REVISED
10/3

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

Llorente V. R. Boñaga, Han-Cheng Zhang, Diane A. Gauthier,
Indrasena Reddy, and Bruce E. Maryanoff*

*Drug Discovery, Johnson & Johnson Pharmaceutical Research & Development,
Spring House, Pennsylvania 19477-0776 USA*

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[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaen-20-one (6p). ^1H NMR (CDCl_3 , 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). ^{13}C NMR (CDCl_3 , 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 $\text{C}_{27}\text{H}_{31}\text{NO}_3$: 417.54. Found: 418.238755 (MH^+).

19-Phenethyl-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 (8). ^1H NMR (CDCl_3 , 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). ^{13}C NMR (CDCl_3 , 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 $\text{C}_{25}\text{H}_{27}\text{NO}_5$: 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[16.2.2.07,12]docosa-1(21),7(12),8,10,18(22)-pentaene-6,13,20-trione (10). ^1H NMR (CDCl_3 , 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). ^{13}C NMR (CDCl_3 , 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)

-
- ^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

Current Data Parameters
 NAME 500_May09-2003
 EXPNO 80
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030509
 Time 15:57
 INSTRUM spect
 PROBHD 5 mm QNP $^1\text{H}/^1\text{F}$
 PULPROG zg30
 TO 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SHL 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
 FID 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

-S18-

Integral

Compound 6m: NOESY

LRB 19007-20-6

Current Data Parameters
 NAME 500_May09-2003
 EXPNO 84
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030510
 Time 2.03
 INSTRUM spect
 PULPROG zgpg30
 TD 1024
 SOLVENT CDCl3
 NS 8
 DS 16
 SWH 4464.265 Hz
 FIDRES 4.359554 Hz
 AQ 0.1147360 sec
 RG 143.7
 DM 112.000 USEC
 DE 6.00 USEC
 TE 300.0 K
 D1 0.0000000 sec
 D11 1.9582306 sec
 D16 0.5000000 sec
 D20 0.0001500 sec
 d20 0.2488500 sec
 TNO 0.0001200 sec

Channel f1
 NUC1 1H
 P1 14.50 USEC
 PL1 29.00 dB
 SFO1 500.1319724 MHz

Gradient Channel
 GPMAX1 100
 GPMAX2 100
 STIME 1.00
 GPX1 0.00 %
 GPX2 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPZ1 40.00 %
 GPZ2 40.00 %
 P116 100.00 USEC

F1 - Acquisition Parameters
 NUC2 13C
 P2 12.00 USEC
 PL2 19.00 dB
 SFO2 125.7613500 MHz

Processing Parameters
 SI 1024
 SF 500.1300104 MHz
 DS 2
 OSINE 0
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing Parameters
 SI 1024
 SF 125.7613500 MHz
 DS 2
 OSINE 0
 LB 0.00 Hz
 GB 0

2D NMR plot parameters
 CK2 15.00 cm
 CK1 15.00 cm
 F2LO 7.514 ppm
 F2HI 37.586 ppm
 F2F0 7.5 ppm
 F2S1 88.5 Hz
 F2S2 7.472 ppm
 F2F1 3737.21 Hz
 F2H1 1.719 ppm
 F2H2 859.84 Hz
 F2P0CH 0.38239 ppm/cm
 F2MCH 191.24350 Hz/cm
 F2P1CH 0.38355 ppm/cm
 F2M1CH 191.82477 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
 PULPROG 5 mm QNP-1H/1
 TOCPRG TOCPRG
 TOCPRG TOCPRG
 SOLVENT DMS
 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 0.0000300 sec
 D1 1.41070700 sec
 D13 0.0000300 sec
 D16 0.0015000 sec
 TWD 0.00022400 sec

CHANNEL f1
 NUC1 1H
 P0 14.50 usec
 P1 14.50 usec
 PL1 6.00 dB
 SF01 500.1319724 MHz

GRADIENT CHANNEL
 GRNAM1 SINE.100
 GRNAM2 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
 NQD 1
 TD 128
 SF01 500.132 MHz
 FIDRES 34.97232 Hz
 SN 8.926 dB

