Supporting Information

Highly Enantioselective Construction of a Chiral Spirocyclic Structure by the [2+2+2] Cycloaddition of Diynes and *exo*-Methylene Cyclic Compounds

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General. Optical rotation was measured using Jasco DIP-1000 polarimeter. IR spectra were recorded with Horiba FT-730 spectrophotometer. NMR spectra were measured with JEOL AL-400 and Lambda-500 spectrometers using tetramethylsilane as an internal standard and CDCl₃ was used as solvent. Mass spectra were measured with JEOL JMS-SX102A and elemental analyses with Perkin Elmer PE2400II. Dehydrated 1,2-dichloroethane (DCE) is commercially available. DCE was dried over molecular sieves 4A (MS 4A) and degassed by argon bubbling before use. Diynes **1a**, **b**¹, **1c**², **1d**³, and **1e**¹, and *exo*-methylene compounds **2b**, **g**⁴, **2c**⁵, and **2d**, **e**⁶ were prepared by the literature procedures. **2a** and **2f**, **h** are commercially available. All reactions were examined under argon atmosphere.

Dibenzyl 2-(but-2-ynyl)-2-(3-phenylprop-2-ynyl)malonate (1e). Colorless oil. IR (CH₂Cl₂) 1734, 1169, 698 cm⁻¹; ¹H NMR δ = 1.69 (s, 3H), 3.03 (s, 2H), 3.23 (s, 2H), 5.16 (s, 4H), 7.26-7.28 (m, 15H). ¹³C NMR δ = 3.43, 23.2, 23.6, 57.3, 67.4, 73.0, 79.4, 83.7, 84.0, 123.0, 127.9, 128.0, 128.1, 128.2, 128.4, 131.7, 135.2, 168.8; HRMS for M + 1 found m/z 451.1936, calcd for C₃₀H₂₇O₄: 451.1909.

Typical experimental procedures (Table 2, Entry 1): [Rh(cod){(*S*)-xylyl-binap}]BF₄ (5.4 mg, 0.005 mmol) and α-methylene-γ-butyrolactone **2a** (29.8 mg, 0.30 mmol) were stirred in DCE (0.3 mL) at 80 °C. To the yellow-orange solution, dibenzyl 2,2-di(but-2-ynyl)malonate **1a** (38.4 mg, 0.10 mmol) in DCE (0.7 mL) was added dropwise over 30 min at 80 °C and the mixture was stirred for 5 min. After completion of the reaction, the solvent was removed under reduced pressure, and the crude products were purified by thin-layer chromatography (benzene/AcOEt = 10/1) to give analytically pure **3aa** (45.1 mg, 94%). The ee was determined by HPLC analysis using a chiral column.

Spiro[butano-4-lactone-2,3'-dibenzyl [2,5]dimethylbicyclo[4.3.0]nona[1,5]diene[8,8]dicarboxylate] (3aa). Pale yellow oil. IR (CH₂Cl₂) 1734, 1240, 1169, 698 cm⁻¹; ¹H NMR δ = 1.66 (s, 3H), 1.72 (s, 3H), 1.83-1.90 (m, 1H), 2.02 (d, *J* = 16.8 Hz, 1H), 2.09-2.16 (m, 1H), 2.65 (d, *J* = 16.8 Hz, 1H), 2.94-3.05 (m, 4H), 4.17-4.28 (m, 2H), 5.06-5.17 (m, 4H), 7.23-7.34 (m, 10H). ¹³C NMR δ = 14.9, 19.5, 32.2, 36.8, 37.5, 38.7, 49.4, 59.0, 65.4, 67.2, 67.3, 120.8, 122.3, 127.9, 128.0, 128.3, 128.3, 128.5, 128.5, 130.3, 133.8, 135.4, 135.5, 170.9, 171.1, 181.3; HRMS for M + 1 found m/z 487.2097, calcd for C₃₀H₃₁O₆: 487.2120. [α]_D³⁴ = -22.43 (*c* 0.21, CHCl₃, 97% ee). Ee was determined by HPLC analysis using Daicel Chiralpak AD-H: (eluent: 30% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 10 min for major isomer and 9 min for minor isomer).

