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Cobalt-Mediated Macrocyclizations. Facile Synthesis of 2-Oxo-Pyridinophanes via [2 + 2 + 2] Cycloaddition of α,ω -Diyne and Isocyanates

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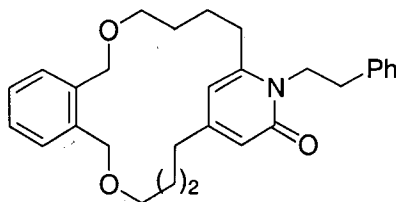
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Supporting Information

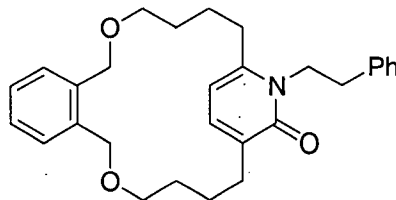
Experimental Section

The general methods and characterization data for bis-alkynes **1**, **5**, **7**, **9**, and **11** were previously reported.¹

Typical Procedure for Synthesis of Macrocyclic 2-Pyridone-cyclophanes. In a 100-mL round-bottom flask, equipped with a condenser and a three-way stopper connected to a balloon of argon, a mixture of diyne **1** (100 mg, 0.34 mmol, 1 equiv) and β -phenethyl isocyanate (477 mg, 3.24 mmol, 9.7 equiv) was pumped briefly and purged three times with argon. Fifty milliliters of 1,2-dimethoxyethane was then added, followed by a 10-mL solution of CpCo(CO)_2 (12.8 μL , 0.10 mmol, 30 mol %), and the remaining volume of the solvent to provide a final 0.005 M concentration (relative to diyne **1**). The resulting light orange solution was then heated at reflux for 24 h. The reaction mixture was then cooled to room temperature. Subsequent removal of the solvents *in vacuo*, followed by flash chromatography (SiO_2 , 3%, 9%, 20%, 33% and 50% ethyl acetate in hexanes, and then 2% methanol in dichloromethane) afforded 69 mg of the *para*-pyridone cyclophane, **2p** (45%) and 34 mg of the *meta*-pyridone-cyclophane **2m** (23%). For best results, newly opened bottles of anhydrous 1,2-dimethoxyethane (EM Science) and CpCo(CO)_2 (Strem Chemicals, Inc.) should be used in the reactions.

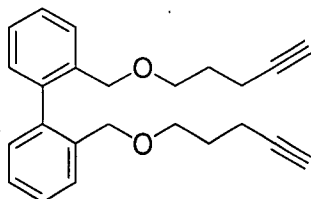


21-Phenethyl-6,15-dioxa-21-aza-tricyclo[18.3.1.08,13]tetracos-1(23),8(13),9,11,20(24)-pentaen-22-one (2m). ^1H NMR (CDCl_3 , 500 MHz): δ 7.12-7.32 (m, 9H), 6.26 (s, 1H), 5.86 (s, 1H), 4.51 (s, 2H), 4.46 (s, 2H), 4.12 (m, 2H), 3.47 (m, 4H), 2.93 (m, 2H), 2.36 (m, 4H), 1.54-1.65 (m, 8H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 163.83, 153.99, 147.64, 138.57, 136.67, 136.31, 129.16, 128.90, 128.64, 128.59, 127.88, 127.73, 126.65, 116.28, 107.87, 70.60, 70.12, 70.01, 69.88, 45.30, 34.76, 34.40, 31.63, 28.11, 27.58, 26.19, 23.80. IR (neat, cm^{-1}): 2936, 2862, 1652, 1569, 1497, 1454, 1257, 1089, 750, 700. HRMS (FAB) Calcd for $\text{C}_{29}\text{H}_{35}\text{NO}_3$: 445.59. Found: 446.270070 (MH^+).

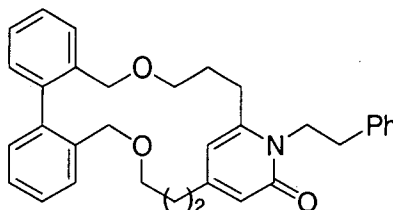


21-Phenethyl-6,15-dioxa-21-aza-tricyclo[18.2.2.08,13]tetracos-1(23),8(13),9,11,20(24)-pentaen-22-one (2p). ^1H NMR (CDCl_3 , 500 MHz): δ 7.36 (m, 1H), 7.18-7.32 (m, 8H), 7.05 (d, $J = 7.0$ Hz, 1H), 5.88 (d, $J = 7.0$ Hz, 1H), 4.328 (s, 2H), 4.327 (s, 2H), 4.23 (br s, 2H), 3.56 (m,

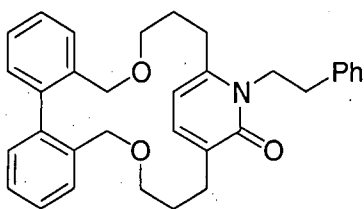
2H), 3.28 (t, $J = 6.9$ Hz, 2H), 2.95 (t, $J = 7.8$ Hz, 2H), 2.65 (br s, 2H), 2.53 (m, 2H), 1.72-1.78 (m, 2H), 1.61-1.66 (m, 2H), 1.46-1.53 (m, 4H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 163.40, 145.91, 138.58, 136.59, 136.43, 136.02, 129.93, 128.90, 128.60, 127.89, 127.59, 127.35, 126.60, 107.24, 69.83, 69.73, 69.55, 69.47, 46.05, 34.95, 32.82, 30.62, 27.89, 27.43, 23.84, 23.02. IR (neat, cm^{-1}): 2938, 2863, 1702, 1643, 1592, 1562, 1496, 1454, 1363, 1258, 1138, 1093, 752, 702. HRMS (FAB) Calcd for $\text{C}_{29}\text{H}_{35}\text{NO}_3$: 445.59. Found: 446.270659 (MH^+).



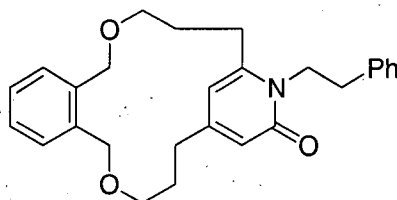
2,2'-Bis-pent-4-ynyloxymethyl-biphenyl (3). Compound **3** was prepared similarly as compound **5**, in 35% yield. ^1H NMR (CDCl_3 , 300 MHz): δ 7.14-7.55 (m, 8H), 4.19 (s, 4H), 3.39 (t, $J = 6.2$ Hz, 4H), 2.23 (td, $J = 7.2, 2.6$ Hz, 4H), 1.91 (t, $J = 2.7$ Hz, 2H), 1.68-1.77 (m, 4H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 139.89, 136.77, 129.97, 128.51, 128.03, 127.46, 84.37, 70.99, 69.38, 68.77, 29.01, 15.68. Anal. Calcd for $\text{C}_{24}\text{H}_{26}\text{O}_2$: C, 83.20, H, 7.56. Found: C, 83.18, H, 7.39.



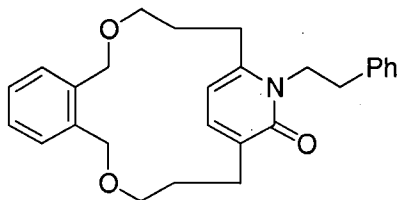
Compound 4m. ^1H NMR (CDCl_3 , 500 MHz): δ 7.57 (d, $J = 2.5$ Hz, 1H), 7.56 (d, $J = 3.0$ Hz, 1H), 7.37-7.41 (m, 3H), 7.27-7.31 (m, 3H), 7.19-7.24 (m, 3H), 7.05-7.09 (m, 2H), 6.23 (s, 1H), 5.71 (s, 1H), 4.37 (d, $J = 11$ Hz, 1H), 4.31 (d, $J = 10.5$ Hz, 1H), 4.21-4.25 (m, 1H), 4.14 (d, $J = 3.5$ Hz, 1H), 4.11 (d, $J = 4.0$ Hz, 1H), 4.05-4.14 (m, 1H), 3.24-3.35 (m, 3H), 3.17-3.21 (m, 1H), 2.96-2.99 (m, 2H), 2.59 (ddd, $J = 12.0, 8.0, 3.5$ Hz, 1H), 2.47 (ddd, $J = 11.0, 7.0, 4.0$ Hz, 1H), 2.40 (ddd, $J = 12.5, 6.0, 3.5$ Hz, 1H), 2.32 (ddd, $J = 11.0, 5.5, 5.5$ Hz, 1H), 1.79-1.83 (m, 2H), 1.65-1.74 (m, 2H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 163.88, 153.55, 146.69, 138.93, 138.65, 138.53, 136.32, 129.48, 129.43, 128.89, 128.64, 127.79, 127.71, 127.65, 127.02, 126.81, 126.73, 126.65, 116.30, 109.52, 70.40, 70.11, 68.03, 67.35, 45.46, 34.80, 30.63, 29.04, 28.73, 27.46. HRMS (FAB) Calcd for $\text{C}_{33}\text{H}_{35}\text{NO}_3$: 493.64. Found: 494.270105 (MH^+).



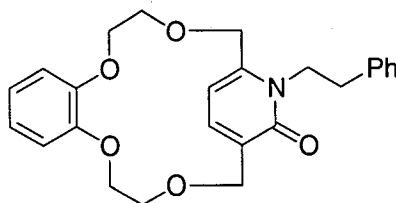
Compound 4p. ^1H NMR (CDCl_3 , 500 MHz): δ 7.64 (d, J = 6.5 Hz, 1H), 7.50 (d, J = 6.5 Hz, 1H), 7.38-7.41 (m, 1H), 7.32-7.35 (m, 1H), 7.19-7.29 (m, 6H), 7.00 (dd, J = 6.0, 0.5 Hz, 1H), 6.97 (dd, J = 19.0, 6.0, Hz, 1H), 6.971 (dd, J = 19.5, 6.0, Hz, 1H), 6.85 (d, J = 6.0 Hz, 1H), 5.69 (d, J = 5.5 Hz, 1H), 4.32 (d, J = 11.5 Hz, 1H), 4.31 (d, J = 11.5 Hz, 1H), 4.23 (br s, 1H), 4.03 (br s, 1H), 3.77 (d, J = 12.0 Hz, 1H), 3.66 (d, J = 11.5 Hz, 1H), 3.23 (ddd, J = 8.5, 4.5, 4.5 Hz, 1H), 3.18 (ddd, J = 8.5, 4.0, 4.0 Hz, 1H), 3.11 (br ddd, J = 8.0, 8.0, 8.0 Hz, 1H), 2.95-3.01 (m, 2H), 2.84-2.89 (m, 1H), 2.71-2.79 (m, 1H), 2.58-2.66 (m, 2H), 2.51-2.55 (m, 1H), 1.93-2.02 (m, 2H), 1.78-1.82 (m, 2H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 163.32, 145.09, 138.60, 138.18, 137.75, 137.02, 136.51, 135.74, 129.50, 129.40, 128.90, 128.74, 128.60, 127.67, 127.62, 126.68, 126.60, 126.49, 126.47, 126.45, 107.67, 70.56, 70.49, 69.45, 67.64, 46.12, 34.98, 29.84, 28.36, 27.58, 26.19. HRMS (FAB) Calcd for $\text{C}_{33}\text{H}_{35}\text{NO}_3$: 493.64. Found: 494.269347 (MH^+).



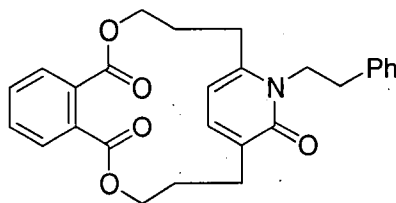
19-Phenethyl-5,14-dioxa-19-aza-tricyclo[16.3.1.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (6m). ^1H NMR (CDCl_3 , 500 MHz): δ 7.17-7.38 (m, 9H), 6.28 (s, 1H), 5.96 (s, 1H), 4.50 (s, 2H), 4.49 (s, 2H), 4.17 (br t, J = 7.4 Hz, 2H), 3.45 (t, J = 6.0 Hz, 2H), 3.41 (t, J = 5.9 Hz, 2H), 2.99 (t, J = 7.6 Hz, 2H), 2.52 (t, J = 6.1 Hz, 4H), 1.93 (quintet, J = 6.1 Hz, 2H), 1.87 (quintet, J = 6.1 Hz, 2H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 163.98, 153.51, 146.76, 138.63, 136.39, 136.20, 129.42, 129.21, 128.91, 128.64, 128.04, 127.98, 126.65, 116.21, 110.65, 70.63, 70.30, 68.02, 45.65, 34.75, 30.43, 29.04, 28.94, 27.40. IR (neat, cm^{-1}): 2926, 2860, 1717, 1658, 1579, 1546, 1454, 1124, 1097, 1076, 752, 701. HRMS (FAB) Calcd for $\text{C}_{27}\text{H}_{31}\text{NO}_3$: 417.54. Found: 418.239232 (MH^+).



19-Phenethyl-5,14-dioxa-19-aza-tricyclo[16.2.2.0^{7,12}]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (6p). ¹H NMR (CDCl₃, 500 MHz): δ 7.19-7.33 (m, 9H), 7.02 (d, *J* = 6.9 Hz, 1H), 5.79 (d, *J* = 6.9 Hz, 1H), 4.58 (m, 1H), 4.06-4.16 (m, 4H), 3.79-3.84 (m, 1H), 3.45-3.63 (m, 4H), 3.07-3.11 (m, 1H), 2.99-3.05 (m, 1H), 2.84-2.90 (m, 1H), 2.68 (ddd, *J* = 14.8, 4.8, 4.8 Hz, 1H), 2.40 (ddd, *J* = 15.3, 9.9, 5.6 Hz, 1H), 2.19-2.26 (m, 1H), 1.83-1.95 (m, 4H). ¹³C NMR (CDCl₃, 125 MHz): δ 163.43, 145.78, 138.91, 136.59, 136.45, 135.48, 129.82, 128.89, 128.55, 128.24, 128.16, 127.46, 127.07, 126.47, 107.14, 71.32, 69.90, 69.42, 69.28, 46.39, 34.69, 31.61, 30.12, 27.69, 26.71. IR (neat, cm⁻¹): 2922, 2860, 1716, 1646, 1595, 1564, 1496, 1454, 1364, 1186, 1124, 1087, 1042, 753, 702. HRMS (FAB) Calcd for C₂₇H₃₁NO₃: 417.54. Found: 418.238755 (MH⁺).

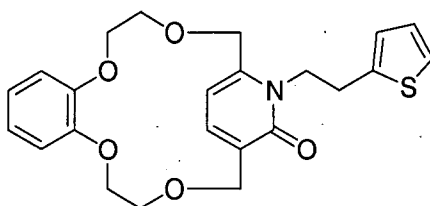


19-Phenethyl-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.0^{7,12}]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (8). ¹H NMR (CDCl₃, 500 MHz): δ 7.29 (d, *J* = 6.7 Hz, 1H), 7.23-7.26 (m, 2H), 7.18-7.20 (m, 3H), 6.73-6.80 (m, 3H), 6.67-6.69 (m, 1H), 6.07 (d, *J* = 6.6 Hz, 1H), 5.08 (d, *J* = 12.1 Hz, 1H), 4.56 (d, *J* = 13.6 Hz, 1H), 4.11-4.25 (m, 4H), 4.05 (ddd, *J* = 11.2, 4.7, 0.8 Hz, 1H), 3.99 (obscured d, *J* = 12.2 Hz, 2H), 3.86 (obscured d, *J* = 13.7 Hz, 1H), 3.85-3.90 (m, 1H), 3.80 (ddd, *J* = 12.0, 7.5, 0.9 Hz, 1H), 3.76 (ddd, *J* = 10.7, 4.7, 0.9 Hz, 1H), 3.71 (ddd, *J* = 12.1, 2.8, 1.2 Hz, 1H), 2.98 (t, *J* = 7.7 Hz, 2H). ¹³C NMR (CDCl₃, 125 MHz): δ 163.27, 148.38, 147.88, 143.65, 139.34, 137.32, 129.02, 128.74, 128.49, 126.34, 120.76, 120.24, 112.29, 111.83, 107.90, 70.33, 68.93, 68.64, 67.93, 67.50, 67.28, 46.67, 34.25. Anal. Calcd for C₂₅H₂₇NO₅: C, 71.24, H, 6.46, N, 3.32. Found: C, 70.62, H, 6.51, N, 3.21.

