Supporting Information

Rh-Catalyzed Cyclization of Diynes and Enynes Initiated by Carbonyl-Directed Activation of Aromatic and Vinylic C-H Bonds

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General. Optical rotation was measured using Jasco DIP-1000 polarimeter. IR spectra were recorded with Horiba FT-210 spectrophotometer. NMR spectra were measured with JEOL AL-400, Lambda-500, and Bruker Avance-600 spectrometers using CDCl₃ and C₆D₆ as solvent. Tetramethylsilane was used as an internal standard for ¹H and ¹³C NMR, and H₃PO₄ for ³¹P NMR. Mass spectra were measured with JEOL JMS-SX102A and elemental analyses with Perkin Elmer PE2400II. Dehydrated Et₂O, THF, and CH₂Cl₂ are commercially available. CH₂Cl₂ was dried over molecular sieves 4A (MS 4A) and degassed by argon bubbling before use. BIPHEP (2,2'-Bis(diphenylphosphino)biphenyl) was purchased from Strem, and (S)-BINAP from Kanto Chemical Corporation. Diynes **1a**,¹ **1b**,¹ **1c**,² **1d**,³ **1e**,⁴ enynes **4a**,⁵ **4b**,⁵ **4c**,⁶ and enone **2e**⁷ were prepared by the literature procedures. The other substrates are commercially available. All reactions were examined under argon atmosphere.

Preparation of [Rh(cod)(biphep)]BF₄: [Rh(cod)₂]BF₄ (40.3 mg, 0.10 mmol) and BIPHEP (52.3 mg, 0.10 mmol) were dissolved into CH₂Cl₂ (2 mL), and the solution was stirred for 10 min. After removal of the solvent under reduced pressure, the solid was filtered under reduced pressure with Et₂O, and then dried in vacuo over 2 h to give analytically pure [Rh(cod)(biphep)]BF₄ quantitatively as yellow solid. ¹H NMR (CDCl₃) δ = 2.00-2.08 (m, 2H), 2.20-2.26 (m, 2H), 2.50-2.54 (m, 2H), 2.74-2.82 (m, 2H), 4.61-4.65 (m, 2H), 4.71-4.74 (m, 2H), 6.34-6.36 (m, 2H), 6.98-7.68 (m, 26H). ³¹P NMR (CDCl₃) δ = 26.03 (d, *J*_{Rh} = 145.7 Hz); Anal. Calcd for C₄₄H₄₀BF₄P₂Rh: C, 64.41; H, 4.91. Found: C, 64.19; H, 5.05.

(2-((1*E*)-1-((*Z*)-4-ethylidene-1-tosylpyrrolidin-3-ylidene)ethyl)phenyl)(phenyl)methanone (3aa).

White solid. mp 143-144 °C (hexane/acetone). IR (C₆H₆) 1662, 1346, 1163, 665 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.37 (d, *J* = 6.8 Hz, 3H), 1.76 (s, 3H), 2.47 (s, 3H), 3.51-3.54 (m, 2H), 3.93-3.99 (m, 2H), 4.65-4.67 (m, 1H), 7.14-7.69 (m, 13H). ¹³C NMR (CDCl₃) δ = 15.3, 21.5, 23.8, 51.0, 52.3, 121.4, 126.7, 127.8, 127.9, 128.9, 129.1, 129.3, 129.6, 129.7, 130.5, 131.3, 132.4, 132.9, 133.0, 137.6, 137.9, 142.7, 143.5, 197.3; Anal. Calcd for C₂₈H₂₇NO₃S: C, 73.49; H, 5.95; N, 3.06. Found: C, 73.48; H, 6.04; N, 2.98. Crystal data for **3aa**: C₂₈H₂₇NO₃S, *M* = 457.59, monoclinic, space group *P*2₁/*c* (no. 14), *a* = 11.1671(6),

$b = 16.1513(9)$, $c = 13.1889(7)$ Å, $\beta = 95.1550(17)^\circ$, $V = 2369.2(2)$ Å³, $T = 123$ K, $Z = 4$, λ (Mo-K α) = 0.71075 Å; number of reflections measured: total 22720 and unique 5395 ($R_{\text{int}} = 0.032$), $R1 = 0.0389$, $wR2 = 0.1207$. CCDC 647537. The *E/Z* geometry was determined by the X-ray measurements.