Spiro[dibenzyl 2,5-dimethylbicyclo[4.3.0]nona-1,5-diene-8,8-dicarboxylate-3,2'-pentano[5]lactone] (3ab). Pale yellow oil. IR (CH₂Cl₂) 1730, 1240, 1157, 698 cm⁻¹; ¹H NMR δ = 1.34-1.42 (m, 1H), 1.60-1.72 (m, 1H), 1.64 (s, 3H), 1.67 (s, 3H), 1.79-1.89 (m, 2H), 2.19 (d, *J* = 16.6 Hz, 1H), 2.77 (d, *J* = 16.6 Hz, 1H), 2.91-3.04 (m, 4H), 4.21-4.28 (m, 1H), 4.36-4.41 (m, 1H), 5.05-5.17 (m, 4H), 7.23-7.36 (m, 10H). ¹³C NMR δ = 15.8, 19.5, 19.5, 26.8, 36.7, 37.4, 40.8, 50.3, 59.0, 67.1, 67.2, 70.5, 120.2, 125.1, 127.9, 128.0, 128.2, 128.5, 128.5, 129.3, 132.1, 135.4, 135.5, 170.9, 171.2, 176.2; HRMS for *M* + 1 found *m/z* 501.2294, calcd for C₃₁H₃₃O₆: 501.2277. [α]_D²⁵ = -23.60 (*c* 0.91, CHCl₃, 99% ee). Ee was determined by HPLC analysis using Daicel Chiralcel OD-H: (eluent: 50% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 12 min for major isomer and 10 min for minor isomer).

Spiro[dibenzyl 2,5-dimethylbicyclo[4.3.0]nona-1,5-diene-8,8-dicarboxylate-3,2'-hexano[6]lactone] (3ac). Colorless oil. IR (CH₂Cl₂) 1734, 1238, 1184, 1059, 698 cm⁻¹; ¹H NMR δ = 1.56-1.90 (m, 6H), 1.71 (s, 3H x 2), 2.25 (d, *J* = 16.8 Hz, 1H), 2.77 (d, *J* = 16.8 Hz, 1H), 2.93-3.05 (m, 4H), 4.19-4.24 (m, 1H), 4.38-4.42 (m, 1H), 5.06-5.14 (m, 4H), 7.23-7.31 (m, 10H). ¹³C NMR δ = 16.2, 19.5, 21.6, 28.4, 28.9, 36.8, 37.6, 38.6, 55.0, 59.0, 67.1, 67.2, 68.4, 120.4, 126.5, 127.9, 128.2, 128.5, 128.5, 129.7, 131.9, 135.4, 135.5, 171.0, 171.3, 178.8; HRMS for *M* + 1 found *m/z* 515.2427, calcd for C₃₂H₃₅O₆: 515.2434. [α]_D²⁴ = -33.70 (*c* 2.26, CHCl₃, 97% ee). Ee was determined by HPLC analysis using Daicel Chiralpak AD-H: (eluent: 50% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 8 min for major isomer and 10 min for minor isomer).