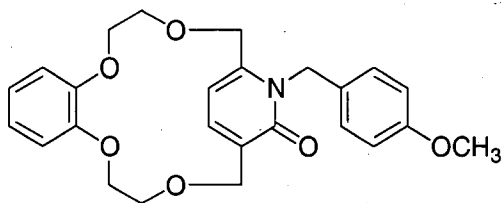


19-Phenethyl-5,14-dioxa-19-aza-tricyclo[16.2.2.0^{7,12}]docosa-1(21),7(12),8,10,18(22)-pentaene-6,13,20-trione (10). ¹H NMR (CDCl₃, 500 MHz): δ 7.75-7.79 (m, 1H), 7.53-7.57 (m, 1H), 7.46-7.50 (m, 2H), 7.14-7.31 (m, 5H), 6.95 (d, *J* = 7.0 Hz, 1H), 5.85 (d, *J* = 7.0 Hz, 1H), 4.53 (ddd, *J* = 11.4, 5.8, 3.8 Hz, 1H), 4.43 (ddd, *J* = 11.5, 8.7, 2.9 Hz, 1H), 4.27 (m, 2H), 4.15 (ddd, *J* = 11.4, 6.6, 3.5 Hz, 1H), 3.82 (ddd, *J* = 13.2, 9.5, 7.1 Hz, 1H), 3.11 (ddd, *J* = 13.8, 5.5, 5.5 Hz, 1H), 2.76-2.84 (m, 2H), 2.73 (obscured ddd, *J* = 15.4, 7.1, 5.3 Hz, 1H), 2.57 (ddd, *J* = 15.5, 7.9, 5.3 Hz, 1H), 2.38-2.46 (m, 1H), 2.35 (ddd, *J* = 15.1, 9.6, 5.9 Hz, 1H), 2.03-2.20 (m, 3H). ¹³C NMR (CDCl₃, 125 MHz): δ 167.40, 166.15, 163.32, 144.84, 138.69, 137.06, 131.21, 130.96, 130.74, 129.27, 128.84, 128.77, 128.59, 128.13, 127.68, 126.54, 107.37, 63.65, 63.18,

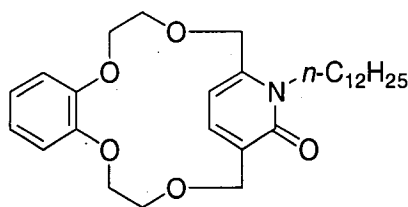
46.21, 34.36, 29.89, 28.78, 25.04, 23.99. HRMS (FAB) Calcd for $C_{27}H_{27}NO_5$: 445.51. Found: 446.198481 (MH^+).



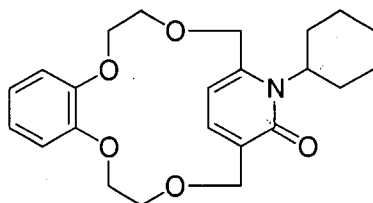
19-[2-(2-Thienyl)ethyl]-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (12). 1H NMR ($CDCl_3$, 500 MHz): δ 7.28 (d, J = 6.7 Hz, 1H), 7.10 (d, J = 5.1 Hz, 1H), 6.88 (dd, J = 5.1, 3.5 Hz, 1H), 6.70-6.81 (m, 5H), 6.07 (d, J = 6.6 Hz, 1H), 5.06 (d, J = 12.2 Hz, 1H), 4.53 (d, J = 13.7 Hz, 1H), 3.98 (obscured d, J = 12.2 Hz, 1H), 3.87 (obscured d, J = 13.8 Hz, 1H), 3.70-4.07 (m, 10H), 3.23-3.30 (m, 1H), 3.12-3.18 (m, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 163.25, 148.36, 147.83, 143.76, 141.37, 137.43, 128.76, 127.01, 125.50, 123.77, 120.81, 120.29, 112.31, 111.80, 107.86, 70.34, 68.90, 68.66, 67.90, 67.58, 67.27, 46.80, 27.99. HRMS (FAB) Calcd for $C_{23}H_{25}NO_5S$: 427.51. Found: 428.154348 (MH^+).



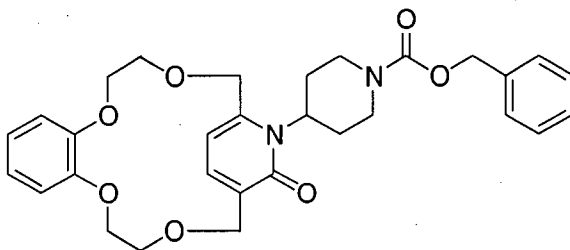
19-(4-Methoxybenzyl)-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (13). 1H NMR ($CDCl_3$, 500 MHz): δ 7.31 (d, J = 6.7 Hz, 1H), 7.11 (d, J = 8.8 Hz, 2H), 6.73-6.86 (m, 6H), 6.11 (d, J = 6.6 Hz, 1H), 5.63 (d, J = 15.5 Hz, 1H), 5.06 (d, J = 12.2 Hz, 1H), 5.03 (d, J = 15.6 Hz, 1H), 4.74 (d, J = 13.5 Hz, 1H), 4.13-4.21 (m, 2H), 4.06 (ddd, J = 11.9, 4.9, 1.1 Hz, 1H), 4.00 (obscured d, J = 12.3 Hz, 1H), 3.97-4.01 (m, 1H), 3.91-3.95 (m, 1H), 3.91 (obscured d, J = 13.5 Hz, 1H), 3.79-3.87 (m, 2H), 3.75 (m, 1H), 3.73 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 163.53, 158.61, 148.52, 148.11, 143.62, 137.48, 129.79, 129.17, 128.16, 120.85, 120.24, 113.95, 112.38, 112.09, 108.39, 70.24, 68.95, 68.64, 68.06, 67.52, 55.23, 45.91. HRMS (FAB) Calcd for $C_{25}H_{27}NO_6$: 437.49. Found: 438.191578 (MH^+).



19-Dodecyl-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (14). ^1H NMR (CDCl_3 , 500 MHz): δ 7.20 (d, J = 6.6 Hz, 1H), 6.64-6.75 (m, 4H), 6.03 (d, J = 6.6 Hz, 1H), 4.95 (d, J = 12.2 Hz, 1H), 4.76 (d, J = 13.5 Hz, 1H), 3.91 (obscured d, J = 13.6 Hz, 1H), 3.65-4.11 (m, 11H), 1.42-1.64 (m, 2H), 1.18 (m, 18H), 0.81 (t, J = 6.9 Hz, 3H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 162.18, 147.44, 146.94, 142.27, 136.10, 127.81, 119.75, 119.17, 111.33, 110.90, 107.08, 69.33, 67.84, 67.63, 66.88, 66.44, 43.63, 30.91, 28.69, 28.62, 28.61, 28.57, 28.50, 28.34, 28.26, 27.72, 26.19, 21.68, 13.11. HRMS (FAB) Calcd for $\text{C}_{29}\text{H}_{43}\text{NO}_5$: 485.66. Found: 486.322307 (MH^+).

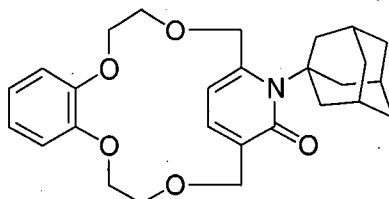


19-Cyclohexyl-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (15). ^1H NMR (CDCl_3 , 500 MHz): δ 7.20 (d, J = 6.5 Hz, 1H), 6.77-6.83 (m, 2H), 6.74-6.76 (m, 1H), 6.69-6.70 (m, 1H), 6.04 (d, J = 6.4 Hz, 1H), 5.01 (d, J = 12.1 Hz, 1H), 4.89 (d, J = 13.7 Hz, 1H), 3.93-4.09 (m, 5H), 3.95 (obscured d, J = 12.4 Hz, 1H), 3.89 (obscured d, J = 12.0 Hz, 1H), 3.74-3.80 (m, 3H), 3.48 (m, 1H), 1.92-1.95 (m, 2H), 1.66-1.72 (m, 2H), 1.30-1.39 (m, 2H), 1.15-1.21 (m, 2H), 1.06-1.14 (m, 2H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 164.06, 148.43, 147.78, 143.65, 136.61, 130.18, 120.69, 120.19, 112.17, 111.43, 108.26, 71.04, 68.89, 68.70, 68.12, 67.41, 67.16, 49.17, 33.96, 29.08, 26.66, 25.61, 24.94. HRMS (FAB) Calcd for $\text{C}_{23}\text{H}_{29}\text{NO}_5$: 399.48. Found: 400.213417 (MH^+).

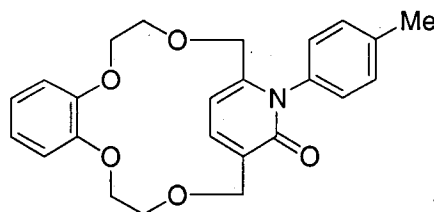


4-(20-Oxo-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-19-yl)-piperidine-1-carboxylic Acid Benzyl Ester (16). ^1H NMR (CDCl_3 , 500 MHz): δ 7.28-7.38 (m, 5H), 7.21 (d, J = 6.5 Hz, 1H), 6.67-6.84 (m, 4H), 6.06

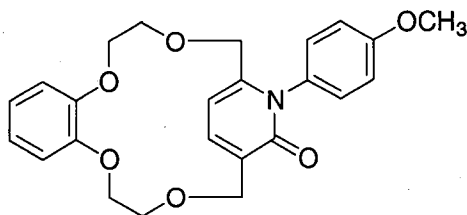
(d, $J = 6.5$ Hz, 1H), 4.98-5.12 (m, 3H), 4.90 (d, $J = 13.8$ Hz, 1H), 4.31 (m, 1H), 3.87-4.20 (m, 5H), 3.98 (obscured d, $J = 14.1$ Hz, 1H), 3.70-3.77 (m, 4H), 2.72-3.02 (m, 4H), 1.47-1.64 (m, 4H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 163.91, 155.08, 148.32, 147.61, 143.30, 136.89, 136.77, 130.38, 128.40, 127.88, 120.89, 120.32, 112.23, 111.32, 108.46, 70.82, 68.81, 68.70, 67.94, 67.38, 67.12, 66.99, 58.61, 43.92, 28.09, 25.97. HRMS (FAB) Calcd for $\text{C}_{30}\text{H}_{34}\text{N}_2\text{O}_7$: 534.60. Found: 535.244505 (MH^+).



19-Adamantan-1-yl-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (17). ^1H NMR (CDCl_3 , 500 MHz): δ 7.30 (d, $J = 6.8$ Hz, 1H), 6.70-6.84 (m, 4H), 6.40 (d, $J = 6.8$ Hz, 1H), 4.83 (d, $J = 14.0$ Hz, 1H), 4.65 (d, $J = 13.6$ Hz, 1H), 4.32 (d, $J = 14.1$ Hz, 1H), 4.17 (d, $J = 13.7$ Hz, 1H), 3.89-3.99 (m, 4H), 3.72-3.81 (m, 3H), 3.53-3.59 (m, 1H), 2.53 (br d, $J = 11.5$ Hz, 2H), 2.29 (br d, $J = 11.1$ Hz, 2H), 2.08 (br s, 2H), 1.98-2.00 (m, 1H), 1.85-1.88 (m, 2H), 1.70 (br d, $J = 11.6$ Hz, 2H), 1.58-1.61 (m, 4H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 166.77, 149.08, 148.40, 146.81, 136.73, 130.35, 121.22, 120.46, 113.49, 112.51, 111.81, 72.76, 69.52, 69.09, 68.63, 68.60, 68.08, 67.22, 40.43, 36.22, 30.67. HRMS (FAB) Calcd for $\text{C}_{27}\text{H}_{33}\text{NO}_5$: 451.55. Found: 452.244071 (MH^+).



19-p-Tolyl-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (18). ^1H NMR (CDCl_3 , 500 MHz): δ 7.42 (d, $J = 6.7$ Hz, 1H), 7.21-7.25 (m, 1H), 7.10-7.15 (m, 2H), 7.01 (dd, $J = 8.0, 2.1$ Hz, 1H), 6.76-6.88 (m, 4H), 6.26 (d, $J = 6.7$ Hz, 1H), 4.97 (d, $J = 12.3$ Hz, 1H), 4.36 (d, $J = 13.2$ Hz, 1H), 4.06-4.17 (m, 3H), 4.07 (obscured d, $J = 12.3$ Hz, 1H), 3.85 (obscured d, $J = 13.3$ Hz, 1H), 3.77-3.97 (m, 5H), 2.35 (s, 3H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 164.06, 148.57, 147.96, 143.84, 138.39, 138.16, 135.27, 130.07, 129.92, 129.60, 129.34, 126.94, 120.81, 120.29, 112.20, 111.62, 107.69, 69.44, 68.52, 68.38, 67.98, 67.93, 66.90, 21.26. HRMS (FAB) Calcd for $\text{C}_{24}\text{H}_{25}\text{NO}_5$: 407.46. Found: 408.180787 (MH^+).



19-(4-Methoxyphenyl)-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (19). ^1H NMR (CDCl_3 , 500 MHz): δ 7.42 (d, J = 6.7 Hz, 1H), 7.15 (dd, J = 8.8, 2.5 Hz, 1H), 7.04 (dd, J = 8.6, 2.5 Hz, 1H), 6.93 (dd, J = 8.6, 2.9 Hz, 1H), 6.77-6.87 (m, 5H), 6.26 (d, J = 6.6 Hz, 1H), 4.97 (d, J = 12.3 Hz, 1H), 4.38 (d, J = 13.3 Hz, 1H), 4.06-4.17 (m, 4H), 3.77-3.97 (m, 5H), 3.79 (obscured s, 3H), 3.85 (obscured d, J = 13.3 Hz, 1H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 164.20, 159.41, 148.59, 147.98, 144.08, 138.16, 131.47, 130.55, 129.61, 128.20, 120.86, 120.31, 114.22, 114.16, 112.23, 111.65, 107.73, 69.47, 68.53, 68.45, 67.97, 66.84, 55.37. HRMS (FAB) Calcd for $\text{C}_{24}\text{H}_{25}\text{NO}_6$: 423.46. Found: 424.176388 (MH^+).

¹ Moretto, A. F.; Zhang, H.-C.; Maryanoff, B. E. *J. Am. Chem. Soc.* **2001**, *123*, 3157-3158.

Single-Crystal X-ray Analysis of 19-Phenethyl-3,6,13,16-tetraoxa-19-azatricyclo[16.2.2.0^{7,12}]docosa-1(21),7,9,11,18(22)-pentaen-20-one (8). Crystals of C₂₅H₂₇NO₅ (MW = 421.48 Da, colorless square plates), which had been recrystallized from EtOAc, were severely twinned. One of these twinned specimens was cut repeatedly with a razor blade, until a small, mostly single-domain piece was obtained. The diffraction pattern from the principal domain of this crystal did not overlap with the diffraction patterns of other domains. This crystal, at -80±2°C, was orthorhombic, space group P2₁2₁2₁ - D (No. 19) with *a* = 10.325(1) Å, *b* = 11.651(1) Å, *c* = 17.868(2) Å, *V* = 2149.4(4) Å³ and *Z* = 4 {*d*_{calcd} = 1.302 g·cm⁻³; μ_a (MoK) = 0.091 mm⁻¹}. A full hemisphere of diffracted intensities (ω -scan width of 0.30°) was measured using graphite-monochromated MoK α radiation on a Bruker SMART APEX CCD Single Crystal Diffraction System. X-rays were provided by a fine-focus sealed x-ray tube operated at 50kV and 35mA. Lattice constants were determined with the Bruker SAINT software package using peak centers for 2466 reflections. A total of 12882 integrated reflection intensities having 2 Θ (MoK) < 56.24° were produced using the Bruker program SAINT. A total of 4741 of these were independent and gave *R*_{int} = 0.057. The Bruker SHELXTL-PC software package was used to solve the structure using "direct methods" techniques. All stages of weighted full-matrix least-squares refinement were conducted using *F*_o² data with the SHELXTL-PC Version 5 software package. Final agreement factors at convergence are: *R*₁(unweighted, based on *F*) = 0.043 for 3116 independent reflections having 2 Θ (MoK) < 56.24° and *I* > 2 σ (*I*); *R*₁(unweighted, based on *F*) = 0.071 and *wR*₂ (weighted, based on *F*²) = 0.072 for all 4741 independent reflections having 2 Θ (MoK) < 56.24°.