Deuterated 3aa (3aa-D). Yellow oil. IR (CDCl₃) 1664, 1346, 1163, 663 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.37 (s, 3H), 1.77 (s, 3H), 2.47 (s, 3H), 3.51-3.55 (m, 2H), 3.93-3.99 (m, 2H), 7.35 (d, $J = 8.0$ Hz, 2H), 7.68 (d, $J = 8.0$ Hz, 2H). ¹³C NMR (CDCl₃) δ = 15.2, 21.5, 23.8, 51.0, 52.4, 127.8, 129.1, 129.7, 130.5, 132.9, 132.9, 137.5, 137.8, 142.6, 143.5, 197.2; HRMS (FAB+) for M found m/z 467.2350, calcd for C₂₈H₁₇D₁₀NO₃S: 467.2339.

1-(2-((1*E*)-1-((*Z*)-4-ethylidene-1-tosylpyrrolidin-3-ylidene)ethyl)phenyl)ethanone (3ab). Yellow oil. IR (CH₂Cl₂) 1687, 1346, 1163, 665 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.33 (d, $J = 7.2$ Hz, 3H), 1.92 (s, 3H), 2.40 (s, 3H), 2.46 (s, 3H), 3.81 (d, $J = 13.4$ Hz, 1H), 3.99 (m, 2H), 4.17 (d, $J = 13.4$ Hz, 1H), 4.39-4.41 (m, 1H), 7.01-7.76 (m, 8H). ¹³C-NMR (CDCl₃) δ = 15.3, 21.5, 23.8, 28.6, 51.5, 52.7, 120.5, 127.3, 127.8, 128.2, 129.2, 129.4, 129.8, 130.8, 132.6, 133.4, 137.1, 142.7, 143.7, 200.1; HRMS (FAB+) for M + 1 found m/z 396.1635, calcd for C₂₃H₂₆NO₃S: 396.1633. The *E/Z* geometry was speculated as below by the geometry of **3aa**.

8-((1*E*)-1-((*Z*)-4-ethylidene-1-tosylpyrrolidin-3-ylidene)ethyl)-3,4-dihydronaphthalen-1(2*H*)-one ((*E*)-3ac). White solid. mp 164-165°C; IR (CH₂Cl₂) 1682, 1346, 1163, 665 cm⁻¹; ¹H NMR (C₆D₆) δ =

1.15 (d, $J = 7.2$ Hz, 3H), 1.53-1.59 (m, 2H), 2.00 (s, 3H), 2.02 (s, 3H), 2.27-2.32 (m, 2H), 2.41-2.45 (m, 2H), 3.97 (d, $J = 14.0$ Hz, 1H), 4.33 (d, $J = 14.0$ Hz, 1H), 4.36 (d, $J = 13.4$ Hz, 1H), 4.56-4.58 (m, 1H), 4.62 (d, $J = 13.4$ Hz, 1H), 6.82-7.93 (m, 7H). ^{13}C NMR (CDCl_3) $\delta = 15.3, 21.5, 23.1, 23.1, 30.7, 40.1, 51.7, 52.7, 119.1, 125.3, 127.8, 127.9, 128.1, 129.7, 129.9, 132.9, 133.1, 133.5, 133.8, 143.6, 144.9, 145.9, 197.7$; HRMS (FAB+) for $\text{M} + 1$ found m/z 422.1785, calcd for $\text{C}_{25}\text{H}_{28}\text{NO}_3\text{S}$: 422.1790.

8-(((1Z)-1-((Z)-4-ethylidene-1-tosylpyrrolidin-3-ylidene)ethyl)-3,4-dihydronaphthalen-1(2H)-one

((Z)-3ac). White solid. mp 149-150 °C; IR (CH_2Cl_2) 1682, 1346, 1163, 665 cm^{-1} ; ^1H NMR (CDCl_3) $\delta = 1.72$ (d, $J = 7.00$ Hz, 3H), 2.06 (s, 2H), 2.08-2.14 (m, 3H), 2.42 (s, 3H), 2.57 (t, $J = 7.5$ Hz, 2H), 2.93-3.07 (m, 2H), 3.31 (d, $J = 13.0$ Hz, 1H), 3.64 (d, $J = 13.0$ Hz, 1H), 3.91 (d, $J = 13.2$ Hz, 1H), 4.03 (d, $J = 13.2$ Hz, 1H), 5.86-5.88 (m, 1H), 6.85-7.59 (m, 7H). ^{13}C NMR (CDCl_3) $\delta = 15.5, 21.5, 22.2, 23.0, 30.6, 40.1, 51.6, 53.0, 121.4, 125.7, 127.3, 127.7, 128.3, 129.3, 129.6, 133.0, 133.1, 134.6, 134.9, 143.4, 145.7, 145.9, 197.7$; HRMS (FAB+) for $\text{M} + \text{Na}$ found m/z 444.1576, calcd for $\text{C}_{25}\text{H}_{27}\text{NO}_3\text{SNa}$: 444.1609. The E/Z geometry was determined by NOESY analysis. (Correlation is depicted by arrow.)

