTRUM spect
PROBHD 5 mm GNP 1H/15
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 264
DS 4
SWH 22380.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DM 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 0.15000001 sec
D11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWPK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SF01 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 mltz16
NUC2 1H
PCPD2 98.40 usec
PL2 -3.00 dB
PL12 19.36 dB
PL13 120.60 dB
SF02 400.1316005 MHz

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 219.169 ppm
F1 22051.21 Hz
F2P -19.179 ppm
F2 -1929.61 Hz
PPHCH 11.91736 ppm/cm
HZCH 1199.04077 Hz/cm

11

Current Data Parameters
NAME sg_11f_51a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041203
Time 12.08
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5592.841 Hz
FIDRES 0.170880 Hz
AQ 2.9295992 sec
RG 203
DW 89.400 usec
DE 6.00 usec
TE 296.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWPR 0.01500000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 11.989 ppm
F1 4797.07 Hz
F2 -1.989 ppm
F2 -785.77 Hz
PNUCH 0.69888 ppm/cm
HZCH 279.64206 Hz/cm

SI-30

Current Data Parameters
NAME sg_11f_51a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041203
Time 12.14
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3654756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 297.0 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWPR 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6262298 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.40 usec
PL2 -3.00 dB
PL12 19.96 dB
PL13 120.00 dB
SFO2 400.1519805 MHz

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

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
FIP 215.000 ppm
F1 21631.75 Hz
F2 -5.000 ppm
F2 -503.06 Hz
PNUCH 11.00000 ppm/cm
HZCH 1106.74048 Hz/cm

```

10 NMR plot parameters
Cx          20.00 cm
CY          10.00 cm
F1P         12.989 ppm
F1          5197.20 Hz
F2P         -0.989 ppm
F2          -395.64 Hz
PPHCH       0.69888 ppm/cm
HZCH        279.64206 Hz/cm

```

SI-31

```

1D NMR plot parameters
CX          20.00 cm
CY          30.00 cm
F1P         215.000 ppm
F1          21631.75 Hz
F2P         -5.000 ppm
F2          -503.06 Hz
PPHON      11.00000 ppm/cm
HZCX       1106.74048 Hz/cm

```

SI-32

Current Data Parameters
NAME adv-VI-199
EXTNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20030723
Time 8.22
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 161.3
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 13.01 cm
PIF 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCH 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

SI-33

Current Data Parameters
NAME adv-VI-199
EXTNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20030731
Time 16.43
INSTRUM drx400
PROBRD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 143
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4597.6
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 6.62 cm
PIF 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCH 11.50000 ppm/cm
HZCM 1157.04639 Hz/cm

Current Data Parameters
NAME adv-VI-242
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030915
Time 14.31
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
MS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 14.82 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPHCH 0.55000 ppm/cm
RZCH 220.07150 Hz/cm

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Current Data Parameters
NAME adv-VI-242
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030915
Time 14.35
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
MS 311
DS 2
SWH 23148.146 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DW 21.500 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 100.6127763 MHz
WDW BW
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.52 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPHCH 11.50000 ppm/cm
RZCH 1157.04700 Hz/cm

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Current Data Parameters
NAME sg_111_106a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041215
Time 13.22
INSTRUM spect
PROBHD 5 mm QNP 1H/15
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 6.00 usec
TE 295.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCHWK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.05 usec
PL1 -3.00 dB
SFO1 400.132007 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 10.00 cm
F1P 10.995 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCH 0.58845 ppm/cm
HZCH 239.46359 Hz/cm

SI-35

Current Data Parameters
NAME sg_111_106a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041215
Time 13.24
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 22980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DM 20.850 usec
DE 6.00 usec
TE 295.7 K
D1 0.15000001 sec
D11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCHWK 0.01500000 sec

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

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

F2 - Processing parameters
SI 32768
SF 100.5127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCH 11.00000 ppm/cm
HZCH 1106.74048 Hz/cm

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Current Data Parameters
NAME sg_111_104a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041215
Time 13.08
INSTRUM spect
PROBHD 5 mm QNP 1H/15
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
DN 104.400 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWAK 0.01500000 sec

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

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

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.39 Hz
PPMCH 0.55845 ppm/cm
HZCH 239.46359 Hz/cm

SI-36

Current Data Parameters
NAME sg_111_104a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041215
Time 13.14
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 290
DS 4
SWH 23980.814 Hz
FIDRES 0.365916 Hz
AQ 1.3664756 sec
RG 2048
DN 20.850 usec
DE 6.00 usec
TE 295.7 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWAK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6222296 MHz

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

F2 - Processing parameters
SI 32768
SF 100.6127590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCH 11.00000 ppm/cm
HZCH 1106.74048 Hz/cm