The structural model incorporated anisotropic thermal parameters for all nonhydrogen atoms and isotropic thermal parameters for all hydrogen atoms. All hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp²- or sp³-hybridization of the carbon atoms and C-H bond lengths of 0.95-0.99 Å) "riding" on their respective carbon atoms. The isotropic thermal parameter of each hydrogen atom was fixed at a value 1.2 times the equivalent isotropic thermal parameter of the carbon atom to which it is covalently bonded.

Table 1. Atomic Coordinates for Nonhydrogen Atoms in Crystalline 19-Phenethyl-3,6,13,16-tetraoxa-19-aza-tricyclo[16.2.2.0^{7,12}]docosa-1(21),7,9,11,18(22)-pentaen-20-one, C₂₅H₂₇NO₅ (8)^a

Atom	Fractional Coordinates			Equivalent Isotropic Thermal Parameter,
Type ^b	10 ⁴ x	10 ⁴ y	10 ⁴ z	U, Å ² x 10 ³ ^c
C ₁	5930(2)	-1250(2)	2369(1)	33(1)
C ₂	7165(2)	-1048(2)	2796(1)	42(1)
O ₃	7003(1)	-205(1)	3372(1)	43(1)
C ₄	6247(2)	-586(2)	3989(1)	44(1)
C ₅	5407(2)	385(2)	4253(1)	39(1)
O ₆	4339(1)	480(1)	3749(1)	36(1)
C ₇	3463(2)	1330(2)	3896(1)	30(1)
C ₈	3653(2)	2228(2)	4385(1)	36(1)
C ₉	2692(2)	3061(2)	4483(1)	40(1)
C ₁₀	1550(2)	2981(2)	4100(1)	42(1)
C ₁₁	1339(2)	2072(2)	3615(1)	38(1)
C ₁₂	2285(2)	1258(2)	3500(1)	32(1)
O ₁₃	2175(1)	358(1)	3012(1)	38(1)
C ₁₄	893(2)	75(2)	2776(1)	42(1)
C ₁₅	973(2)	-995(2)	2317(1)	46(1)
O ₁₆	1386(1)	-730(1)	1575(1)	50(1)
C ₁₇	2275(2)	-1538(2)	1264(1)	49(1)
C ₁₈	3587(2)	-1484(2)	1628(1)	35(1)
N ₁₉	4321(2)	-507(1)	1519(1)	32(1)
C ₂₀	5519(2)	-343(2)	1873(1)	33(1)
C ₂₁	5214(2)	-2209(2)	2437(1)	37(1)
C ₂₂	4015(2)	-2335(2)	2070(1)	37(1)
C ₂₃	3927(2)	405(2)	997(1)	39(1)
C ₂₄	4491(2)	235(2)	216(1)	45(1)
C ₂₅	4247(2)	1269(2)	-271(1)	36(1)
C ₂₆	5216(2)	2050(2)	-417(1)	49(1)
C ₂₇	4978(2)	3027(2)	-842(1)	56(1)
C ₂₈	3762(3)	3233(2)	-1117(1)	50(1)
C ₂₉	2794(2)	2462(2)	-978(1)	47(1)
C ₃₀	3026(2)	1484(2)	-560(1)	41(1)
O ₃₁	6153(1)	530(1)	1744(1)	45(1)

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- ^a The numbers in parentheses are the estimated standard deviations in the last significant digit.
^b Atoms are labeled in agreement with Figure 1.
^c This is one-third of the trace of the orthogonalized U_{ij} tensor.

Table 2. Anisotropic Thermal Parameters for Nonhydrogen Atoms in Crystalline **8**^{a,b}

Atom Type ^c	Anisotropic Thermal Parameters ($\text{\AA}^2 \times 10^3$)					
	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C ₁	35(1)	31(1)	33(1)	-3(1)	3(1)	8(1)
C ₂	35(1)	47(1)	45(1)	-3(1)	0(1)	8(1)
O ₃	38(1)	50(1)	42(1)	-7(1)	0(1)	2(1)
C ₄	44(1)	50(1)	38(1)	1(1)	-7(1)	8(1)
C ₅	37(1)	44(1)	35(1)	-3(1)	-5(1)	3(1)
O ₆	30(1)	38(1)	38(1)	-6(1)	-6(1)	2(1)
C ₇	31(1)	30(1)	29(1)	5(1)	5(1)	-2(1)
C ₈	40(1)	38(1)	30(1)	-1(1)	4(1)	-6(1)
C ₉	49(2)	34(1)	39(1)	-4(1)	11(1)	-4(1)
C ₁₀	43(1)	37(1)	45(1)	2(1)	8(1)	10(1)
C ₁₁	35(1)	42(1)	38(1)	4(1)	0(1)	2(1)
C ₁₂	34(1)	32(1)	30(1)	3(1)	3(1)	1(1)
O ₁₃	30(1)	43(1)	42(1)	-8(1)	-6(1)	3(1)
C ₁₄	29(1)	55(1)	42(1)	-5(1)	-4(1)	3(1)
C ₁₅	34(1)	57(1)	47(1)	-9(1)	0(1)	-6(1)
O ₁₆	42(1)	67(1)	40(1)	-10(1)	-6(1)	8(1)
C ₁₇	50(2)	55(1)	42(1)	-16(1)	-3(1)	-2(1)
C ₁₈	38(1)	35(1)	33(1)	-9(1)	2(1)	2(1)
N ₁₉	37(1)	28(1)	31(1)	0(1)	-3(1)	4(1)
C ₂₀	38(1)	32(1)	30(1)	-4(1)	3(1)	5(1)
C ₂₁	51(2)	27(1)	34(1)	4(1)	5(1)	10(1)
C ₂₂	41(1)	30(1)	39(1)	-3(1)	6(1)	-5(1)
C ₂₃	48(1)	34(1)	36(1)	0(1)	-5(1)	10(1)
C ₂₄	55(2)	43(1)	36(1)	0(1)	1(1)	15(1)
C ₂₅	40(1)	39(1)	29(1)	-6(1)	0(1)	6(1)
C ₂₆	36(1)	62(2)	48(1)	0(1)	-5(1)	2(1)
C ₂₇	56(2)	52(2)	60(2)	9(1)	3(1)	-12(1)
C ₂₈	60(2)	48(1)	42(1)	9(1)	4(1)	9(1)
C ₂₉	42(1)	57(1)	43(1)	5(1)	-6(1)	12(1)
C ₃₀	40(1)	41(1)	42(1)	1(1)	0(1)	-1(1)
O ₃₁	48(1)	34(1)	52(1)	2(1)	0(1)	-7(1)

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.^b The form of the anisotropic thermal parameter is: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^{*}b^{*} + 2U_{13}hla^{*}c^{*} + 2U_{23}klb^{*}c^{*})]$.^c Atoms are labeled in agreement with Figure 1.

Table 3. Atomic Coordinates for Hydrogen Atoms in Crystalline **8**^a

Atom Type ^b	Fractional Coordinates		
	10 ⁴ x	10 ⁴ y	10 ⁴ z
H _{2a}	7454	-1779	3024
H _{2b}	7847	-790	2445
H _{4a}	6822	-843	4399
H _{4b}	5700	-1243	3835
H _{5a}	5091	230	4767
H _{5b}	5908	1109	4260
H ₈	4441	2282	4658
H ₉	2833	3684	4816
H ₁₀	900	3549	4166
H ₁₁	535	2010	3359
H _{14a}	524	709	2475
H _{14b}	329	-52	3216
H _{15a}	1594	-1535	2550
H _{15b}	114	-1370	2300
H _{17a}	2370	-1386	721
H _{17b}	1919	-2322	1323
H ₂₁	5527	-2819	2741
H ₂₂	3516	-3013	2135
H _{23a}	4216	1156	1195
H _{23b}	2970	421	963
H _{24a}	5435	99	254
H _{24b}	4093	-450	-18
H ₂₆	6061	1919	-225
H ₂₇	5659	3553	-941
H ₂₈	3593	3905	-1402
H ₂₉	1951	2599	-1170
H ₃₀	2343	955	-472

^a All hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp²- or sp³-hybridization of the carbon atoms and C-H bond lengths of 0.95 -0.99 Å) "riding" on their respective carbon atoms. The isotropic thermal parameter of each hydrogen atom was fixed at a value 1.2 times the equivalent isotropic thermal parameter of the carbon atom to which it is covalently bonded.

^b Hydrogen atoms are labeled with the same numerical subscript(s) as the carbon atom to which they are covalently bonded; they also have an additional literal subscript (a or b) where necessary to distinguish between hydrogens bonded to the same carbon atom.

Table 4. Bond Lengths in Crystalline **8**^a

Type ^b	Length, Å	Type ^b	Length, Å
C ₂ -O ₃	1.432(2)	C ₁₈ -N ₁₉	1.381(2)
O ₃ -C ₄	1.422(2)	N ₁₉ -C ₂₀	1.402(2)
C ₅ -O ₆	1.428(2)		
O ₁₃ -C ₁₄	1.428(2)	N ₁₉ -C ₂₃	1.472(2)
C ₁₅ -O ₁₆	1.426(2)		
O ₁₆ -C ₁₇	1.429(2)	C ₂₀ -O ₃₁	1.231(2)
C ₁ -C ₂₀	1.444(2)	C ₇ -C ₈	1.377(2)
		C ₇ -C ₁₂	1.410(3)
C ₁ -C ₂₁	1.345(3)	C ₈ -C ₉	1.399(3)
		C ₉ -C ₁₀	1.366(3)
O ₆ -C ₇	1.367(2)	C ₁₀ -C ₁₁	1.386(3)
C ₁₂ -O ₁₃	1.368(2)	C ₁₁ -C ₁₂	1.377(3)
		C ₁₈ -C ₂₂	1.344(2)
C ₁ -C ₂	1.504(3)	C ₂₁ -C ₂₂	1.409(3)
C ₄ -C ₅	1.502(3)	C ₂₅ -C ₂₆	1.377(3)
C ₁₄ -C ₁₅	1.494(2)	C ₂₅ -C ₃₀	1.386(3)
C ₁₇ -C ₁₈	1.503(3)	C ₂₆ -C ₂₇	1.390(3)
C ₂₃ -C ₂₄	1.526(2)	C ₂₇ -C ₂₈	1.369(3)
C ₂₄ -C ₂₅	1.507(3)	C ₂₈ -C ₂₉	1.367(3)
C ₂₉ -C ₃₀	1.382(3)		

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.^b Atoms are labeled in agreement with Figure 1.

Table 5. Bond Angles in Crystalline **8**^a

Type ^b	Angle, (deg)	Type ^b	Angle, (deg)
O ₃ C ₂ C ₁	111.9(2)	O ₁₃ C ₁₂ C ₁₁	124.3(2)
C ₄ O ₃ C ₂	114.0(1)	O ₁₃ C ₁₂ C ₇	116.0(2)
O ₃ C ₄ C ₅	109.0(2)	C ₁₁ C ₁₂ C ₇	119.8(2)
O ₆ C ₅ C ₄	107.8(2)	C ₂₂ C ₁₈ N ₁₉	120.7(2)
C ₇ O ₆ C ₅	116.5(1)	C ₂₂ C ₁₈ C ₁₇	121.3(2)
C ₁₂ O ₁₃ C ₁₄	116.3(2)	N ₁₉ C ₁₈ C ₁₇	117.9(2)
O ₁₃ C ₁₄ C ₁₅	107.7(2)	C ₁₈ N ₁₉ C ₂₀	122.3(2)
O ₁₆ C ₁₅ C ₁₄	110.3(2)	C ₁₈ N ₁₉ C ₂₃	122.2(2)
C ₁₅ O ₁₆ C ₁₇	114.3(2)	C ₂₀ N ₁₉ C ₂₃	115.5(2)
O ₁₆ C ₁₇ C ₁₈	112.5(2)	O ₃₁ C ₂₀ N ₁₉	119.9(2)
N ₁₉ C ₂₃ C ₂₄	112.4(2)	O ₃₁ C ₂₀ C ₁	124.3(2)
C ₂₅ C ₂₄ C ₂₃	111.1(2)	N ₁₉ C ₂₀ C ₁	115.9(2)
		C ₁ C ₂₁ C ₂₂	121.9(2)
C ₂₁ C ₁ C ₂₀	120.1(2)	C ₁₈ C ₂₂ C ₂₁	119.1(2)
C ₂₁ C ₁ C ₂	123.4(2)	C ₂₆ C ₂₅ C ₃₀	118.1(2)
C ₂₀ C ₁ C ₂	116.5(2)	C ₂₆ C ₂₅ C ₂₄	121.1(2)
O ₆ C ₇ C ₈	125.3(2)	C ₃₀ C ₂₅ C ₂₄	120.8(2)
O ₆ C ₇ C ₁₂	115.5(2)	C ₂₅ C ₂₆ C ₂₇	121.1(2)
C ₈ C ₇ C ₁₂	119.2(2)	C ₂₈ C ₂₇ C ₂₆	120.1(2)
C ₇ C ₈ C ₉	120.3(2)	C ₂₉ C ₂₈ C ₂₇	119.3(2)
C ₁₀ C ₉ C ₈	120.2(2)	C ₂₈ C ₂₉ C ₃₀	120.9(2)
C ₉ C ₁₀ C ₁₁	120.0(2)	C ₂₉ C ₃₀ C ₂₅	120.5(2)
C ₁₂ C ₁₁ C ₁₀	120.5(2)		

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.^b Atoms are labeled in agreement with Figure 1.

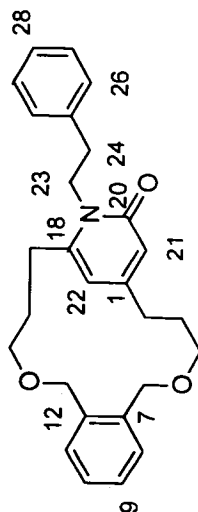
Table 6. Torsion Angles in Crystalline **8**^a