(2-(((Z)-4-benzylidene-1-tosylpyrrolidin-3-ylidene)(phenyl)methyl)phenyl)(phenyl)methanone

(3ba). Yellow oil. IR (CH_2Cl_2) 1662, 1348, 1163, 702 cm^{-1} ; ^1H NMR of major isomer (CDCl_3) $\delta = 2.38$ (s, 3H), 3.85-4.37 (m, 4H), 6.04 (s, 1H), 7.08-7.69 (m, 23H). ^1H NMR of minor isomer (CDCl_3) $\delta = 2.45$ (s, 3H), 3.69-4.44 (m, 4H), 6.16 (s, 1H), 7.08-7.69 (m, 23H). ^{13}C NMR (CDCl_3) $\delta = 21.5$ (major), 21.5 (minor), 51.3 (minor), 51.9 (major), 52.2 (minor), 52.6 (major), 126.6, 127.0, 127.4, 127.4, 127.7, 127.9, 128.0, 128.2, 128.3, 128.4, 128.5, 128.5, 128.7, 128.9, 129.3, 129.3, 129.7, 129.8, 129.8, 129.9, 130.1, 130.2, 130.3, 130.6, 130.9, 131.3, 132.6, 132.8, 132.8, 132.9, 133.1, 133.3, 133.4, 133.7, 134.3, 136.0, 136.3, 136.4, 136.4, 136.5, 136.8, 138.3, 139.2, 140.6, 141.1, 141.8, 142.6, 143.6, 143.6, 196.3 (major), 196.6 (minor); HRMS (FAB+) for $\text{M} + 1$ found m/z 582.2136, calcd for $\text{C}_{38}\text{H}_{32}\text{NO}_3\text{S}$:

582.2103. The *E/Z* geometry of each isomer was determined as below by comparing the chemical shift of the olefinic protons of **3ba** with that of (*E*)-**3ca**.

(2-((1*E*)-((*Z*)-4-ethylidene-1-tosylpyrrolidin-3-ylidene)(phenyl)methyl)phenyl)(phenyl)methanone ((*E*)-3ca**)**. White Solid. mp 150-151 °C; IR (CH₂Cl₂) 1664, 1348, 1163, 704 cm⁻¹; ¹H NMR δ = 1.43 (d, *J* = 7.3 Hz, 3H), 2.48 (s, 3H), 3.64 (d, *J* = 13.9 Hz, 1H), 3.91 (d, *J* = 13.4 Hz, 1H), 4.05-4.08 (m, 2H), 5.11-5.16 (m, 1H), 7.01-7.71 (m, 18H). ¹³C NMR = 15.4, 21.5, 50.5, 53.5, 124.0, 126.5, 127.3, 127.8, 127.8, 129.1, 129.7, 129.9, 130.5, 131.0, 132.1, 132.6, 133.1, 134.3, 134.5, 136.7, 139.2, 141.1, 141.7, 143.6, 196.9; HRMS (FAB⁺) for M + Na found *m/z* 542.1733, calcd for C₃₃H₂₉NO₃SNa: 542.1766. The *E/Z* geometry was determined by NOESY analysis. (Correlation is depicted by arrow.)