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
 CK2 15.00 cm
 CK1 15.00 cm
 F2P0 7.488 dB
 F2P0 3744.74 Hz
 F2P0 1.743 dB
 F2P0 871.73 Hz
 F2P0 7.487 dB
 F2P0 3744.33 Hz
 F2P0 1.681 dB
 F2P0 840.80 Hz
 F2P0 0.3857 dB/cm
 F2P0 191.353 Hz/cm
 F2P0 0.3970 Hz/cm
 F2P0 193.5683 Hz/cm

LAB 19007-20-6

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

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
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5000.000 Hz
 FIDRES 0.076294 Hz
 AQ 6.5538499 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
 F3 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_Jun02-2003
EXPNO 51
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030602
Time 15.54
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG noesygpo.09
TO 1024
SOLVENT CDCl3
NS 8
DS 16
SWH 4370.629 Hz
FIDRES 4.268193 Hz
AQ 0.1171956 sec
RG 143.7
RW 114.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.95617294 sec
D8 0.50000000 sec
D16 0.00015000 sec
d20 0.24885000 sec
d20 0.00011440 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

GRAN1 SINE 100
GRAN2 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 %

F1 - Acquisition Parameters

NUC2 129
P1 12.00 usec
P2 12.00 usec
PL1 1.50 dB
PL2 1.50 dB
SFO2 500.130129 MHz

F2 - Processing Parameters

SI 1024
SF 500.130129 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing Parameters

SI 1024
SF 500.130129 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters

CX2 15.00 cm
CX1 15.00 cm
F2L0 7.303 ppm
F2L1 7.303 ppm
F2L2 7.303 ppm
F2L3 7.303 ppm
F2L4 7.303 ppm
F2L5 7.303 ppm
F2L6 7.303 ppm
F2L7 7.303 ppm
F2L8 7.303 ppm
F2L9 7.303 ppm
F2L10 7.303 ppm
F2L11 7.303 ppm
F2L12 7.303 ppm
F2L13 7.303 ppm
F2L14 7.303 ppm
F2L15 7.303 ppm
F2L16 7.303 ppm
F2L17 7.303 ppm
F2L18 7.303 ppm
F2L19 7.303 ppm
F2L20 7.303 ppm
F2L21 7.303 ppm
F2L22 7.303 ppm
F2L23 7.303 ppm
F2L24 7.303 ppm
F2L25 7.303 ppm
F2L26 7.303 ppm
F2L27 7.303 ppm
F2L28 7.303 ppm
F2L29 7.303 ppm
F2L30 7.303 ppm
F2L31 7.303 ppm
F2L32 7.303 ppm
F2L33 7.303 ppm
F2L34 7.303 ppm
F2L35 7.303 ppm
F2L36 7.303 ppm
F2L37 7.303 ppm
F2L38 7.303 ppm
F2L39 7.303 ppm
F2L40 7.303 ppm
F2L41 7.303 ppm
F2L42 7.303 ppm
F2L43 7.303 ppm
F2L44 7.303 ppm
F2L45 7.303 ppm
F2L46 7.303 ppm
F2L47 7.303 ppm
F2L48 7.303 ppm
F2L49 7.303 ppm
F2L50 7.303 ppm
F2L51 7.303 ppm
F2L52 7.303 ppm
F2L53 7.303 ppm
F2L54 7.303 ppm
F2L55 7.303 ppm
F2L56 7.303 ppm
F2L57 7.303 ppm
F2L58 7.303 ppm
F2L59 7.303 ppm
F2L60 7.303 ppm
F2L61 7.303 ppm
F2L62 7.303 ppm
F2L63 7.303 ppm
F2L64 7.303 ppm
F2L65 7.303 ppm
F2L66 7.303 ppm
F2L67 7.303 ppm
F2L68 7.303 ppm
F2L69 7.303 ppm
F2L70 7.303 ppm
F2L71 7.303 ppm
F2L72 7.303 ppm
F2L73 7.303 ppm
F2L74 7.303 ppm
F2L75 7.303 ppm
F2L76 7.303 ppm
F2L77 7.303 ppm
F2L78 7.303 ppm
F2L79 7.303 ppm
F2L80 7.303 ppm
F2L81 7.303 ppm
F2L82 7.303 ppm
F2L83 7.303 ppm
F2L84 7.303 ppm
F2L85 7.303 ppm
F2L86 7.303 ppm
F2L87 7.303 ppm
F2L88 7.303 ppm
F2L89 7.303 ppm
F2L90 7.303 ppm
F2L91 7.303 ppm
F2L92 7.303 ppm
F2L93 7.303 ppm
F2L94 7.303 ppm
F2L95 7.303 ppm
F2L96 7.303 ppm
F2L97 7.303 ppm
F2L98 7.303 ppm
F2L99 7.303 ppm
F2L100 7.303 ppm