Type ^b	Angle, (deg)	Type ^b	Angle, (deg)
C ₂₁ -C ₁ -C ₂ -O ₃	-107.7(2)	C ₁₇ -C ₁₈ -N ₁₉ -C ₂₀	175.9(2)
C ₂₀ -C ₁ -C ₂ -O ₃	71.7(2)	C ₂₂ -C ₁₈ -N ₁₉ -C ₂₃	175.5(2)
C ₁ -C ₂ -O ₃ -C ₄	71.2(2)	C ₁₇ -C ₁₈ -N ₁₉ -C ₂₃	-7.1(3)
C ₂ -O ₃ -C ₄ -C ₅	-141.7(2)	C ₁₈ -N ₁₉ -C ₂₀ -O ₃₁	178.3(2)
O ₃ -C ₄ -C ₅ -O ₆	77.9(2)	C ₂₃ -N ₁₉ -C ₂₀ -O ₃₁	1.1(2)
C ₄ -C ₅ -O ₆ -C ₇	-179.6(2)	C ₁₈ -N ₁₉ -C ₂₀ -C ₁	-1.1(2)
C ₅ -O ₆ -C ₇ -C ₈	13.9(2)	C ₂₃ -N ₁₉ -C ₂₀ -C ₁	-178.3(2)
C ₅ -O ₆ -C ₇ -C ₁₂	-166.7(2)	C ₂₁ -C ₁ -C ₂₀ -O ₃₁	-175.5(2)
O ₆ -C ₇ -C ₈ -C ₉	178.9(2)	C ₂ -C ₁ -C ₂₀ -O ₃₁	5.1(3)
C ₁₂ -C ₇ -C ₈ -C ₉	-0.4(3)	C ₂₁ -C ₁ -C ₂₀ -N ₁₉	3.8(2)
C ₇ -C ₈ -C ₉ -C ₁₀	0.8(3)	C ₂ -C ₁ -C ₂₀ -N ₁₉	-175.6(2)
C ₈ -C ₉ -C ₁₀ -C ₁₁	0.1(3)	C ₂₀ -C ₁ -C ₂₁ -C ₂₂	-4.1(3)
C ₉ -C ₁₀ -C ₁₁ -C ₁₂	-1.5(3)	C ₂ -C ₁ -C ₂₁ -C ₂₂	175.3(2)
C ₁₀ -C ₁₁ -C ₁₂ -O ₁₃	-177.6(2)	N ₁₉ -C ₁₈ -C ₂₂ -C ₂₁	1.4(3)
C ₁₀ -C ₁₁ -C ₁₂ -C ₇	1.9(3)	C ₁₇ -C ₁₈ -C ₂₂ -C ₂₁	-175.9(2)
O ₆ -C ₇ -C ₁₂ -O ₁₃	-0.8(2)	C ₁ -C ₂₁ -C ₂₂ -C ₁₈	1.4(3)
C ₈ -C ₇ -C ₁₂ -O ₁₃	178.6(1)	C ₁₈ -N ₁₉ -C ₂₃ -C ₂₄	-92.0(2)
O ₆ -C ₇ -C ₁₂ -C ₁₁	179.6(2)	C ₂₀ -N ₁₉ -C ₂₃ -C ₂₄	85.1(2)
C ₈ -C ₇ -C ₁₂ -C ₁₁	-0.9(3)	N ₁₉ -C ₂₃ -C ₂₄ -C ₂₅	-171.1(2)
C ₁₁ -C ₁₂ -O ₁₃ -C ₁₄	-18.2(2)	C ₂₃ -C ₂₄ -C ₂₅ -C ₂₆	103.0(2)
C ₇ -C ₁₂ -O ₁₃ -C ₁₄	162.3(2)	C ₂₃ -C ₂₄ -C ₂₅ -C ₃₀	-74.6(2)
C ₁₂ -O ₁₃ -C ₁₄ -C ₁₅	-174.1(1)	C ₃₀ -C ₂₅ -C ₂₆ -C ₂₇	0.1(3)
O ₁₃ -C ₁₄ -C ₁₅ -O ₁₆	-79.9(2)	C ₂₄ -C ₂₅ -C ₂₆ -C ₂₇	-177.5(2)
C ₁₄ -C ₁₅ -O ₁₆ -C ₁₇	140.2(2)	C ₂₅ -C ₂₆ -C ₂₇ -C ₂₈	0.6(3)
C ₁₅ -O ₁₆ -C ₁₇ -C ₁₈	-70.4(2)	C ₂₆ -C ₂₇ -C ₂₈ -C ₂₉	-0.8(4)
O ₁₆ -C ₁₇ -C ₁₈ -C ₂₂	110.0(2)	C ₂₇ -C ₂₈ -C ₂₉ -C ₃₀	0.4(3)
O ₁₆ -C ₁₇ -C ₁₈ -N ₁₉	-67.3(2)	C ₂₈ -C ₂₉ -C ₃₀ -C ₂₅	0.3(3)
C ₂₂ -C ₁₈ -N ₁₉ -C ₂₀	-1.5(3)	C ₂₆ -C ₂₅ -C ₃₀ -C ₂₉	-0.6(3)
C ₂₄ -C ₂₅ -C ₃₀ -C ₂₉	177.0(2)		

^a The numbers in parentheses are the estimated standard deviations in the last significant digit.^b Atoms are labeled in agreement with Figure 1.

Compound 6m: ¹H NMR (500 MHz)

LRB 19007-20-6



Compound 6m

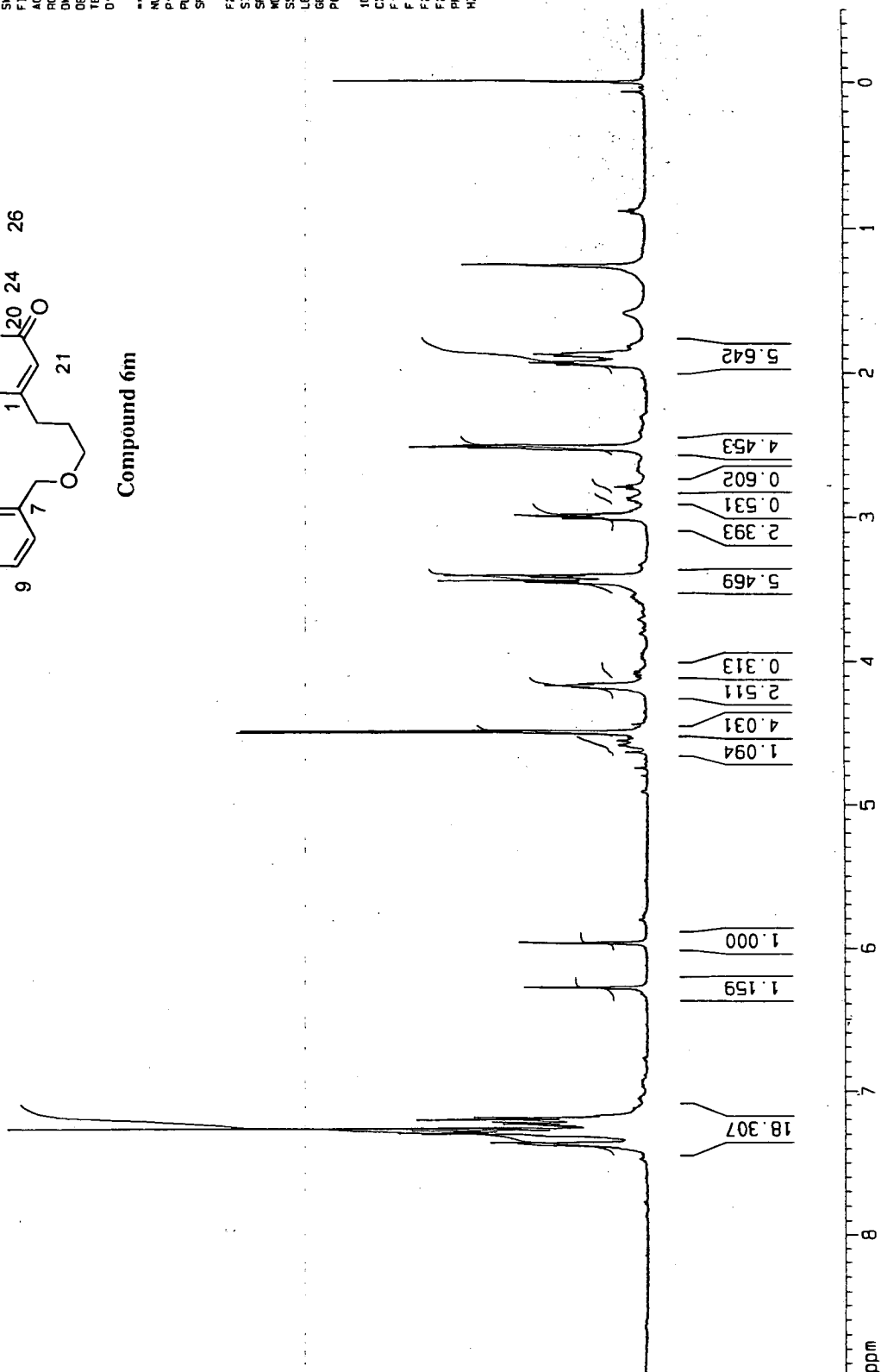
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 NAME 500_May09-2003
 EXPNO 80
 PROCNO 1

F2 - Acquisition Parameters
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 Time 15:57
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 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SFORES 5000.000 Hz
 FIDRES 0.076294 Hz
 AQ 6.5536499 sec
 RG 287.4
 DM 100.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 14.50 usec
 PL1 6.00 dB
 SF01 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300131 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 FIP 9.500 ppm
 F1 4751.23 Hz
 F2 -0.500 ppm
 F2 -250.07 Hz
 PPMCM 0.45455 ppm/cm
 HZCM 227.33182 Hz/cm



Compound 6m: COSY

LRB 19007-20-6

Current Data Parameters
NAME 500_May09-2003
EXPNO 81
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030509
Time 16.01
INSTRUM spect
PROBHD 5 mm QNP-1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1
DS 8
SWH 4464.286 Hz
FIDRES 2.179827 Hz
AQ 0.2294260 sec
RG 143.7
DM 112.000 usec
DE 6.00 usec
TE 300.0 K
D0 0.00000300 sec
D1 1.41070700 sec
D13 0.00000300 sec
D16 0.00015000 sec
D19 0.00022400 sec

CHANNEL f1
NUC1 1H
P1 14.50 usec
PL1 0.00 dB
SFO1 500.1319724 MHz

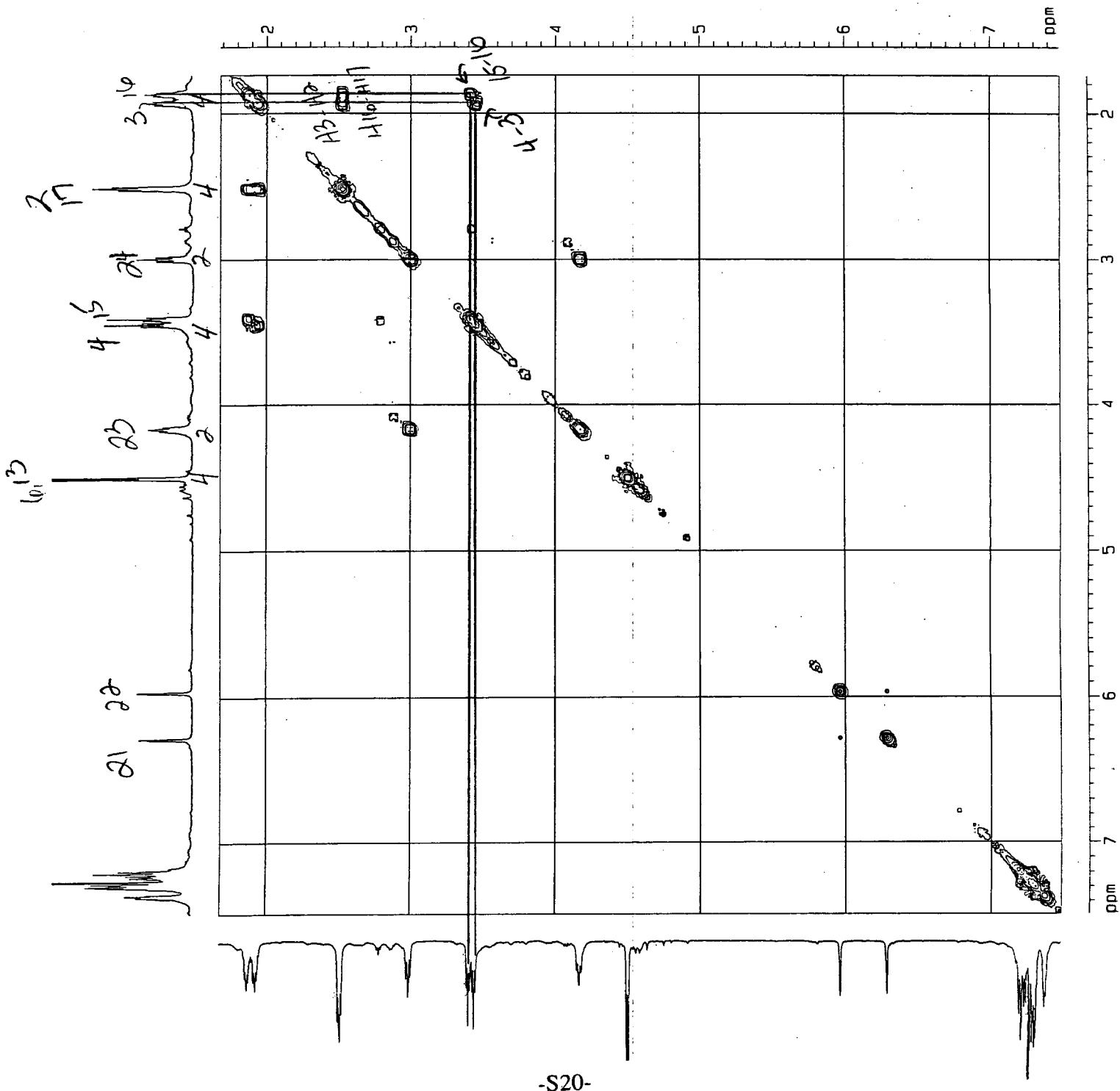
GRADIENT CHANNEL
GPMAX1 SINE 100
GPMAX2 SINE 100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 10.00 %
GPZ2 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
WDW 1
SSB 0
LB 0
GB 0
PC 1.40

F2 - Processing parameters
SI 1024
SF 500.1300108 MHz
WDW SINE
SSB 0
LB 0
GB 0
PC 1.40

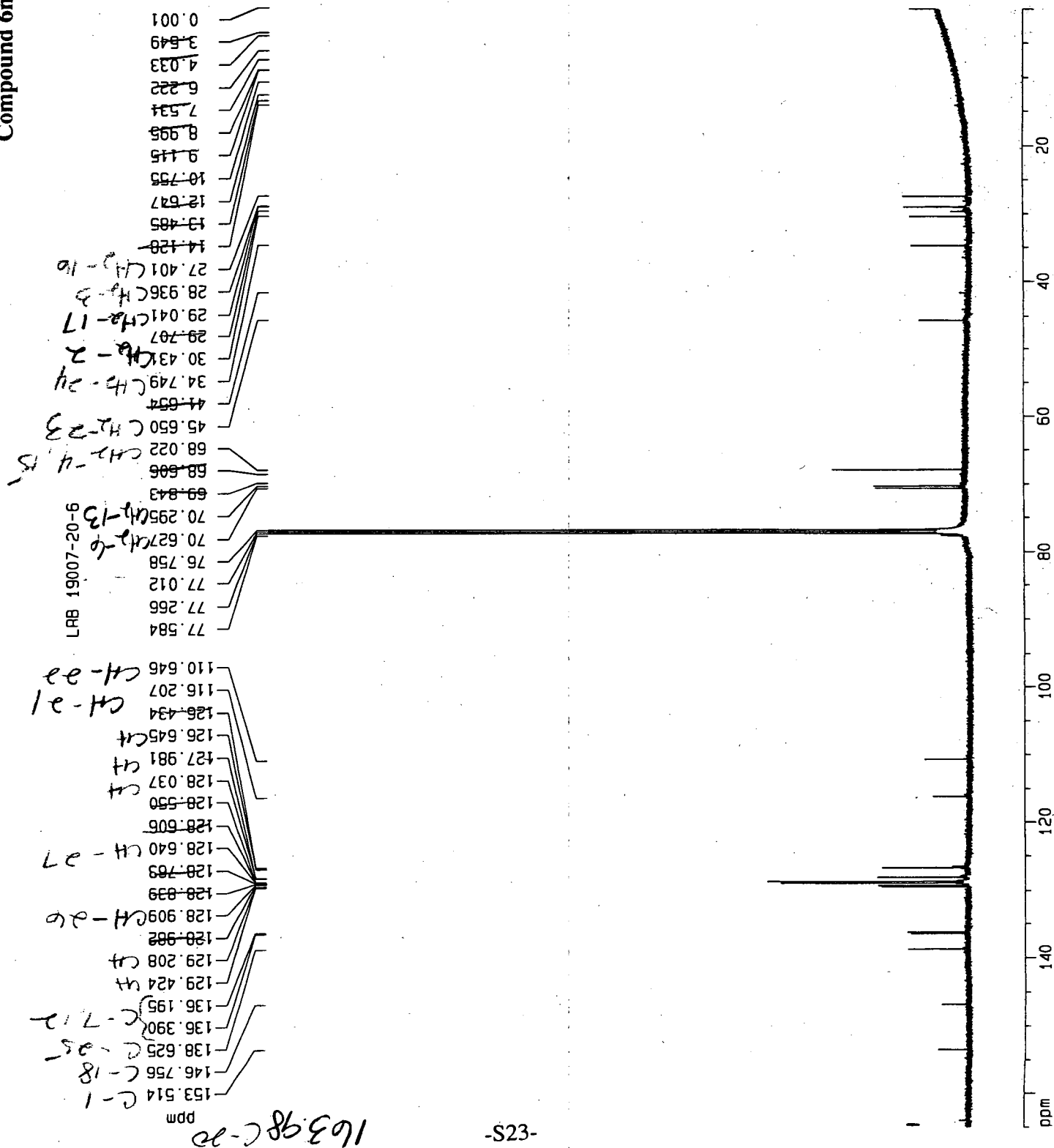
F1 - Processing parameters
SI 1024
SF 500.1300108 MHz
WDW SINE
SSB 0
LB 0
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
FAPLO 7.488 ppm
FALO 3744.74 Hz
FAPHI 1.743 ppm
FALO 871.73 Hz
FAPLO 7.487 ppm
FALO 3744.33 Hz
FAPHI 1.681 ppm
FAPLO 840.80 Hz
FAPHI 0.3857 ppm/cm
FAPLO 191.353 Hz/cm
FAPHI 0.3970 Hz/cm
FAPLO 193.5683 Hz/cm