(3*Z*,4*E*)-dibenzyl 3-(1-(2-benzoylphenyl)ethylidene)-4-ethylidenecyclopentane-1,1-dicarboxylate (3da). Yellow oil. IR (CH₂Cl₂) 1734, 1664, 1348, 1286, 1242, 700 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.37 (d, *J* = 6.8 Hz, 3H), 1.84 (s, 3H), 2.56 (d, *J* = 17.1 Hz, 1H), 2.61 (d, *J* = 17.2 Hz, 1H), 2.91 (d, *J* = 17.1 Hz, 1H), 3.03 (d, *J* = 17.2 Hz, 1H), 4.67-4.69 (m, 1H), 5.04-5.13 (m, 4H), 7.15-7.82 (m, 19H). ¹³C NMR (CDCl₃) δ = 15.3, 24.0, 37.5, 39.3, 57.0, 67.1, 67.1, 121.7, 126.1, 127.9, 128.0, 128.1, 128.2, 128.4, 128.5, 129.3, 129.5, 129.6, 130.8, 132.4, 134.2, 135.5, 135.5, 136.4, 137.9, 137.9, 144.0, 170.9, 171.3, 197.4; HRMS (FAB⁺) for M - 1 found *m/z* 569.2279, calcd for C₃₈H₃₃O₅: 569.2328. The *E/Z* geometry was speculated by the geometry of **3aa**.

(4*Z*,5*E*)-tetraethyl 4-(1-(2-benzoylphenyl)ethylidene)-5-ethylidenecyclohexane-1,1,2,2-tetracarboxylate ((*Z*)-3ea). White solid. mp 94-95 °C. IR (CH₂Cl₂) 1738, 1261, 1041, 706 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.21-1.31 (m, 15H), 1.93 (s, 3H), 2.72-3.20 (m, 4H), 4.13- 4.34 (m, 8H), 5.22 (m, 1H), 7.15-7.81 (m, 9H). ¹³C NMR (CDCl₃) δ = 13.3, 13.8, 13.9, 21.4, 31.8, 33.6, 59.9, 61.4, 61.5, 61.6, 124.7, 127.0, 128.2, 128.8, 130.1, 130.5, 130.6, 131.2, 132.7, 132.9, 136.8, 169.3, 169.6, 196.9; HRMS (FAB+) for M + Na found m/z 627.2584, calcd for C₃₅H₄₀O₉Na: 627.2570. The *E/Z* geometry was determined by the NOE analyses of (*Z*)-3ea and (*E*)-3ea. (Correlations are depicted by arrows.)

(2*E*,4*E*)-4-((*Z*)-4-ethylidene-1-tosylpyrrolidin-3-ylidene)-1,4-diphenylbut-2-en-1-one (3ad). Yellow oil. IR (CH₂Cl₂) 1599, 1346, 1159, 663 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.76 (d, *J* = 7.0 Hz, 3H), 1.89 (s, 3H), 2.44 (s, 3H), 4.03-4.08 (m, 4H), 5.86-5.90 (m, 1H), 6.46 (s, 1H), 7.02-7.85 (m, 14H). ¹³C NMR (CDCl₃) δ = 15.8, 20.6, 21.8, 51.3, 53.7, 124.6, 125.7, 128.1, 128.4, 128.6, 128.9, 129.0, 129.2, 130.0, 132.2, 133.1, 133.2, 135.2, 137.0, 137.9, 143.9, 155.9, 193.5; HRMS (FAB+) for M + 1 found m/z 484.1929, calcd for C₃₀H₃₀NO₃S: 484.1946. The *E/Z* geometry was determined by NOESY analysis. (Correlations are depicted by arrows.)

(2*E*,4*Z*)-4-((*Z*)-4-ethylidene-1-tosylpyrrolidin-3-ylidene)-1-phenylpent-2-en-1-one (3ae). Colorless oil. IR (neat) 1599, 1346, 1163, 663 cm⁻¹; ¹H NMR (CDCl₃) 1.74 (d, *J* = 7.2 Hz, 3H), 1.96 (s, 3H), 2.22 (s, 3H), 2.44 (s, 3H), 3.98 (s, 2H), 4.01 (s, 2H), 5.80-5.82 (m, 1H), 6.57 (s, 1H), 7.31-7.90 (m, 9H). ¹³C NMR (CDCl₃) δ = 15.5, 18.0, 18.7, 21.5, 51.1, 52.9, 122.1, 123.5, 127.8, 128.1, 128.5, 128.6, 129.7, 132.7, 132.9, 134.8, 134.9, 139.0, 143.7, 158.8, 191.1; HRMS (FAB+) for M + 1 found m/z 422.1743, calcd for C₂₅H₂₈NO₃S: 422.1790. The *E/Z* geometry was determined by NOESY analysis. (Correlations are depicted by arrows.)

