Supporting Information

Palladium- and Nickel-Catalyzed Intramolecular Cyanoboration of Alkynes

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Experimental procedures and characterization data for the new compounds

General. All reactions were performed under a nitrogen atmosphere with magnetic stirring. Column chromatography was performed with Wakogel C-200 (silica gel, 75-150 μ m, Wako). ^1H and ^{13}C NMR spectra were recorded on a Mercury vx400 spectrometer at ambient temperature. ^1H NMR data are reported as follows: chemical shift in ppm downfield from tetramethylsilane (δ scale), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sep = septet, m = multiplet, and br = broad), coupling constant (Hz), and integration. ^{13}C NMR data are reported in ppm downfield from tetramethylsilane (δ scale). High resolution mass spectra were recorded on a JEOL JMS-HX110A (FAB) or a JEOL JMS-SX102A (EI) spectrometer. Infrared spectra were recorded on a Mettler-Toledo ReactIR 4000 spectrometer.

Anhydrous THF (Kanto) and dioxane (Wako) were purchased from the commercial sources. Toluene was distilled from LiAlH_4 under nitrogen. $\text{Pd}(\text{acac})_2$ (Mitsuwa), PdCl_2 (Furuya Kinzoku, Co.), $\text{Ni}(\text{COD})_2$ (Aldrich), $\text{Rh}(\text{acac})(\text{COD})$ (Aldrich), *p*-iodonitrobenzene (TCI), *p*-iodotoluene (TCI), pinacol (TCI), acetic anhydride (Wako), tri(*t*-butyl)phosphine (Aldrich), KF (spray dried, Wako), 1,3-bis(diphenylphosphino)propane (dppp, Kanto), and 1,4-bis(diphenylphosphino)butane (dppb, Kanto) were used as received from the commercial sources. Methyl vinyl ketone (Wako) was obtained from the commercial source and purified by distillation.

$\text{Pd}(\text{PPh}_3)_4$,¹ $\text{Pd}_2(\text{dba})_3 \cdot \text{benzene}$,² $\text{PdCl}_2(\text{MeCN})_2$,³ bis(diisopropylamino)cyanoborane,⁴ and 4-methyl-1-heptyn-4-ol⁵ were prepared according to the literature methods.

Preparation of (diisopropylamino)cyanoboryl homopropargyl ethers **3**.

(a) Representative procedure for the preparation of **3 from tertiary homopropargyl alcohols (eq 2).** To a solution of bis(diisopropylamino)cyanoborane (474 mg, 2.0 mmol) in THF (1 mL) was added 4-methyl-1-heptyn-4-ol (265 mg, 2.1 mmol) at room temperature. The mixture was heated at 40 °C for 20 h. Evaporation of the solvent followed by bulb-to-bulb distillation (120–150 °C/1.0 mmHg) afforded **3a** (445 mg, 85%) as colorless oil. **3a**: ^1H NMR (C_6D_6) δ 0.82 (t, J = 7.2 Hz, 3H), 0.88 (d, J = 6.6 Hz, 6H), 1.13 (dd, J = 6.6, 2.4 Hz, 6H), 1.17–1.32 (m, 2H), 1.43 (s, 3H), 1.71–1.87 (m, 3H), 2.41 (dd, J = 16.8, 2.4 Hz, 1H), 2.50 (dd, J = 16.8, 2.4 Hz, 1H), 3.02 (sep, J = 6.6 Hz, 1H), 4.01 (sep, J = 6.6 Hz, 1H); ^{13}C NMR (C_6D_6) δ 14.7, 17.7, 22.3, 23.1, 26.2, 31.8, 42.9, 45.0, 49.8, 71.5, 78.7, 81.1; ^{11}B NMR (C_6D_6) δ 16.9; IR (neat) 2215 cm^{-1} . Anal. calcd. for $\text{C}_{15}\text{H}_{27}\text{BN}_2\text{O}$: C, 68.71; H, 10.38; N, 10.68. Found: C, 68.63; H, 10.19; N, 10.43.

(b) Representative procedure for the preparation of **3 from secondary homopropargyl alcohols.** To a solution of dichloro(diisopropylamino)borane (2.18 g, 12.0 mmol) in CH_2Cl_2 (20 mL) were added triethylamine (1.67 mL, 12.0 mmol) and 5-heptyn-3-ol (1.12 g, 10.0 mmol) at –78 °C. The reaction mixture was stirred for 4 h, while the reaction temperature was gradually raised to 0 °C. The mixture was further stirred at room temperature for 2 h. To this mixture was added trimethylsilyl cyanide (1.87 mL, 14 mmol) at room temperature, and the resultant mixture was stirred for 1 h at room temperature. After evaporation of the solvent was added hexane to the residue. The insoluble precipitates were filtered off and washed with hexane twice. Evaporation of the solvent followed by bulb-to-bulb distillation (100 °C/2 mmHg) yielded **3e** (2.32 g, 78%).