LAB 19007-20-6



Compound 6m: ^{13}C NMR (125 MHz)

Current Data Parameters
 NAME 500_May09-2003
 EXPNO 85
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030510
 Time 22.54
 INSTRUM spect
 PROBO 5 mm GNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT COC13
 NS 24576
 DS 4
 SWH 22727.273 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418420 sec
 RG 512
 DM 22.000 usec
 DE 6.00 usec
 TE 300.0 K
 d1 1.50000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 ^{13}C
 P1 9.10 usec
 PL1 5.00 dB
 SF01 125.7684284 MHz

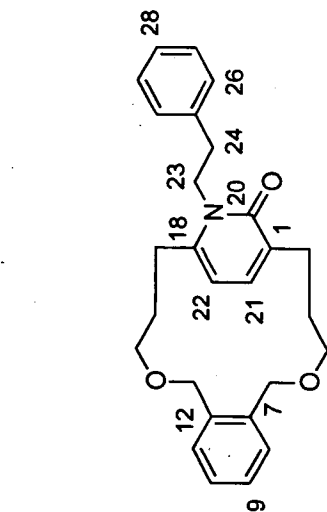
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ^1H
 PCPD2 116.00 usec
 PL2 6.00 dB
 PL12 24.00 dB
 PL13 27.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 65536
 SF 125.7577896 MHz
 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 185.000 ppm
 F1 20750.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 8.25000 ppm/cm
 HZCM 1037.50171 Hz/cm

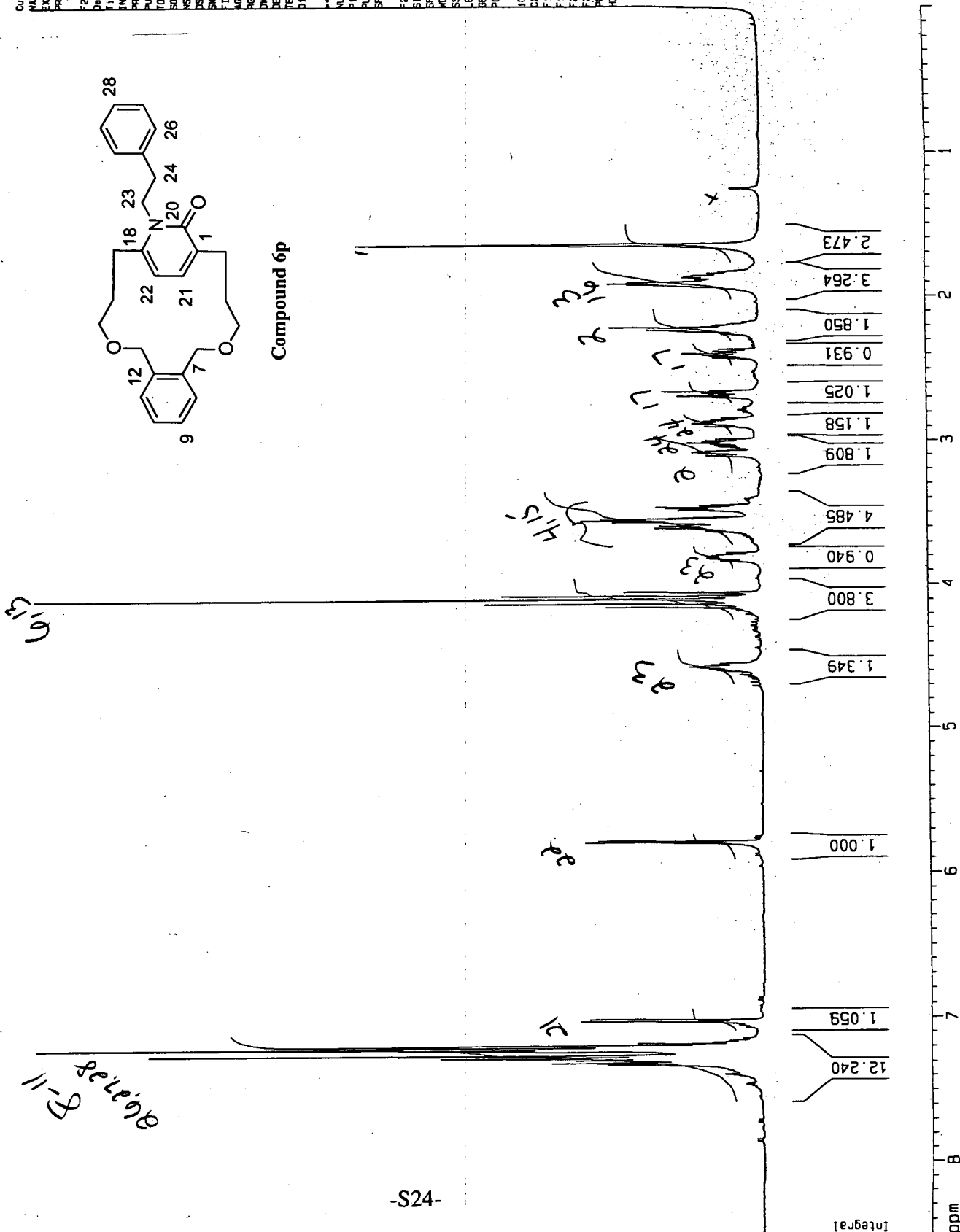
Compound 6p: ¹H NMR (500 MHz)

LRB-19007-20-2A1



Compound 6p

Current Data Parameters
 NAME 500_Jun02-2003
 EXPNO 50
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20030602
 Time 15.52
 INSTRUM spect
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5000.000 Hz
 FIDRES 0.076294 Hz
 AQ 6.5536499 sec
 RG 203.2
 DM 100.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 CHANNEL f1
 NUC1 1H
 P1 14.50 usec
 PL1 6.00 dB
 SF01 500.1324006 MHz
 F2 - Processing parameters
 SI 32768
 SF 500.1300129 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 FID NMR plot parameters
 CX 22.00 cm
 FIP 8.500 ppm
 F1 4251.10 Hz
 F2 0.000 ppm
 F2 0.00 Hz
 PRNCH 0.38636 ppm/cm
 HZCH 193.23204 Hz/cm



Compound 6p: NOESY

LRB-19007-20-2A1

Current Data Parameters
NAME 500_19007-20-2A1
EXPNO 51
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030602
Time 15.54
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TO 1024
SOLVENT CDCl3
NS 8
DS 16
SWH 4370.629 Hz
FIDRES 4.268193 Hz
AQ 0.1171956 sec
RG 143.7
DE 114.400 uSec
TE 300.2
AQ 0.0005559 sec
D1 1.95617294 sec
D8 0.50000000 sec
D16 0.00015000 sec
D20 0.24885000 sec
IND 0.00011440 sec

CHANNEL f1

NUC1 1H
P1 14.50 uSec
P2 29.00 uSec
PL1 1.50 dB
PL2 1.50 dB
SFO1 500.1318817 MHz

GRADIENT CHANNEL

GAMMA1 SINE 100
GAMMA2 SINE 100
DPX1 0.00 %
DPX2 0.00 %
DPY1 0.00 %
DPY2 0.00 %
P16 -40.00 %
P18 1000.00 uSec

F1 - Acquisition Parameters

NUC2 129
TO 128
SFO1 500.1319 MHz
FIDRES 34.149542 Hz
SN 8.739 dB

F2 - Processing Parameters

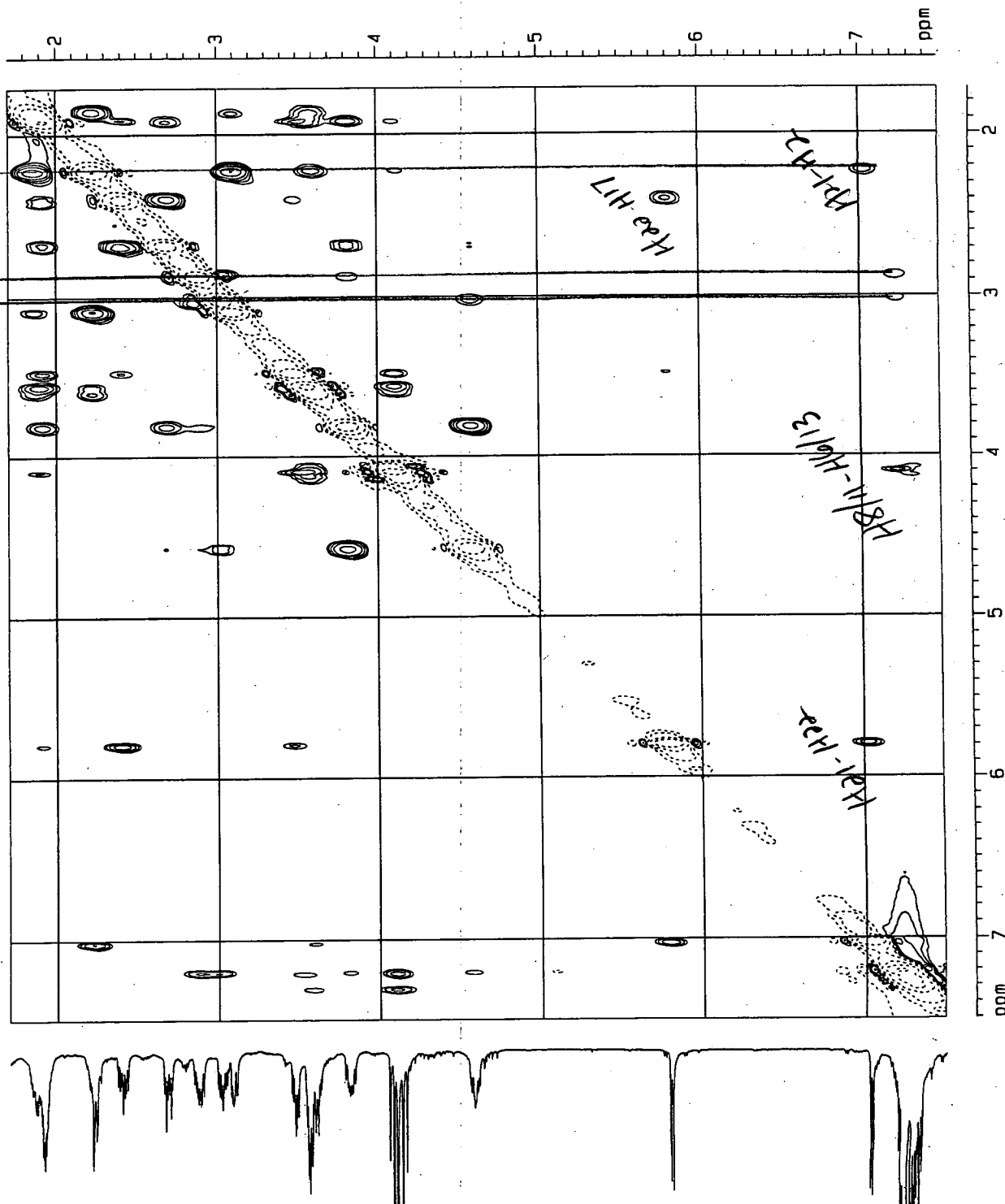
SF 500.1300129 MHz
WDW HANN
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing Parameters

SF 500.1300129 MHz
WDW HANN
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters

CX2 15.00 cm
CX1 15.00 cm
F2L0 7.305 dB
F2L1 3725.32 Hz
F2H1 1.14 cm
F2L2 7.305 dB
F2H2 3746.79 Hz
F1H1 1.705 dB
F1H2 852.95 Hz
F2PCDH 0.38631 dB/cm
F2PCD1 193.20686 Hz/cm
F1PCDH 0.38074 dB/cm
F1PCD1 192.92230 Hz/cm



Compound 6p: Inverse HETCOR

LRB-19007-20-2A1

Current Data Parameters
NAME LRB-19007-20-2A1
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030710
Time 16.30
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
AQ 1.000000
SOLVENT CDCl3
NS 8
DS 8
SWH 470.859 Hz
F2 125.760 MHz
AQ 0.224312 sec
RG 18390.4
DM 114.400 usec
DE 8.00 usec
TE 300.2 K
CLOCK 145.000000
CH012 0.0000330 sec
D1 1.4700000 sec
d2 0.0034000 sec
d3 0.0000000 sec
d18 0.0015000 sec
d20 0.0024250 sec
d40 0.0003313 sec

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

NUC1 1H
P1 14.50 usec
PL 0.00 dB
PR 1.00 usec
SFO1 500.1318017 MHz

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

CPDPRG2 zgpg30
NUC2 13C
P2 9.00 usec
PL 0.00 dB
PR 1.00 usec
SFO2 125.7601200 MHz

===== GRADIENT CHANNEL =====

GP1M1 SINE 100
SFO1M1 125.7601200 MHz
SFO1M2 125.7601200 MHz
GPR1 0.00 %
GPR2 0.00 %
GPR3 0.00 %
GPR4 0.00 %
GPR5 0.00 %
GPR6 0.00 %
GPR7 0.00 %
GPR8 0.00 %
GPR9 0.00 %
GPR10 0.00 %
GPR11 0.00 %
GPR12 0.00 %
GPR13 0.00 %
GPR14 0.00 %
GPR15 0.00 %
GPR16 0.00 %
GPR17 0.00 %
GPR18 0.00 %
GPR19 0.00 %
GPR20 0.00 %
GPR21 0.00 %
GPR22 0.00 %
GPR23 0.00 %
GPR24 0.00 %
GPR25 0.00 %
GPR26 0.00 %
GPR27 0.00 %
GPR28 0.00 %
GPR29 0.00 %
GPR30 0.00 %
GPR31 0.00 %
GPR32 0.00 %
GPR33 0.00 %
GPR34 0.00 %
GPR35 0.00 %
GPR36 0.00 %
GPR37 0.00 %
GPR38 0.00 %
GPR39 0.00 %
GPR40 0.00 %
GPR41 0.00 %
GPR42 0.00 %
GPR43 0.00 %
GPR44 0.00 %
GPR45 0.00 %
GPR46 0.00 %
GPR47 0.00 %
GPR48 0.00 %
GPR49 0.00 %
GPR50 0.00 %
GPR51 0.00 %
GPR52 0.00 %
GPR53 0.00 %
GPR54 0.00 %
GPR55 0.00 %
GPR56 0.00 %
GPR57 0.00 %
GPR58 0.00 %
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GPR62 0.00 %
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GPR64 0.00 %
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GPR66 0.00 %
GPR67 0.00 %
GPR68 0.00 %
GPR69 0.00 %
GPR70 0.00 %
GPR71 0.00 %
GPR72 0.00 %
GPR73 0.00 %
GPR74 0.00 %
GPR75 0.00 %
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GPR77 0.00 %
GPR78 0.00 %
GPR79 0.00 %
GPR80 0.00 %
GPR81 0.00 %
GPR82 0.00 %
GPR83 0.00 %
GPR84 0.00 %
GPR85 0.00 %
GPR86 0.00 %
GPR87 0.00 %
GPR88 0.00 %
GPR89 0.00 %
GPR90 0.00 %
GPR91 0.00 %
GPR92 0.00 %
GPR93 0.00 %
GPR94 0.00 %
GPR95 0.00 %
GPR96 0.00 %
GPR97 0.00 %
GPR98 0.00 %
GPR99 0.00 %
GPR100 0.00 %