(2-(((Z)-4-ethylidene-1-tosylpyrrolidin-3-yl)methyl)phenyl)(phenyl)methanone (5aa). Yellow oil. IR (CH₂Cl₂) 1662, 1346, 1161, 665 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.45 (d, *J* = 5.6 Hz, 3H), 2.41 (s, 3H), 2.59 (dd, *J* = 9.3, 13.4 Hz, 1H), 2.85-2.92 (m, 2H), 2.97-3.02 (m, 1H), 3.11 (dd, *J* = 6.5, 8.9 Hz, 1H), 3.71-3.73 (m, 2H), 5.13-5.14 (m, 1H), 7.24-7.78 (m, 13H). ¹³C NMR (CDCl₃) δ = 14.4, 21.5, 36.5, 44.5, 49.6, 52.9, 117.6, 125.8, 127.8, 128.5, 129.3, 129.6, 130.2, 130.5, 131.1, 133.3, 137.7, 138.2, 138.4, 138.9, 143.5, 198.2; HRMS (FAB+) for M found *m/z* 445.1747, calcd for C₂₇H₂₇O₃NS: 445.1712. [α]_D¹⁸ = -33.93 (*c* 0.96, CHCl₃, 97% ee). Ee was determined by HPLC analysis using Daicel Chiralpak IA: (eluent: 20% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 12 min for major isomer and 10 min for minor isomer).

(2-(((Z)-4-benzylidene-1-tosylpyrrolidin-3-yl)methyl)phenyl)(phenyl)methanone (5ba). Colorless oil. IR (CDCl₃) 1660, 1346, 1165, 663 cm⁻¹; ¹H NMR (CDCl₃) δ = 2.39 (s, 3H), 2.73-2.78 (m, 1H), 3.09-3.12 (m, 4H), 4.00 (d, *J* = 14.8 Hz, 1H), 4.16 (d, *J* = 14.8 Hz, 1H), 6.08 (s, 3H), 7.03-7.80 (m, 18H). ¹³C NMR (CDCl₃) δ = 21.5, 37.0, 46.5, 50.6, 51.6, 123.6, 125.9, 127.0, 127.7, 128.1, 128.5, 128.5, 129.4, 129.7, 130.2, 130.6, 131.3, 132.8, 133.3, 136.4, 137.8, 138.3, 138.6, 139.5, 143.6, 198.2; HRMS (FAB+) for M + 1 found *m/z* 508.1956, calcd for C₃₂H₃₀O₃NS: 508.1946. [α]_D²⁰ = -2.29 (*c* 1.07, 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: 20 min for major isomer and 19 min for minor isomer).

(2E)-4-((Z)-4-ethylidene-1-tosylpyrrolidin-3-yl)-1,3-diphenylbut-2-en-1-one (5ad). Pale yellow oil. IR (CH₂Cl₂) 1599, 1346, 1159, 663 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.49 (d, *J* = 6.6 Hz, 3H), 2.38 (s, 3H), 2.63 (dd, *J* = 9.4, 13.3 Hz, 1H), 2.85-2.97 (m, 2H), 3.10-3.14 (m, 2H), 3.74 (d, *J* = 13.9 Hz, 1H), 3.80 (d, *J* = 13.9 Hz, 1H), 5.23-5.24 (m, 1H), 7.11-7.64 (m, 15H). ¹³C NMR (CDCl₃) δ = 14.4, 21.5, 36.5, 44.4, 49.6, 52.9, 117.6, 126.1, 126.6, 127.8, 128.5, 129.0, 129.5, 130.7, 130.8, 131.3, 134.4, 138.5, 138.7, 138.9, 143.5, 146.1, 196.0; HRMS (FAB+) for M + 1 found *m/z* 472.1953, calcd for C₂₉H₃₀O₃NS: 472.1946. [α]_D²¹ = -44.44 (*c* 0.86, CHCl₃, 97% ee). Ee was determined by HPLC analysis using Daicel Chiralpak IB: (eluent: 20% 2-propanol in hexane, flow rate: 1.0 mL/min, retention time: 9 min for major isomer and 10 min for minor isomer).