Spiro[cyclopentanone-2,3'-dibenzyl [2,5]dimethylbicyclo[4.3.0]nona[1,5]diene[8,8]dicarboxylate] (3ad). Colorless oil. IR (CH₂Cl₂) 1734, 1240, 1161, 698 cm⁻¹; ¹H NMR δ = 1.51 (s, 3H), 1.53-1.59 (m, 1H), 1.69 (s, 3H), 1.74-1.99 (m, 3H), 2.13-2.37 (m, 4H), 2.92-3.06 (m, 4H), 5.07-5.17 (m, 4H), 7.23-7.32 (m, 10H). ¹³C NMR δ = 14.8, 18.6, 19.6, 31.3, 36.8, 37.3, 38.1, 39.3, 55.8, 59.1, 67.1, 67.2, 120.5, 124.6, 127.9, 128.2, 128.5, 130.1, 133.3, 135.4, 135.5, 171.1, 171.3, 224.9; HRMS for *M* + 1 found *m/z* 485.2345, calcd for C₃₁H₃₃O₅: 485.2328. [α]_D²⁴ = -20.48 (*c* 0.87, CHCl₃, 81% ee). Ee was determined by HPLC analysis using Daicel Chiralpak AD-H: (eluent: 30% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 12 min for major isomer and 9 min for minor isomer).

Spiro[dibenzyl 2,5-dimethylbicyclo[4.3.0]nona-1,5-diene-8,8-dicarboxylate-3,2'-[1,2,3,4]tetrahydronaphthalen[1]one] (3ae). Yellow oil. IR (neat) 1734, 1674, 1236, 744 cm⁻¹; ¹H NMR δ = 1.57 (s, 3H), 1.72 (s, 3H), 1.82-1.97 (m, 2H), 2.20 (d, *J* = 16.2 Hz, 1H), 2.47 (d, *J* = 16.2 Hz, 1H), 2.75 (dt, *J*_d = 17.3 Hz, *J*_f = 4.3 Hz, 1H), 2.93-3.08 (m, 5H), 5.07-5.19 (m, 4H), 7.21-7.36 (m, 12H), 7.44-7.48 (m, 1H), 8.06 (d, *J* = 7.8 Hz, 1H). ¹³C NMR δ = 15.8, 19.5, 25.1, 28.5, 36.8, 37.2, 37.5, 52.6, 59.1, 67.1, 67.2, 120.5, 126.6, 126.7, 127.1, 127.8, 127.9, 127.9, 128.2, 128.5, 129.7, 132.7, 133.0, 133.2, 133.2, 135.5, 135.6, 143.1, 171.1, 171.4, 202.6; HRMS for *M* + 1 found *m/z* 547.2471, calcd for C₃₆H₃₅O₅: 547.2485. [α]_D²⁴ = -35.60 (*c* 1.91, CHCl₃, 80% ee). Ee was determined by HPLC analysis using Daicel Chiralpak AD-H: (eluent: 50% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 7 min for major isomer and 8 min for minor isomer).

Spiro[butano-4-lactone-2,3'-dibenzyl bicyclo[4.3.0]nona[1,5]diene[8,8]dicarboxylate] (3ba). Pale yellow oil. IR (CH₂Cl₂) 1734, 1238, 1184, 698 cm⁻¹; ¹H NMR δ = 1.95-2.02 (m, 1H), 2.14-2.19 (m, 1H), 2.21-2.27 (m, 1H), 2.72-2.83 (m, 4H), 4.22-4.25 (m, 2H), 5.08-5.15 (m, 4H), 5.43 (s, 1H), 5.55 (bs, 1H), 7.24-7.31 (m, 10H). ¹³C NMR δ = 31.6, 35.8, 38.2, 38.6, 44.2, 59.0, 65.2, 67.2, 67.3, 115.0, 116.6, 127.9, 128.0, 128.3, 128.5, 128.5, 135.2, 135.3, 135.7, 138.6, 170.5, 170.8, 181.1; HRMS for *M* + 1 found *m/z* 459.1821, calcd for C₂₈H₂₇O₆: 459.1808. [α]_D²⁶ = 15.55 (*c* 1.47, CHCl₃, 84% ee). Ee was determined by HPLC analysis using Daicel Chiralpak AD-H: (eluent: 40% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 10 min for major isomer and 13 min for minor isomer).