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

TD 65536
 SOLVENT CDCl3
 NS 12467

DS 4
 SWH 22727.273 Hz
 FIDRES 0.346791 Hz

RG 256
 DW 22.000 usec
 DE 6.00 usec

TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec

d12 0.00002000 sec

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

CHANNEL f2
 CPOPRG2 waltz16
 NUC2 1H
 PCPO2 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 0

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
 PPMCM 8.50000 ppm/cm
 HZCM 1068.94116 Hz/cm

Compound 8: ^1H NMR (500 MHz)

LRB 19007-11-30

Compound 8

Current Data Parameters
 NAME 500_Oct17-2002
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021017
 Time 23.20
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6009.615 Hz
 FIDRES 0.091699 Hz
 AQ 5.4526453 sec
 RG 114
 DN 83.200 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 500.1325006 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300141 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CX 22.00 cm
 FIP 8.000 ppm
 F1 4001.04 Hz
 F2 2.000 ppm
 FZ 1000.26 Hz
 PPKCH 0.27273 ppm/cm
 HZCM 136.39909 Hz/cm

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 megarpgp2.0g
TD 65536
F2 - Processing parameters
SI 500.1300141 MHz
SF 500.1300141 MHz
WDW EM
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

F1 - Acquisition parameters

NUC1 1H
P1 14.50 uSec
P2 29.00 uSec
PL1 6.00 dB
SF01 500.1318403 MHz

GRADIENT CHANNEL

GP1M1 SINE 100
GP1M2 SINE 100
GP1M3 SINE 100
GP1M4 SINE 100
GP1M5 SINE 100
GP1M6 SINE 100
GP1M7 SINE 100
GP1M8 SINE 100
GP1M9 SINE 100
GP1M10 SINE 100
GP1M11 SINE 100
GP1M12 SINE 100
GP1M13 SINE 100
GP1M14 SINE 100
GP1M15 SINE 100
GP1M16 SINE 100
GP1M17 SINE 100
GP1M18 SINE 100
GP1M19 SINE 100
GP1M20 SINE 100

F2 - Processing parameters

SI 500.1300141 MHz
SF 500.1300141 MHz
WDW EM
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters

SI 1024
SF 500.1300141 MHz
WDW EM
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

20 mag plot parameters

CX2 15.00 cm
CX1 15.00 cm
F2L0 7.547 ppm
F2L1 3774.30 Hz
F2M1 2.774 ppm
F2M2 1387.24 Hz
F2L2 7.480 ppm
F2L3 3740.88 Hz
F2M3 2.799 ppm
F2M4 1389.78 Hz
F2M5 0.31615 ppm/c
F2M6 155.1731 Hz/c
F2M7 155.07162 Hz/c

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 246
SOLVENT CDCl3
NS 8
DS 0
SWH 4340.278 Hz
FIDRES 2.119276 Hz
AQ 0.2359798 sec
RG 64
DM 115.200 usec
DE 6.00 usec
TE 300.0 K
d0 0.0000300 sec
d1 1.4045620 sec
d13 0.0000300 sec
d15 0.0001500 sec
TM0 0.0002300 sec

***** CHANNEL f1 *****

NUC1 1H
P1 14.50 usec
PL1 0.00 dB
RF1 500.131872 MHz
SF01 500.131872 MHz

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

GPNAME1 SINE 100
GPNAME2 SINE 100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 10.00 %
GPZ2 10.00 %
PI16 1000.00 usec

F1 - Acquisition parameters

MD0 1
TO 128
SF01 500.131872 MHz
FIDRES 33.508423 Hz
SN 8.676 ppm

F2 - Processing parameters

SI 1024
SF 500.1300141 MHz
SINE
NS 0
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters

SI 1024
MC2 0
SF 500.1300141 MHz
SINE
NS 0
SSB 0
LB 0.00 Hz
GB 0

2D MAG plot parameters

CX2 15.00 cm
CX1 15.00 cm
F2RLO 7.412 ppm
F2LO 3705.92 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
FAPPCM 0.30905 ppm/cm
F2APPCM 154.55587 Hz/cm
F1APPCM 0.30736 ppm/cm
F1HZCM 153.71817 Hz/cm

-S31-

LRB 19007-11-3D

LAB 19007-11-3D

NAME	500_Oct17-2002
EXPNO	33
PROCNO	1

2 - Acquisition Parameters

[illegible]

***** CHANNEL 11 *****

NUC1	TH
P1	14.50 usec
P2	29.00 usec
PL1	6.00 dB
SFO1	500.1318403 MHz

----- CHANNEL 12 -----

NUC2	13C
P3	9.00 usec
PL2	5.00 dB
SF02	129.7677996 MHz

----- GRADIENT CHANNEL -----

CONTIN	SINE, 100
CONTIN2	SINE, 100
CONTIN3	SINE, 100
CONTIN4	0.00 %

0.00%

GPV1	0.00 %
GPV2	0.00 %
GPV3	0.00 %
GPV4	50.00 %

40, 10 3

```

P15      . 1000.00 usec
F1 - Acquisition parameters
ND0      . 2

```

125.7678 MHz

FIDES	78.596581 Hz
SM	159.983 ppm

F2 - Processing parameters

500,1300141 0017

OSINE
2
0.00 Hz
0

1.40

F3 - Processing parameters

END

0.00 Hz

15.00 cm

CX1	15.00 ea
F2PLO	7.618 ppm
F2LO	3809.84 Hz
F2PHI	2.874 ppm

152.494 ppm

F1LO	19177.33 Hz
F1PHI	0.936 ppm
F1M	117.55 Hz
F2PMCH	0.31624 ppm/cm

10.10391 ppm/cm

1270.64526 Hz/cm
F3H2CM

Compound 8: HMBC

LAB 19007-11-30

Current Data Parameters
500_00117-2002
33

NAME
EXPNO
PROCNO

Date_ 20021010
Time 1:52

INSTRUM spect

PROBHD 5 mm QNP 1H

PULPROG zgpg30

TO 20.48

SOLVENT CDCl3

NS 32

DS 16

SWH 4200.45 Hz

FIDRES 2.000245 Hz

AQ 0.235264 sec

RG 2048.5

DN 116.000 usec

DE 8.00 usec

TE 300.2 K

CHS12 145.000000

AD 0.0000300 sec

DD 1.41460398 sec

DE 0.00344828 sec

DS 0.00500000 sec

DS 0.00500000 sec

D15 0.00015000 sec

D16 0.0002485 sec

INQ 0.0002485 sec

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

NUC1 1H

PC 14.50 usec

PL 20.00 usec

PL1 8.00 dB

PR1 500.1310403 MHz

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

NUC2 13C

PC 9.90 usec

PL 5.00 dB

PR2 125.7677956 MHz

===== CHANNEL f3 =====

===== CHANNEL f4 =====

===== CHANNEL f5 =====

===== CHANNEL f6 =====

===== CHANNEL f7 =====

===== CHANNEL f8 =====

===== CHANNEL f9 =====

===== CHANNEL f10 =====

===== CHANNEL f11 =====

===== CHANNEL f12 =====

===== CHANNEL f13 =====

===== CHANNEL f14 =====

===== CHANNEL f15 =====

===== CHANNEL f16 =====

===== CHANNEL f17 =====

===== CHANNEL f18 =====

===== CHANNEL f19 =====

===== CHANNEL f20 =====

===== CHANNEL f21 =====

===== CHANNEL f22 =====

===== CHANNEL f23 =====

===== CHANNEL f24 =====

===== CHANNEL f25 =====

===== CHANNEL f26 =====

===== CHANNEL f27 =====

===== CHANNEL f28 =====

===== CHANNEL f29 =====

===== CHANNEL f30 =====

===== CHANNEL f31 =====

===== CHANNEL f32 =====

===== CHANNEL f33 =====

===== CHANNEL f34 =====

===== CHANNEL f35 =====

===== CHANNEL f36 =====

===== CHANNEL f37 =====

===== CHANNEL f38 =====

===== CHANNEL f39 =====

===== CHANNEL f40 =====

Compound 8: ^{13}C NMR (125 MHz)