(3E)-dibenzyl 3-ethylidene-4-((E)-4-oxo-2,4-diphenylbut-2-enyl)cyclopentane-1,1-dicarboxylate (5cd). Colorless oil. IR (neat) 1732, 1601, 1448, 752, 696 cm⁻¹; ¹H NMR (CDCl₃) δ = 1.55 (d, *J* = 8.4 Hz, 3H), 1.85 (dd, *J* = 10.1, 13.2 Hz, 1H), 2.35 (dd, *J* = 6.1, 13.3 Hz, 1H), 2.52 (dd, *J* = 10.1, 13.2 Hz, 1H), 2.76-2.84 (m, 1H), 2.88 (d, *J* = 17.1 Hz, 1H), 3.00 (d, *J* = 17.1 Hz, 1H), 3.31 (dd, *J* = 6.1, 13.3 Hz, 1H), 4.96-4.98 (m, 2H), 5.02 (d, *J* = 12.4 Hz, 1H), 5.08 (d, *J* = 12.4 Hz, 1H), 5.27-5.29 (m, 1H), 7.07-7.55 (m, 21H). ¹³C NMR (CDCl₃) δ = 14.5, 37.2, 37.5, 39.8, 43.8, 58.5, 67.0, 67.1, 116.6, 125.7, 127.0, 127.8, 127.9, 128.1, 128.2, 128.4, 128.5, 128.9, 130.3, 130.6, 130.9, 134.6, 135.4, 135.5, 139.0, 139.7, 141.8, 146.1, 171.4, 171.6, 196.5; HRMS (FAB+) for M + 1 found *m/z* 585.2625, calcd for C₃₉H₃₇O₅: 585.2642. [α]_D²² = -27.9 (*c* 1.31, CHCl₃, 96% ee). Ee was determined by HPLC analysis using a chiral column (Chiralpak IA: eluent: 20% 2-propanol in hexane, flow rate: 0.5 mL/min, retention time: 21 min for major isomer and 20 min for minor isomer).

phenyl(2-((*E*)-1,2-diphenylvinyl)phenyl)methanone (7). Colorless oil. IR (CDCl₃) 1664, 698 cm⁻¹; ¹H NMR (CDCl₃) δ = 6.62-7.61 (m, 20H). ¹³C NMR (CDCl₃) δ = 126.8, 127.2, 127.4, 127.8, 127.9, 128.0, 129.3, 129.6, 129.9, 130.4, 130.8, 132.4, 132.6, 137.1, 138.0, 139.6, 139.8, 141.2, 143.7, 198.8; HRMS (FAB⁺) for M + 1 found m/z 361.1584, calcd for C₂₇H₂₁O: 361.1592.

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 FREQU 27118.64 Hz
 SCANS 146
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 27.1 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

DFILE C:\NMR\3ab-H.als
 COMNT
 DATIM Fri Mar 23 13:53:24 2007
 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 26.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.00 Hz
 RGAIN 19

DFILE C:\NMR\3ab-C.als
 COMNT
 DATIM Fri Mar 23 14:00:52 2007
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 125
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 27.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

DFILE C:\NMR\E-3ac-H.als
 COMNT
 DATIM Mon Mar 19 18:26:14 2007
 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 26.3 c
 SLVNT C6D6
 EXREF 7.27 ppm
 BF 0.12 Hz
 RGAIN 18

DFILE C:\NMR\E-3ac-C.als
 COMNT 1127
 DATIM Mon Mar 19 11:42:27 2007
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 203
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 25.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 24

DFILE C:\NMR\Z-3ac-H.als
 COMNT 1127
 DATIM Mon Mar 19 11:22:23 2007
 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 27.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 22

DFILE C:\NMR\Z-3ac-C.als
 COMNT
 DATIM
 OBNUC 13C
 EXMOD
 OBFRQ 150.91 MHz
 OBSET 8.59 KHz
 OBFIN 3.80 Hz
 POINT 65536
 FREQU 34722.22 Hz
 SCANS 8
 ACQTM 0.0000 sec
 PD 0.0000 sec
 PW1 10.00 usec
 IRNUC
 CTEMP 0.0 c
 SLVNT
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 0

DFILE C:\NMR\3ba-H.als
 COMNT
 DATIM Wed Mar 14 21:24:44 2007
 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 26.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 17

DFILE C:\NMR\3ba-C.als
 COMNT
 DATIM Wed Mar 14 21:51:30 2007
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 508
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 25.2 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 24

DFILE C:\NMR\3ca-H.als
 COMNT
 DATIM Fri Apr 13 08:15:13 2007
 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 26.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.00 Hz
 RGAIN 19