Spiro[butano-4-lactone-2,3'-[2,5]dimethyl[8]tosyl[8]azabicyclo[4.3.0]nona[1,5]diene] (3ca). White Solid. mp 140 °C (decayed); IR (CH₂Cl₂) 1765, 1344, 1163, 1095, 665 cm⁻¹; ¹H NMR δ = 1.66 (s, 3H), 1.70 (s, 3H), 2.00-2.11 (m, 2H), 2.16-2.21 (m, 1H), 2.44 (s, 3H), 2.60 (d, *J* = 16.8 Hz, 1H), 3.84 (d, *J* = 15.0 Hz, 1H), 3.88 (d, *J* = 15.0 Hz, 1H), 4.06-4.09 (m, 2H), 4.20-4.35 (m, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 8.0 Hz, 2H). ¹³C NMR δ = 15.0, 19.4, 21.5, 32.1, 38.4, 49.1, 50.3, 50.7, 65.3, 121.0, 122.2, 127.4, 127.7, 129.8, 130.9, 133.0, 143.8, 180.6; HRMS for *M* + 1 found *m/z* 374.1443, calcd for C₂₀H₂₄O₄NS: 374.1426. [α]_D²⁴ = -36.66 (*c* 1.68, CHCl₃, 97% ee). Ee was determined by HPLC analysis using Daicel Chiralpak IA: (eluent: 30% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 11 min for major isomer and 12 min for minor isomer).

(R)-Spiro[2,5-dimethyl-8-tosyl-8-azabicyclo[4.3.0]nona-1,5-diene-3,2'-pentano[5]lactone] (3cb). White Solid. mp 180 °C (decayed); IR (KBr disk) 1716, 1342, 1165, 1151, 661 cm⁻¹; ¹H NMR δ = 1.50-1.58 (m, 1H), 1.64 (s, 3H), 1.66 (s, 3H), 1.71-1.75 (m, 1H), 1.84-1.94 (m, 2H), 2.24 (d, *J* = 16.8 Hz, 1H), 2.44 (s, 3H), 2.72 (d, *J* = 16.8 Hz, 1H), 3.81 (d, *J* = 12.2 Hz, 1H), 3.84 (d, *J* = 12.2 Hz, 1H), 4.06-4.09 (m, 2H), 4.25-4.32 (m, 1H), 4.39-4.44 (m, 1H), 7.34 (d, *J* = 8.3 Hz, 2H), 7.72 (d, *J* = 8.3 Hz, 2H). ¹³C NMR δ = 15.8, 19.2, 19.4, 21.5, 27.2, 40.7, 50.0, 50.2, 50.6, 70.6, 120.5, 124.8, 126.3, 127.7, 129.2, 129.7, 132.9, 143.7, 175.5; Anal. Calcd for C₂₁H₂₅O₄NS: C, 65.09; H, 6.50; N, 3.61. Found: C, 65.10; H, 6.48; N, 3.61. HRMS for *M* + 1 found *m/z* 388.1617, calcd for C₂₁H₂₆O₄NS: 388.1582. [α]_D²³ = -39.99 (*c* 1.71, CHCl₃, 97% ee). Ee was determined by HPLC analysis using Daicel Chiralpak IA: (eluent: 50% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 17 min for major isomer and 20 min for minor isomer). Crystal data for **3cb**: C₂₁H₂₅NO₄S, *M* = 387.49, monoclinic, space group *P*2₁ (no. 4), *a* = 7.536(5), *b* = 9.322(5), *c* = 13.574(6) Å, β = 90.09(2)°, *V* = 953.6(9) Å³, *T* = 123 K, *Z* = 2, μ(Mo-Kα) = 1.967 cm⁻¹; number of reflections measured: total 9301 and unique 4138 (*R*_{int} = 0.016), *R*1 = 0.045, *wR*2 = 0.124, Flack parameter (Friedel pairs = 1822) -0.04(5).