F1 - Acquisition Parameters

NUC1 1H
P1 14.50 usec
PL 0.00 dB
PR 1.00 usec
SFO1 500.1318017 MHz

F2 - Processing Parameters

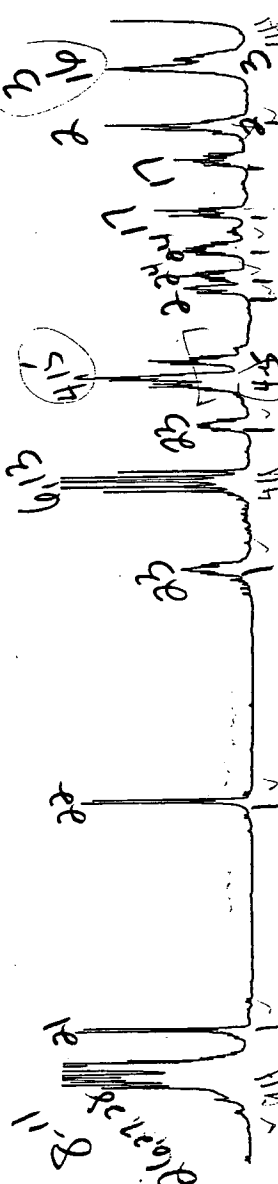
SI 500.130129 MHz
SF 500.130129 MHz
WDW EM
SSB 0
GB 0
PC 1.00

F1 - Processing Parameters

SI 500.130129 MHz
SF 500.130129 MHz
WDW EM
SSB 0
GB 0
PC 1.00

2D NMR Data Parameters

CH 15.00 sec
C1 1.00 sec
F2 1.00 sec
F3 1.00 sec
F4 1.00 sec
F5 1.00 sec
F6 1.00 sec
F7 1.00 sec
F8 1.00 sec
F9 1.00 sec
F10 1.00 sec
F11 1.00 sec
F12 1.00 sec
F13 1.00 sec
F14 1.00 sec
F15 1.00 sec
F16 1.00 sec
F17 1.00 sec
F18 1.00 sec
F19 1.00 sec
F20 1.00 sec
F21 1.00 sec
F22 1.00 sec
F23 1.00 sec
F24 1.00 sec
F25 1.00 sec
F26 1.00 sec
F27 1.00 sec
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F30 1.00 sec
F31 1.00 sec
F32 1.00 sec
F33 1.00 sec
F34 1.00 sec
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F36 1.00 sec
F37 1.00 sec
F38 1.00 sec
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F40 1.00 sec
F41 1.00 sec
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F43 1.00 sec
F44 1.00 sec
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F58 1.00 sec
F59 1.00 sec
F60 1.00 sec
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F62 1.00 sec
F63 1.00 sec
F64 1.00 sec
F65 1.00 sec
F66 1.00 sec
F67 1.00 sec
F68 1.00 sec
F69 1.00 sec
F70 1.00 sec
F71 1.00 sec
F72 1.00 sec
F73 1.00 sec
F74 1.00 sec
F75 1.00 sec
F76 1.00 sec
F77 1.00 sec
F78 1.00 sec
F79 1.00 sec
F80 1.00 sec
F81 1.00 sec
F82 1.00 sec
F83 1.00 sec
F84 1.00 sec
F85 1.00 sec
F86 1.00 sec
F87 1.00 sec
F88 1.00 sec
F89 1.00 sec
F90 1.00 sec
F91 1.00 sec
F92 1.00 sec
F93 1.00 sec
F94 1.00 sec
F95 1.00 sec
F96 1.00 sec
F97 1.00 sec
F98 1.00 sec
F99 1.00 sec
F100 1.00 sec



30.1 31.6 34.6	27.6																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																	</
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LAB-19007-20-2A1

NAME
EXPNO
PROCNO

The figure displays a spectrum plot with a grid. The vertical axis is labeled 'ppm' and ranges from 40 to 160. The horizontal axis represents chemical shift. Handwritten labels on the plot include '107.141-22', '138.9C-25', '145.7C-18', and '163.4C-20'. A list of parameters is provided at the bottom, divided into 'F1 - Acquisition parameters' and 'F2 - Processing parameters'.

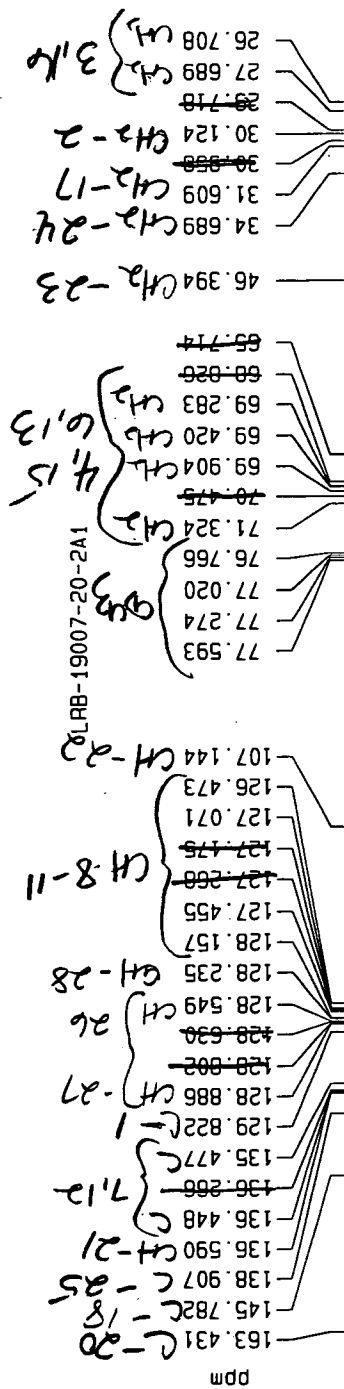
F1 - Acquisition parameters

DATE	20030602
TIME	17:11
INSTRUM	AVANCE
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	65536
TO	20.68
SOLVENT	CDCl3
NS	16
DS	4
SWH	4370.620 Hz
FIDRES	2.134098 Hz
AQ	0.2343412 sec
RG	20642.5
RGF	11.000000
DE	1.000000
TE	300.0 K
CHX2	145.0000000
GB	0.00000000 sec
GB1	0.00000000 sec
GB2	0.00000000 sec
GB3	0.00000000 sec
GB4	0.00000000 sec
GB5	0.00000000 sec
GB6	0.00000000 sec
GB7	0.00000000 sec
GB8	0.00000000 sec
GB9	0.00000000 sec
GB10	0.00000000 sec
GB11	0.00000000 sec
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GB64	0.00000000 sec
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GB67	0.00000000 sec
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GB69	0.00000000 sec
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GB96	0.00000000 sec
GB97	0.00000000 sec
GB98	0.00000000 sec
GB99	0.00000000 sec
GB100	0.00000000 sec

F2 - Processing parameters

SI	2048
SD	500.1300129 MHz
SB	0.00 Hz
SC	0.00 Hz
SD	0.00 Hz
SE	0.00 Hz
SF	500.1300129 MHz
SH	0.00 Hz
SI	2048
SD	500.1300129 MHz
SB	0.00 Hz
SC	0.00 Hz
SD	0.00 Hz
SE	0.00 Hz
SF	500.1300129 MHz
SH	0.00 Hz
SI	2048
SD	500.1300129 MHz
SB	0.00 Hz
SC	0.00 Hz
SD	0.00 Hz
SE	0.00 Hz
SF	500.1300129 MHz
SH	0.00 Hz</

Compound 6p: ¹³C NMR (125 MHz)



Current Data Parameters
 NAME 500_Jun02-2003
 EXPNO 54
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030603
 Time 8.26
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl₃
 NS 12467
 DS 4
 SWH 22727.273 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418420 sec
 RG 256
 DW 22.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00020000 sec

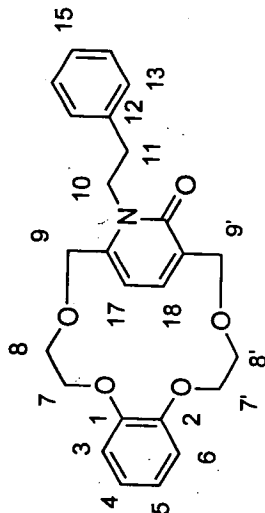
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.10 usec
 PL1 5.00 dB
 SF01 125.7684284 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 116.00 usec
 PL2 6.00 dB
 PL12 24.00 dB
 PL13 27.00 dB
 SF02 500.1320005 MHz

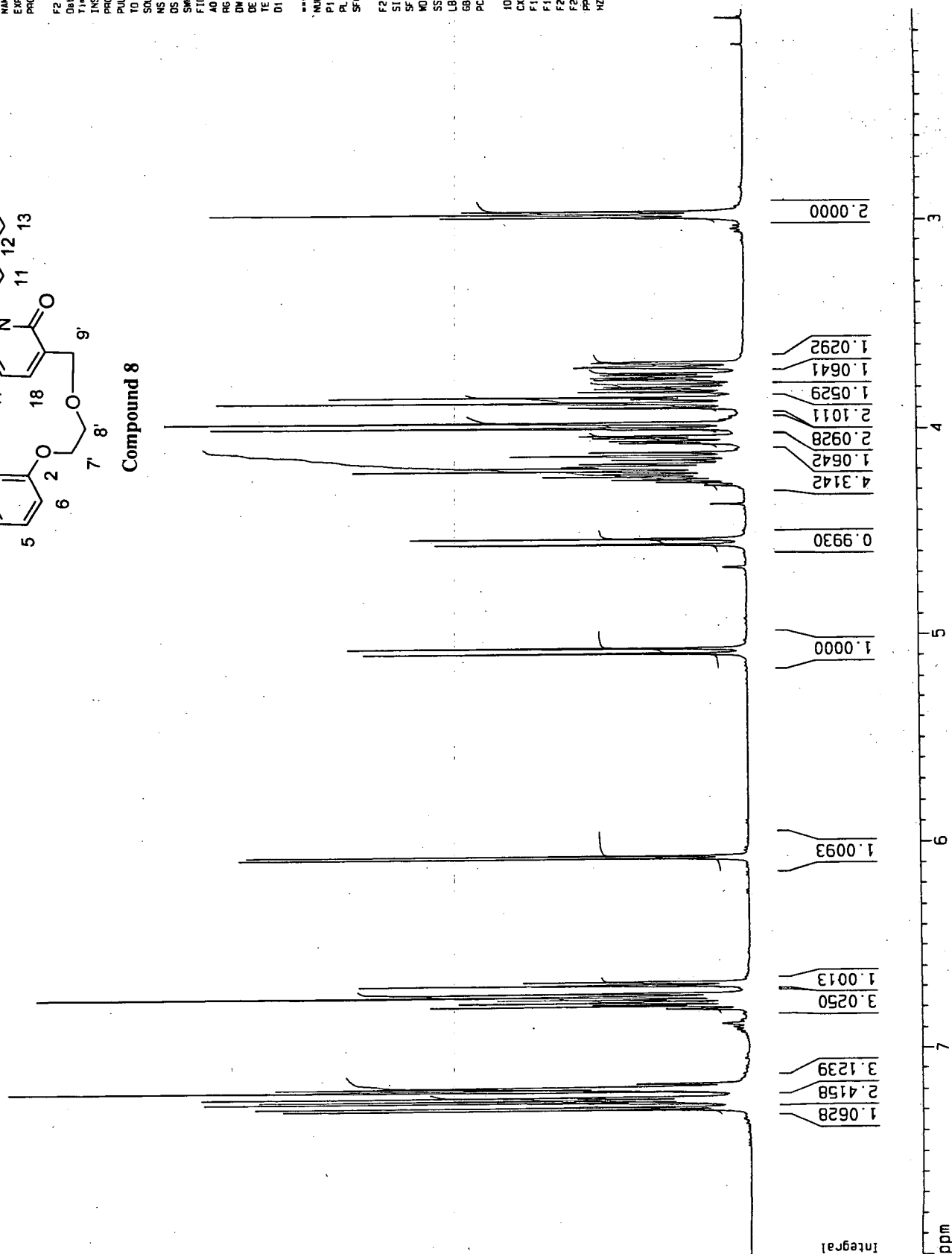
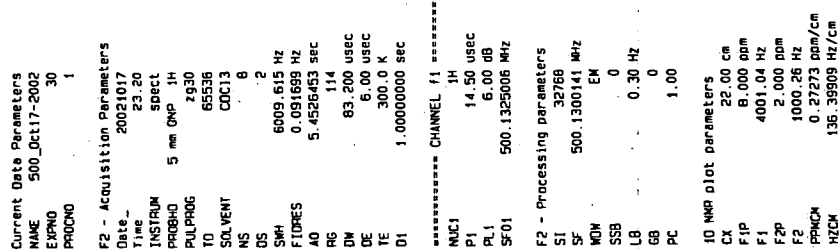
F2 - Processing parameters
 SI 32768
 SF 125.7577909 MHz
 EQ
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 FIP 170.000 ppm
 F1 21378.82 Hz
 F2 0.000 ppm
 F2 0.00 Hz
 PPMCH 8.50000 ppm/cm
 HZCM 1068.94116 Hz/cm

LAB 19007-11-30



Compound 8



Compound 8: NOESY

LRB 19007-11-30

Current Data Parameters
NAME 500_0c17-2002
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters

Date_ 20021017
Time 23.21
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG mzgpgpg0
TD 65536
F2 - Processing parameters
SI 500.1300141 MHz
SF 500.1300141 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Acquisition parameters

NUC1 1H
P1 14.50 uSec
P2 29.00 uSec
PL1 8.00 dB
SF01 500.1318403 MHz

***** GRADIENT CHANNEL *****

GPMH1 SINE:100
GPMH2 SINE:100
GPR1 0.00 %
GPR2 0.00 %
GPR3 0.00 %
GPR4 0.00 %
GPR5 0.00 %
GPR6 0.00 %
GPR7 0.00 %
GPR8 0.00 %
GPR9 0.00 %
GPR10 0.00 %
GPR11 0.00 %
GPR12 0.00 %
GPR13 0.00 %
GPR14 0.00 %
GPR15 0.00 %
GPR16 0.00 %
GPR17 0.00 %
GPR18 0.00 %
GPR19 0.00 %
GPR20 0.00 %
GPR21 0.00 %
GPR22 0.00 %
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GPR93 0.00 %
GPR94 0.00 %
GPR95 0.00 %
GPR96 0.00 %
GPR97 0.00 %
GPR98 0.00 %
GPR99 0.00 %
GPR100 0.00 %

***** CHANNEL 1 *****

NUC1 1H
P1 14.50 uSec
P2 29.00 uSec
PL1 8.00 dB
SF01 500.1318403 MHz

***** GRADIENT CHANNEL *****

GPMH1 SINE:100
GPMH2 SINE:100
GPR1 0.00 %
GPR2 0.00 %
GPR3 0.00 %
GPR4 0.00 %
GPR5 0.00 %
GPR6 0.00 %
GPR7 0.00 %
GPR8 0.00 %
GPR9 0.00 %
GPR10 0.00 %
GPR11 0.00 %
GPR12 0.00 %
GPR13 0.00 %
GPR14 0.00 %
GPR15 0.00 %
GPR16 0.00 %
GPR17 0.00 %
GPR18 0.00 %
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GPR94 0.00 %
GPR95 0.00 %
GPR96 0.00 %
GPR97 0.00 %
GPR98 0.00 %
GPR99 0.00 %
GPR100 0.00 %