DFILE C:\NMR\3ca-C.als
 COMNT Applied
 DATIM Thu Apr 05 20:47:35 2007
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27210.88 Hz
 SCANS 157
 ACQTM 1.2042 sec
 PD 1.7940 sec
 PW1 4.80 usec
 IRNUC 1H
 CTEMP 25.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 24

DFILE C:\NMR\3da-H.als
 COMNT
 DATIM Thu Apr 12 17:06:07 2007
 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 25.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 14

DFILE C:\NMR\3da-C.als
 COMNT
 DATIM Thu Apr 12 17:13:32 2007
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 101
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 26.0 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 24

DFILE C:\NMR\Z-3ea-H.als
 COMNT
 DATIM Tue Mar 13 18:14:14 2007
 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 14

(Z)-3ea

DFILE C:\NMR\Z-3ea-C.als
 COMNT
 DATIM Fri Mar 16 12:57:27 2007
 OBNUC ¹³C
 EXMOD bcm
 OBFRQ 125.40 MHz
 OBSET 140.00 KHz
 OBFIN 2343.75 Hz
 POINT 65536
 FREQU 50000.00 Hz
 SCANS 820
 ACQTM 1.3107 sec
 PD 1.6893 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 25.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 31

(Z)-3ea

DFILE C:\NMR\3ad-H.als
 COMNT
 DATIM Sat Oct 7 14:20:42 2006
 OBNUC 1H
 EXMOD non
 OBFRQ 500.00 MHz
 OBSET 160.00 KHz
 OBFIN 2369.59 Hz
 POINT 32768
 FREQU 5998.80 Hz
 SCANS 16
 ACQTM 5.4624 sec
 PD 1.5376 sec
 PW1 6.20 usec
 IRNUC 1H
 CTEMP 25.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 20.00 Hz
 RGAIN 21

DFILE C:\NMR\3ad-C.als
 COMNT
 DATIM Sat Oct 7 14:34:50 2006
 OBNUC ¹³C
 EXMOD bcm
 OBFRQ 125.65 MHz
 OBSET 120.00 KHz
 OBFIN 7259.72 Hz
 POINT 32768
 FREQU 26385.22 Hz
 SCANS 200
 ACQTM 1.2419 sec
 PD 1.7581 sec
 PW1 4.75 usec
 IRNUC ¹H
 CTEMP 27.0 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 20.00 Hz
 RGAIN 29

DFILE C:\NMR\3ae-H.als
 COMNT
 DATIM Tue Mar 20 15:11:44 2007
 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 25.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 19

DFILE C:\NMR\3ae-C.als
 COMNT 1127
 DATIM Fri Mar 23 16:21:51 2007
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 190
 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 0.12 Hz
 RGAIN 24

DFILE C:\NMR\5aa-H.als
 COMNT
 DATIM Fri Dec 01 10:29:12 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 27.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 21

DFILE C:\NMR\5aa-C.als
 COMNT
 DATIM Fri Dec 01 10:55:54 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 26.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 24

DFILE C:\NMR\5ba-H.als
 COMNT
 DATIM Mon Dec 18 16:15:57 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 23.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 20

DFILE C:\NMR\5ba-C.als
 COMNT
 DATIM Sat Dec 16 16:41:50 2006
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 134
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 33.2 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

DFILE C:\NMR\5ad-H.als
 COMNT
 DATIM Mon Mar 19 15:31:27 2007
 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 25.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 15

DFILE C:\NMR\5ad-C.als
 COMNT
 DATIM Mon Mar 19 15:38:28 2007
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 105
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 26.3 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

DFILE C:\NMR\5cd-H.als
 COMNT
 DATIM Wed Mar 21 18:14:37 2007
 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 26.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 18

DFILE C:\NMR\5cd-C.als
 COMNT
 DATIM Tue Apr 03 19:54:49 2007
 OBNUC ¹³C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 29962.55 Hz
 SCANS 256
 ACQTM 1.0936 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC ¹H
 CTEMP 26.0 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25

DFILE C:\NMR\7.als
 COMNT
 DATIM Fri Apr 27 09:55:45 2007
 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 26.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 19

DFILE C:\NMR\7-13C.als
 COMNT
 DATIM Fri Apr 27 10:07:17 2007
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 202
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 4.60 usec
 IRNUC 1H
 CTEMP 27.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 25