Spiro[butano-4-lactone-2,3'-[2,5]diethyl[8]oxabicyclo[4.3.0]nona[1,5]diene] (3da). Dark yellow oil. IR (neat) 1766, 1169, 1026, 766 cm⁻¹; ¹H NMR δ = 1.02 (t, *J* = 7.6 Hz, 3H), 1.07 (t, *J* = 7.6 Hz, 3H), 1.90-2.35 (m, 7H), 2.72 (d, *J* = 16.6 Hz, 1H), 4.24-4.30 (m, 1H), 4.34-4.40 (m, 1H), 4.47-4.55 (m, 4H). ¹³C NMR δ = 11.9, 13.3, 24.2, 26.8, 32.6, 37.0, 49.5, 65.3, 69.3, 69.8, 124.4, 126.3, 130.2, 134.2, 181.3; HRMS for *M* + 1 found *m/z* 249.1515, calcd for C₁₅H₂₁O₃: 249.1491. [α]_D²⁷ = -29.25 (*c* 0.61, CHCl₃, 92% ee). Ee was determined by HPLC analysis using Daicel Chiralpak AD-H: (eluent: 20% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 6 min for major isomer and 5 min for minor isomer).

Spiro[butano-4-lactone-2,3'-dibenzyl

[2]methyl[5]phenylbicyclo[4.3.0]nona[1,5]diene[8,8]dicarboxylate] (3ea). Pale yellow oil. IR (CH₂Cl₂) 1734, 1240, 1173, 698 cm⁻¹; ¹H NMR δ = 1.77 (s, 3H), 1.93-2.01 (m, 1H), 2.31 (ddd, *J* = 3.5, 7.2, 13.3 Hz, 1H), 2.51 (d, *J* = 15.9 Hz, 1H), 3.01 (d, *J* = 15.9 Hz, 1H), 3.12 (d, *J* = 15.9 Hz, 1H x 2), 3.23-3.24 (m, 2H), 4.17-4.23 (m, 1H), 4.30 (ddd, *J* = 3.7, 3.7, 3.7 Hz, 1H), 5.03-5.16 (m, 4H), 7.13-7.15 (m, 2H), 7.22-7.36 (m, 13H). ¹³C NMR δ = 15.1, 31.0, 37.0, 37.1, 38.8, 49.9, 59.3, 65.5, 67.3, 124.1, 125.3, 126.9, 127.8, 128.0, 128.2, 128.3, 128.5, 133.0, 134.8, 135.2, 135.3, 140.4, 170.6, 171.0, 181.1; HRMS for *M* found *m/z* 548.2200, calcd for C₃₅H₃₂O₆: 548.2199. [α]_D²⁴ = 75.02 (*c* 2.22, CHCl₃, >99% ee). Ee was determined by HPLC analysis using Daicel Chiralpak AD-H: (eluent: 30% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 11 min for major isomer and 14 min for minor isomer).

8,8-Dibenzyl 3-methyl 2,3,5-trimethylbicyclo[4.3.0]nona-1,5-diene-3,8,8-tricarboxylate (3af). Pale yellow oil. IR (CH₂Cl₂) 1732, 1238, 698 cm⁻¹; ¹H NMR δ = 1.12 (s, 3H), 1.64 (s, 3H), 1.69 (s, 3H), 1.94 (d, *J* = 16.6 Hz, 1H), 2.72 (d, *J* = 16.6 Hz, 1H), 2.69-3.04 (m, 4H), 3.65 (s, 3H), 5.10-5.13 (m, 4H), 7.22-7.36 (m, 10H). ¹³C NMR δ = 15.2, 19.3, 21.0, 36.8, 37.6, 41.6, 48.4, 51.9, 59.0, 67.1, 122.2, 124.6, 127.8, 127.9, 128.2, 128.5, 128.5, 128.9, 132.0, 135.5, 171.2, 171.2, 177.7; HRMS for *M* + 1 found *m/z* 489.2285, calcd for C₃₀H₃₃O₆: 489.2277. [α]_D²³ = 40.59 (*c* 0.74, CHCl₃, >99% ee). Ee was determined by HPLC analysis using Daicel Chiralcel OD-H: (eluent: 10% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 7 min for major isomer and 8 min for minor isomer).