***** CHANNEL 1 *****

NUC1 1H
P1 14.50 uSec
P2 29.00 uSec
PL1 8.00 dB
SF01 500.1318403 MHz

***** GRADIENT CHANNEL *****

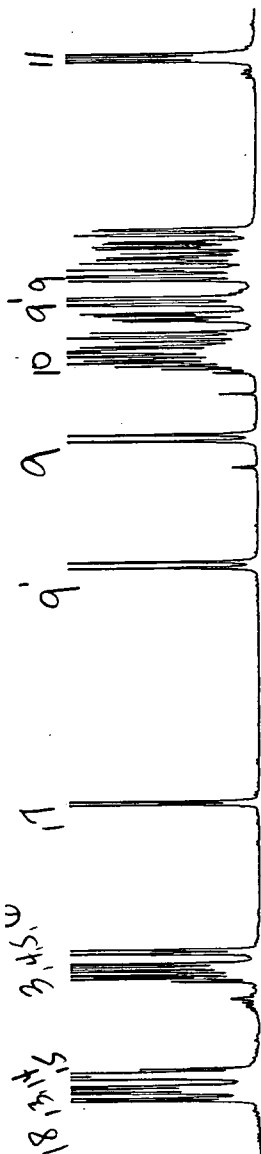
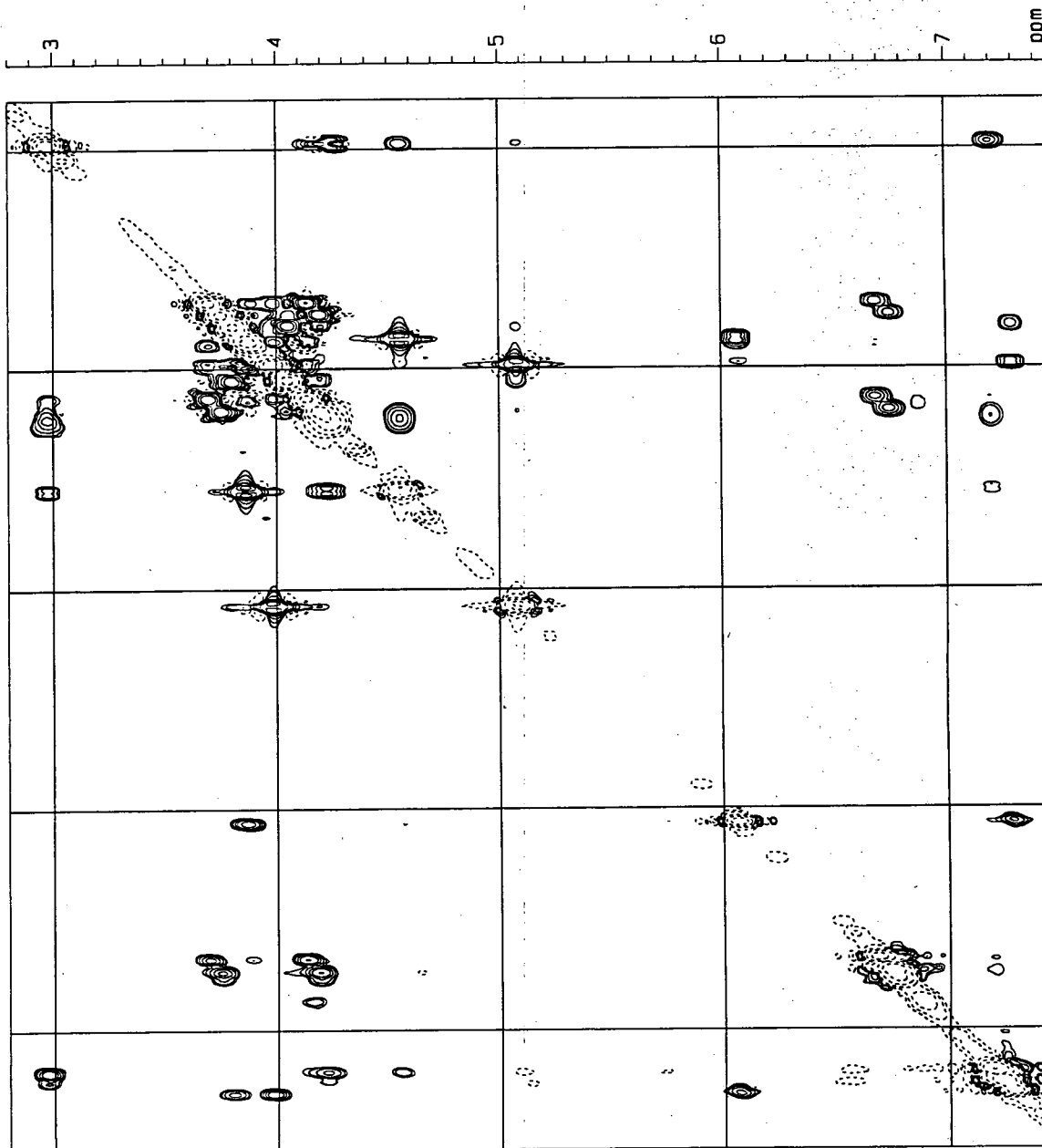
GPMH1 SINE:100
GPMH2 SINE:100
GPR1 0.00 %
GPR2 0.00 %
GPR3 0.00 %
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GPR80 0.00 %
GPR81 0.00 %
GPR82 0.00 %
GPR83 0.00 %
GPR84 0.00 %
GPR85 0.00 %
GPR86 0.00 %
GPR87 0.00 %
GPR88 0.00 %
GPR89 0.00 %
GPR90 0.00 %
GPR91 0.00 %
GPR92 0.00 %
GPR93 0.00 %
GPR94 0.00 %
GPR95 0.00 %
GPR96 0.00 %
GPR97 0.00 %
GPR98 0.00 %
GPR99 0.00 %
GPR100 0.00 %

***** CHANNEL 1 *****

NUC1 1H
P1 14.50 uSec
P2 29.00 uSec
PL1 8.00 dB
SF01 500.1318403 MHz

***** GRADIENT CHANNEL *****

GPMH1 SINE:100
GPMH2 SINE:100
GPR1 0.00 %
GPR2 0.00 %
GPR3 0.00 %
GPR4 0.00 %
GPR5 0.00 %
GPR6 0.00 %
GPR7 0.00 %
GPR8 0.00 %
GPR9 0.00 %
GPR10 0.00 %
GPR11 0.00 %
GPR12 0.00 %
GPR13 0.00 %
GPR14 0.00 %
GPR15 0.00 %
GPR16 0.00 %
GPR17 0.00 %
GPR18 0.00 %
GPR19 0.00 %
GPR20 0.00 %
GPR21 0.00 %
GPR22 0.00 %
GPR23 0.00 %
GPR24 0.00 %
GPR25 0.00 %
GPR26 0.00 %
GPR27 0.00 %
GPR28 0.00 %
GPR29 0.00 %
GPR30 0.00 %
GPR31 0.00 %
GPR32 0.00 %
GPR33 0.00 %
GPR34 0.00 %
GPR35 0.00 %
GPR36 0.00 %
GPR37 0.00 %
GPR38 0.00 %
GPR39 0.00 %
GPR40 0.00 %
GPR41 0.00 %
GPR42 0.00 %
GPR43 0.00 %
GPR44 0.00 %
GPR45 0.00 %
GPR46 0.00 %
GPR47 0.00 %
GPR48 0.00 %
GPR49 0.00 %
GPR50 0.00 %
GPR51 0.00 %
GPR52 0.00 %
GPR53 0.00 %
GPR54 0.00 %
GPR55 0.00 %
GPR56 0.00 %
GPR57 0.00 %
GPR58 0.00 %
GPR59 0.00 %
GPR60 0.00 %
GPR61 0.00 %
GPR62 0.00 %
GPR63 0.00 %
GPR64 0.00 %
GPR65 0.00 %
GPR66 0.00 %
GPR67 0.00 %
GPR68 0.00 %
GPR69 0.00 %
GPR70 0.00 %
GPR71 0.00 %
GPR72 0.00 %
GPR73 0.00 %
GPR74 0.00 %
GPR75 0.00 %
GPR76 0.00 %
GPR77 0.00 %
GPR78 0.00 %
GPR79 0.00 %
GPR80 0.00 %
GPR81 0.00 %
GPR82 0.00 %
GPR83 0.00 %
GPR84 0.00 %
GPR85 0.00 %
GPR86 0.00 %
GPR87 0.00 %
GPR88 0.00 %
GPR89 0.00 %
GPR90 0.00 %
GPR91 0.00 %
GPR92 0.00 %
GPR93 0.00 %
GPR94 0.00 %
GPR95 0.00 %
GPR96 0.00 %
GPR97 0.00 %
GPR98 0.00 %
GPR99 0.00 %
GPR100 0.00 %



Compound 8: COSY

LRB 19007-11-30

Current Data Parameters
NAME 500.DC107-2002
EXPNO 81
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021007
Time 18.20
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 2048
SOLVENT CDCl3
NS 8
DS 4
SWH 4340.278 Hz
FIDRES 2.118278 Hz
AQ 0.2359798 sec
RG 64
DM 115.200 usec
DE 6.00 usec
TE 300.0 K
d0 0.00000300 sec
d1 1.40456200 sec
d13 0.00000300 sec
d15 0.00015000 sec
TM0 0.00023040 sec

Channel f1
NUC1 1H
P1 14.50 usec
PL1 0.00 dB
RF1 500.131072 MHz
SF01 500.131072 MHz

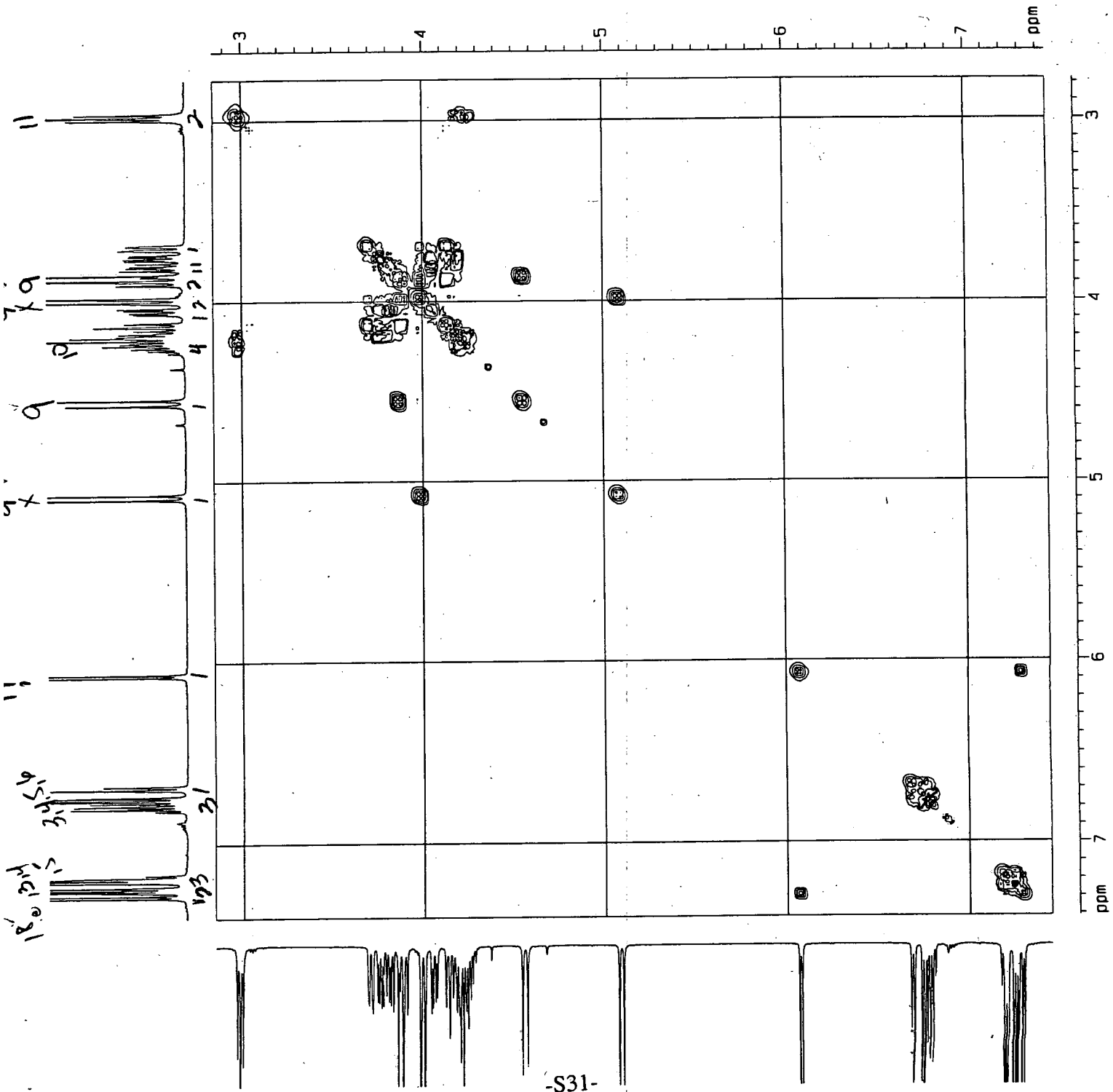
Gradient Channel
GPM1 SINE 100
GPM2 SINE 100
GPX1 0.00 %
GPX2 0.00 %
GPT1 0.00 %
GPT2 0.00 %
GPT3 10.00 %
GPT4 10.00 %
P16 1000.00 usec

F1 - Acquisition Parameters
MD0 1
TD 1328
SF01 500.131072 MHz
FIDRES 33.508423 Hz
Sf 8.676 ppm

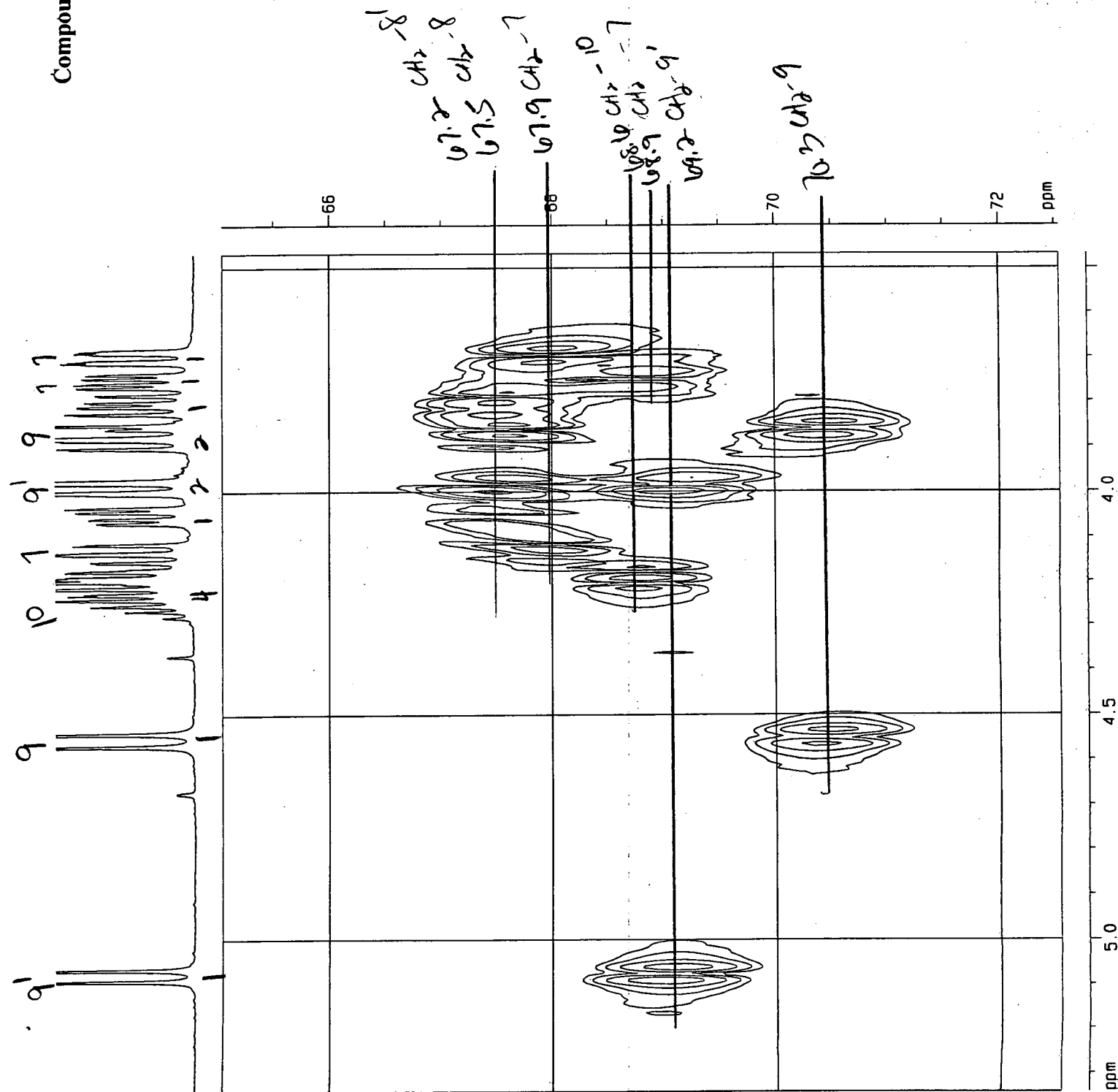
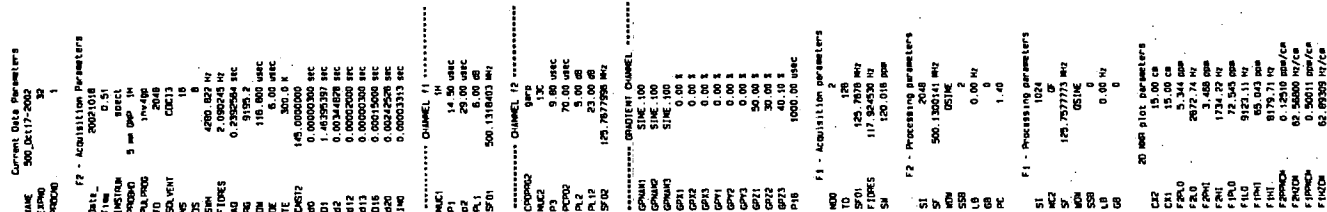
F2 - Processing Parameters
SI 1024
SF 500.1300141 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing Parameters
SI 1024
MC2 0
SF 500.1300141 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