8,8-Dibenzyl 3-methyl 2,5-dimethyl-3-phenylbicyclo[4.3.0]nona-1,5-diene-3,8,8-tricarboxylate (3ag). Colorless oil. IR (CH₂Cl₂) 1732, 1236, 1182, 698 cm⁻¹; ¹H NMR δ = 1.64 (s, 3H x 2), 2.47 (d, J = 16.4 Hz, 1H), 2.93-3.17 (m, 5H), 3.70 (s, 3H), 5.10-5.17 (m, 4H), 7.09-7.33 (m, 15H). ¹³C NMR δ = 17.5, 19.1, 36.8, 37.9, 43.9, 52.1, 58.6, 58.8, 59.1, 67.1, 122.6, 124.0, 126.5, 126.9, 127.5, 127.8, 128.0, 128.2, 128.5, 129.0, 135.5, 135.6, 142.9, 171.1, 171.3, 174.8, 175.2; HRMS for M found m/z 550.2357, calcd for C₃₅H₃₄O₆: 489.2277. $[\alpha]_D^{27}$ = 43.24 (c 1.51, CHCl₃, 93% ee). Ee was determined by HPLC analysis using Daicel Chiralcel OD-H: (eluent: 10% 2-propanol in hexane, flow rate: 0.5 mL/min, retention time: 15 min for major isomer and 17 min for minor isomer).

8,8-Dibenzyl 3-methyl 2,5-dimethylbicyclo[4.3.0]nona-1,5-diene-3,8,8-tricarboxylate (3ah). Yellow oil. IR (CH₂Cl₂) 1734, 1238, 1167, 739 cm⁻¹; ¹H NMR δ = 1.68 (s, 3H), 1.75 (s, 3H), 2.28-2.35 (m, 1H), 2.49 (dd, J = 6.7, 16.9 Hz, 1H), 2.95-3.07 (m, 5H), 3.64 (s, 3H), 5.11-5.12 (m, 4H), 7.21-7.31 (m, 10H). ¹³C NMR δ = 18.3, 19.2, 32.7, 36.9, 37.3, 45.7, 51.7, 58.9, 67.0, 67.1, 118.9, 122.6, 127.8, 127.8, 128.0, 128.1, 128.4, 128.5, 129.3, 132.9, 135.5, 171.1, 171.3, 174.9; HRMS for M found m/z 474.2040, calcd for C₂₉H₃₀O₆: 474.2042. $[\alpha]_D^{28}$ = 73.30 (c 1.88, CHCl₃, 91% ee). Ee was determined by HPLC analysis using Daicel Chiralcel OD-H: (eluent: 5% 2-propanol in hexane, flow rate: 0.5 mL/min, retention time: 25 min for major isomer and 27 min for minor isomer).

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 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 274
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 26.4 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

3ab
¹³C-NMR

DFILE C:\spiroNMR\3ac-1H.als
 COMNT
 DATIM Thu Apr 27 22:47:59 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 24.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 17

3ac
¹H-NMR

DFILE C:\spiroNMR\3ac-13C.als
 COMNT
 DATIM Thu Apr 27 22:59:04 2006
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 189
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 24.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

3ac
¹³C-NMR

DFILE C:\spiroNMR\3ad-1H.als
 COMNT
 DATIM Mon Jun 05 21:50:08 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 4
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 22.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 16

3ad
¹H-NMR

DFILE C:\spiroNMR\3ad-13C.als
 COMNT
 DATIM Mon Jun 05 22:16:30 2006
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 513
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 23.8 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

3ad
¹³C-NMR

DFILE C:\spiroNMR\3ae-1H.als
 COMNT
 DATIM Wed May 24 10:05:58 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 16
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 24.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 17

3ae
¹H-NMR

DFILE C:\spiroNMR\3ae-13C.als
 COMNT
 DATIM Wed May 24 10:31:50 2006
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 506
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 24.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 24

DFILE C:\spiroNMR\3ba-1H.als
 COMNT
 DATIM Wed Jun 28 16:21:25 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 4
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 22.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 15

3ba
¹H-NMR

DFILE C:\spiroNMR\3ba-13C.als
 COMNT
 DATIM Wed Jun 28 16:30:00 2006
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 130
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 22.6 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 24

3ba
¹³C-NMR

DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFRQ
 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

C:\spiroNMR'
 Mon Feb 27 21:32:34 2006
 1H
 NON
 399.65 MHz
 124.00 KHz
 10500.00 Hz
 32768
 7992.01 Hz
 16
 4.1001 sec
 2.9000 sec
 6.60 usec
 1H
 26.2 c
 CDCL3
 0.00 ppm
 0.12 Hz
 17

3ca
¹H-NMR

DFILE C:\spiroNMR\
COMNT
DATIM Mon Feb 27 10:25:20 2006
OBNUC ¹³C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 513
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 4.60 usec
IRNUC ¹H
CTEMP 25.9 c
SLVNT CDCL₃
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 25

3ca
¹³C-NMR

DFILE C:\spiroNMR\
 COMNT
 DATIM Fri Mar 17 09:48:23 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 4
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 26.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 15

3cb
¹H-NMR

DFILE C:\spiroNMR` 3
 COMNT
 DATIM Fri Mar 17 09:56:18 2006
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 131
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 26.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

3cb
¹³C-NMR

DFILE
 COMNT
 DATIM Sat Apr 15 16:27:14 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 16
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 24.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 16

3da
¹H-NMR

DFILE C:\spiroNMR\ 3
 COMNT
 DATIM Tue May 30 10:30:09 2006
 OBNUC ¹³C
 EXMOD bcm
 OBFRQ 125.40 MHz
 OBSET 140.00 KHz
 OBFIN 2343.75 Hz
 POINT 32768
 FREQU 25000.00 Hz
 SCANS 144
 ACQTM 1.3107 sec
 PD 1.6893 sec
 PW1 5.50 usec
 IRNUC ¹H
 CTEMP 27.3 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 31

3da
¹³C-NMR

DFILE C:\spiroNMR\
 COMNT
 DATIM Fri Jun 02 18:47:09 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 25.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 16

3ea
¹H-NMR

DFILE C:\spiroNMR\
 COMNT
 DATIM Fri Jun 02 18:57:28 2006
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 176
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 24.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

3ea
¹³C-NMR

DFILE C:\spiroNMR\3af-1H.als
 COMNT
 DATIM Thu Feb 02 13:42:08 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 16
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 26.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 19

3af
¹H-NMR

DFILE C:\spiroNMR\3af-13C.als
 COMNT
 DATIM Wed Jan 25 14:02:29 2006
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 201
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 25.9 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

3af
¹³C-NMR

DFILE C:\spiroNMR\3ag-1H.als
 COMNT
 DATIM Sat Jul 08 09:39:23 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 16
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 22.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 14

3ag
¹H-NMR

DFILE C:\spiroNMR\3ag-13C.als
 COMNT Applied
 DATIM Sat Jul 08 09:51:53 2006
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27210.88 Hz
 SCANS 214
 ACQTM 1.2042 sec
 PD 1.7940 sec
 PW1 4.80 usec
 IRNUC ¹H
 CTEMP 22.0 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 24

3ag
¹³C-NMR

DFILE C:\spiroNMR\
 COMNT
 DATIM Fri Apr 14 20:13:43 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 7992.01 Hz
 SCANS 16
 ACQTM 4.1001 sec
 PD 2.9000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 24.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 13

3ah
¹H-NMR

DFILE C:\spiroNMR' 3
 COMNT 1127
 DATIM Fri Apr 14 20:25:07 2006
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 201
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 25.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

3ah
¹³C-NMR