20 MHz plot parameters
C12 15.00 cm
C11 15.00 cm
F2RLO 7.412 ppm
F2LO 3705.82 Hz
F2PH1 2.776 ppm
F2PH1 1388.34 Hz
F1RLO 7.454 ppm
F1LO 3728.02 Hz
F1PH1 2.844 ppm
F1PH1 1422.24 Hz
F2PHCH 0.30905 ppm/cg
F2PHCH 154.55587 Hz/cg
F1PHCH 0.30736 ppm/cg
F1PHCH 153.71817 Hz/cg



LRB 19007-11-3D



Compound 8: HMBC

LRB 19007-11-3D

Current Data Parameters
NAME 500_0117-2002
EXPNO 33
PROCNO 1

F2 - Acquisition Parameters

Date_ 20021016
Time 1:52
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 2048
SOLVENT CDCl3
NS 32
DS 16
SWH 4200.000 Hz
FIDRES 2.000245 Hz
AQ 0.2302564 sec
RG 2042.5
AQ 0.0000000 sec
DE 116.800 UREX
TE 300.2 K
DPC12 145.0000000
d0 0.0000300 sec
d1 1.41400398 sec
d2 0.0334828 sec
d3 0.0000000 sec
d4 0.0000000 sec
d5 0.0000000 sec
d6 0.0019000 sec
d7 0.0002465 sec
TAD 0.0002465 sec

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

NUC1 1H
P1 14.00 UREX
PL1 20.00 UREX
PL2 6.00 dB
SFO1 500.1318403 MHz

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

NUC2 13C
P2 9.00 UREX
PL2 5.00 dB
SFO2 125.7675956 MHz

===== GUNITARY CHANNEL =====

GRAN1 SINE 100
GRAN2 SINE 100
GPH1 0.00 Hz
GPH2 0.00 Hz
GPH3 0.00 Hz
GPH4 0.00 Hz
GPH5 0.00 Hz
GPH6 0.00 Hz
GPH7 0.00 Hz
GPH8 0.00 Hz
GPH9 0.00 Hz
GPH10 0.00 Hz
GPH11 0.00 Hz
GPH12 0.00 Hz
GPH13 0.00 Hz
GPH14 0.00 Hz
GPH15 0.00 Hz
GPH16 0.00 Hz
GPH17 0.00 Hz
GPH18 0.00 Hz
GPH19 0.00 Hz
GPH20 0.00 Hz
GPH21 0.00 Hz
GPH22 0.00 Hz
GPH23 0.00 Hz
GPH24 0.00 Hz
GPH25 0.00 Hz
GPH26 0.00 Hz
GPH27 0.00 Hz
GPH28 0.00 Hz
GPH29 0.00 Hz
GPH30 0.00 Hz
GPH31 0.00 Hz
GPH32 0.00 Hz
GPH33 0.00 Hz
GPH34 0.00 Hz
GPH35 0.00 Hz
GPH36 0.00 Hz
GPH37 0.00 Hz
GPH38 0.00 Hz
GPH39 0.00 Hz
GPH40 0.00 Hz
GPH41 0.00 Hz
GPH42 0.00 Hz
GPH43 0.00 Hz
GPH44 0.00 Hz
GPH45 0.00 Hz
GPH46 0.00 Hz
GPH47 0.00 Hz
GPH48 0.00 Hz
GPH49 0.00 Hz
GPH50 0.00 Hz
GPH51 0.00 Hz
GPH52 0.00 Hz
GPH53 0.00 Hz
GPH54 0.00 Hz
GPH55 0.00 Hz
GPH56 0.00 Hz
GPH57 0.00 Hz
GPH58 0.00 Hz
GPH59 0.00 Hz
GPH60 0.00 Hz
GPH61 0.00 Hz
GPH62 0.00 Hz
GPH63 0.00 Hz
GPH64 0.00 Hz
GPH65 0.00 Hz
GPH66 0.00 Hz
GPH67 0.00 Hz
GPH68 0.00 Hz
GPH69 0.00 Hz
GPH70 0.00 Hz
GPH71 0.00 Hz
GPH72 0.00 Hz
GPH73 0.00 Hz
GPH74 0.00 Hz
GPH75 0.00 Hz
GPH76 0.00 Hz
GPH77 0.00 Hz
GPH78 0.00 Hz
GPH79 0.00 Hz
GPH80 0.00 Hz
GPH81 0.00 Hz
GPH82 0.00 Hz
GPH83 0.00 Hz
GPH84 0.00 Hz
GPH85 0.00 Hz
GPH86 0.00 Hz
GPH87 0.00 Hz
GPH88 0.00 Hz
GPH89 0.00 Hz
GPH90 0.00 Hz
GPH91 0.00 Hz
GPH92 0.00 Hz
GPH93 0.00 Hz
GPH94 0.00 Hz
GPH95 0.00 Hz
GPH96 0.00 Hz
GPH97 0.00 Hz
GPH98 0.00 Hz
GPH99 0.00 Hz
GPH100 0.00 Hz

===== F1 - Acquisition parameters =====

NUC1 1H
P1 14.00 UREX
PL1 20.00 UREX
PL2 6.00 dB
SFO1 500.1318403 MHz

===== F2 - Processing parameters =====

SF 500.130141 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

===== F3 - Processing parameters =====

SF 1024
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

===== F4 - Processing parameters =====

SF 1024
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

===== F5 - Processing parameters =====

SF 1024
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

===== F6 - Processing parameters =====

SF 1024
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

===== F7 - Processing parameters =====

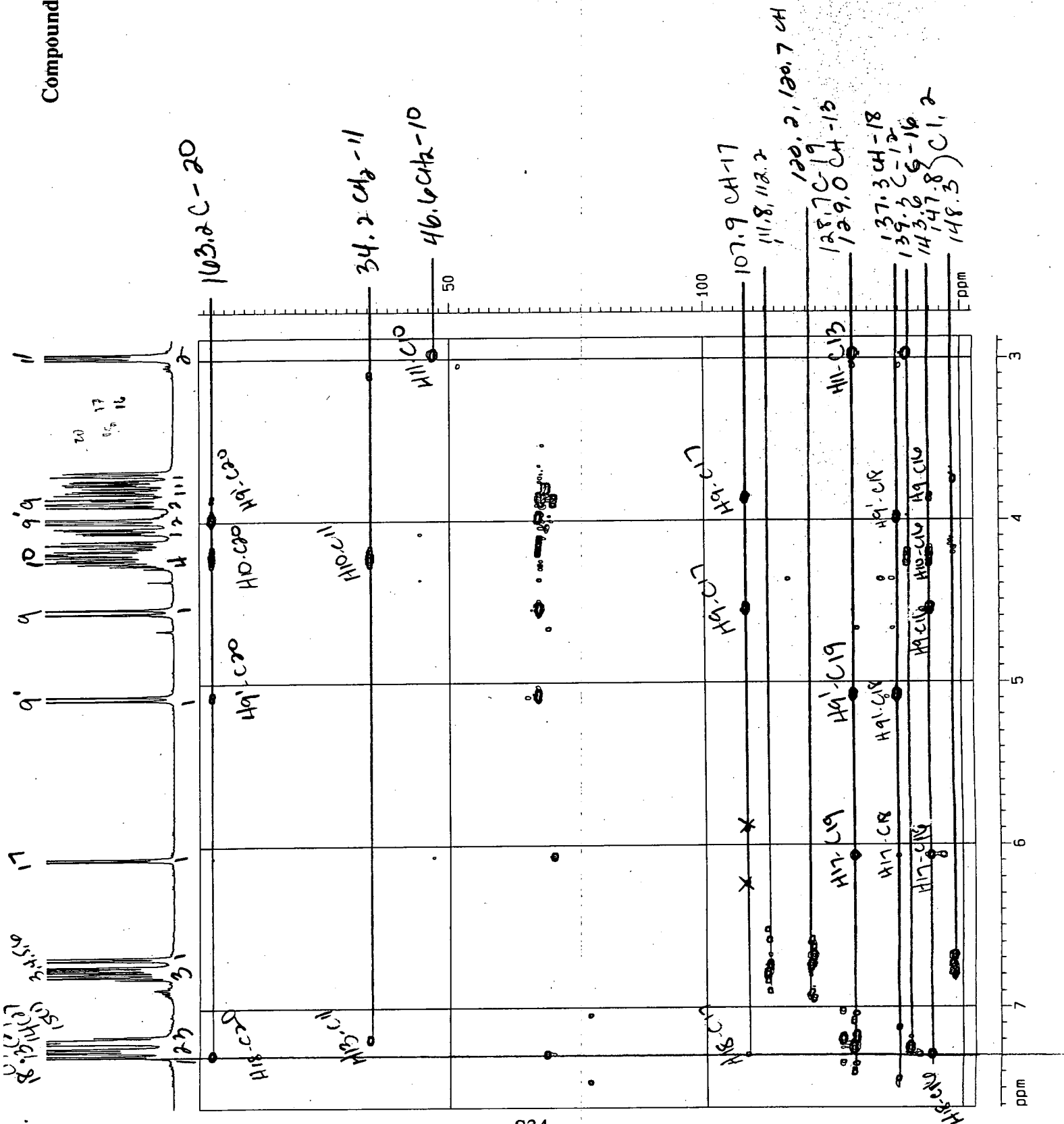
SF 1024
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

===== F8 - Processing parameters =====

SF 1024
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

===== F9 - Processing parameters =====

SF 1024
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40



Compound 8: HMBC

LAB 19007-11-30

Current Data Parameters
500_00117-2002
33

F2 - Acquisition Parameters

Date_ 20021010
Time 1:52
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TO 20.48
SOLVENT CDCl3
NS 32
DS 16
SWH 4200.00 Hz
FIDRES 2.000000 Hz
AQ 0.235264 sec
RG 204.425
GB 116.000 usec
DE 6.00 usec
TE 300.2 K
DPC12 145.000000
d0 0.0000300 sec
d1 1.41460398 sec
d2 0.00344828 sec
d3 0.00000000 sec
d15 0.00000000 sec
d16 0.00015000 sec
TNO 0.00002485 sec

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

NUC1 1H
P1 14.50
PL1 20.00 usec
PR1 8.00 dB
SFO1 500.1310403 MHz

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

NUC2 13C
P2 9.00 usec
PL2 5.00 dB
SFO2 125.7677956 MHz

===== GRABF2 CHANNEL =====

CPDPRG1 SINE 100
CPDPRG2 SINE 100
GPR1 0.00 Hz
GPR2 0.00 Hz
GPR3 0.00 Hz
GPR4 0.00 Hz
GPR5 0.00 Hz
GPR6 0.00 Hz
GPR7 0.00 Hz
GPR8 0.00 Hz
GPR9 0.00 Hz
GPR10 0.00 Hz
GPR11 0.00 Hz
GPR12 0.00 Hz
GPR13 0.00 Hz
GPR14 0.00 Hz
GPR15 0.00 Hz
GPR16 0.00 Hz
GPR17 0.00 Hz
GPR18 0.00 Hz
GPR19 0.00 Hz
GPR20 0.00 Hz
GPR21 0.00 Hz
GPR22 0.00 Hz
GPR23 0.00 Hz
GPR24 0.00 Hz
GPR25 0.00 Hz
GPR26 0.00 Hz
GPR27 0.00 Hz
GPR28 0.00 Hz
GPR29 0.00 Hz
GPR30 0.00 Hz
GPR31 0.00 Hz
GPR32 0.00 Hz
GPR33 0.00 Hz
GPR34 0.00 Hz
GPR35 0.00 Hz
GPR36 0.00 Hz
GPR37 0.00 Hz
GPR38 0.00 Hz
GPR39 0.00 Hz
GPR40 0.00 Hz
GPR41 0.00 Hz
GPR42 0.00 Hz
GPR43 0.00 Hz
GPR44 0.00 Hz
GPR45 0.00 Hz
GPR46 0.00 Hz
GPR47 0.00 Hz
GPR48 0.00 Hz
GPR49 0.00 Hz
GPR50 0.00 Hz
GPR51 0.00 Hz
GPR52 0.00 Hz
GPR53 0.00 Hz
GPR54 0.00 Hz
GPR55 0.00 Hz
GPR56 0.00 Hz
GPR57 0.00 Hz
GPR58 0.00 Hz
GPR59 0.00 Hz
GPR60 0.00 Hz
GPR61 0.00 Hz
GPR62 0.00 Hz
GPR63 0.00 Hz
GPR64 0.00 Hz
GPR65 0.00 Hz
GPR66 0.00 Hz
GPR67 0.00 Hz
GPR68 0.00 Hz
GPR69 0.00 Hz
GPR70 0.00 Hz
GPR71 0.00 Hz
GPR72 0.00 Hz
GPR73 0.00 Hz
GPR74 0.00 Hz
GPR75 0.00 Hz
GPR76 0.00 Hz
GPR77 0.00 Hz
GPR78 0.00 Hz
GPR79 0.00 Hz
GPR80 0.00 Hz
GPR81 0.00 Hz
GPR82 0.00 Hz
GPR83 0.00 Hz
GPR84 0.00 Hz
GPR85 0.00 Hz
GPR86 0.00 Hz
GPR87 0.00 Hz
GPR88 0.00 Hz
GPR89 0.00 Hz
GPR90 0.00 Hz
GPR91 0.00 Hz
GPR92 0.00 Hz
GPR93 0.00 Hz
GPR94 0.00 Hz
GPR95 0.00 Hz
GPR96 0.00 Hz
GPR97 0.00 Hz
GPR98 0.00 Hz
GPR99 0.00 Hz
GPR100 0.00 Hz

===== F1 - Acquisition Parameters =====

NUC1 1H
P1 14.50
PL1 20.00 usec
PR1 8.00 dB
SFO1 500.1310403 MHz

===== F2 - Processing Parameters =====

SI 500.1300141 MHz
SF 2048
MD 2
OS 0.00 Hz
LB 0.00 Hz
GB 0.00 Hz
PC 1.40

===== F1 - Processing Parameters =====

SI 125.7677956 MHz
SF 2048
MD 2
OS 0.00 Hz
LB 0.00 Hz
GB 0.00 Hz
PC 1.40

===== F2 - Processing Parameters =====

SI 125.7677956 MHz
SF 2048
MD 2
OS 0.00 Hz
LB 0.00 Hz
GB 0.00 Hz
PC 1.40

===== F1 - Processing Parameters =====

SI 125.7677956 MHz
SF 2048
MD 2
OS 0.00 Hz
LB 0.00 Hz
GB 0.00 Hz
PC 1.40

===== F2 - Processing Parameters =====

SI 125.7677956 MHz
SF 2048
MD 2
OS 0.00 Hz
LB 0.00 Hz
GB 0.00 Hz
PC 1.40

===== F1 - Processing Parameters =====

SI 125.7677956 MHz
SF 2048
MD 2
OS 0.00 Hz
LB 0.00 Hz
GB 0.00 Hz
PC 1.40

===== F2 - Processing Parameters =====

SI 125.7677956 MHz
SF 2048
MD 2
OS 0.00 Hz
LB 0.00 Hz
GB 0.00 Hz
PC 1.40