(1) Coulson, D. R. *Inorg. Synth.* **1972**, 13, 121.

(2) Ukai, T.; Kawazura, H.; Ishi, Y.; Bonnet, J. J.; Ibers, J. A. *J. Organomet. Chem.* **1974**, 65, 253.

(3) Doyle, J. R.; Slade, P. E.; Jonassen, H. B. *Inorg. Synth.* **1960**, 6, 218.

(4) Sugimoto, M.; Yamamoto, Y.; Ito, Y. *Chem. Comm.* **2002**, 1392.

(5) Nakagawa, T.; Kasatkin, A.; Sato, F. *Tetrahedron Lett.* **1995**, 36, 3207.

(c) Representative procedure for the preparation of 3 from primary homopropargyl alcohols (3 steps). *Step 1:* A mixture of boric acid (2.51 g, 40.7 mmol) and 3-hexyn-1-ol (20 mL, 183 mmol) in toluene (10 mL) was stirred under reflux for 20 h with removal of water by a Dean-Stark trap. Evaporation of the solvent followed by distillation (160 °C/1 mmHg) yielded tri(hex-3-ynyloxy)borane (12.1 g, 98% based on boric acid). *Step 2:* A mixture of tri(hex-3-ynyloxy)borane (5.9 g, 19.6 mmol), boron trichloride dimethylsulfide complex (1.79 g, 10.0 mmol), and chlorobis(diisopropylamino)borane (7.25 g, 29.4 mmol) was heated with stirring at 80 °C for 20 h. Distillation (76 °C/1 mmHg) yielded chloro(diisopropylamino)(hex-3-ynyloxy)borane (11.3g, 79%). *Step 3:* A mixture of chloro(diisopropylamino)(hex-3-ynyloxy)borane (11.3 g, 46 mmol) and trimethylsilyl cyanide (7.4 mL, 55.5 mmol) was heated with stirring at room temperature for 18 h. Distillation (107 °C/3 mmHg) yielded **3g** (10.2 g, 94%).

Intramolecular Cyanoboration

(a) General procedure for the optimization of the reaction conditions (Table 1). A mixture of a catalyst (0.015 mmol Pd or Ni), **3a** (79 mg, 0.30 mmol), and *m*-dimethoxybenzene (internal standard, 20.0 μ L, 21.1 mg) in toluene-*d*₈ (0.50 mL) was stirred at 50 °C. After 3 h, the reaction mixture was cooled to room temperature and was subjected to a ¹H NMR measurement. In cases where cyanoboration was taking place, the mixture was heated further at 50 °C. When no reaction was observed by the NMR measurement, the mixture was heated to 80 °C for 3 h, and the progress of the reaction was then checked by an NMR measurement. Finally, the temperature was raised to 110 °C (bath temp.) to observe the progress of the cyclization, if no reaction took place at 80 °C. Final reaction yield was determined from the integrations of ¹H NMR spectrum of the reaction mixture.

(b) Representative procedure for the cyanoboration of 3 in a preparative scale (Table 2). A mixture of Pd(PPh₃)₄ (578 mg, 0.50 mmol) and **3g** (2.34 g, 10.0 mmol) in toluene (17 mL) was heated at 120 °C for 43 h with stirring. Evaporation of the solvent followed by bulb-to-bulb distillation (120 °C/1.0 mmHg) afforded **4g** (2.20 g, 94%).

(c) Procedure for the one-pot reaction of 2a with bis(diisopropylamino)cyanoborane (eq 3). To a mixture of Pd₂(dba)₃•benzene (15

mg, 0.015 mmol) and bis(diisopropylamino)cyanoborane (171 mg, 0.72 mmol) in toluene (1.0 mL) was added the homopropargylic alcohol **2a** (76 mg, 0.60 mmol) dropwise over 1 min at 120 °C (bath temperature). The mixture was further stirred for 3 h at 120 °C. After the mixture was cooled to room temperature, the solvent was evaporated. To the residue, benzene-*d*₆ (ca. 1 mL) and 2,6-dimethylanisol (internal standard, 19.5 mg, 0.14 mmol) were added. The reaction yield (94%) was determined by a ¹H NMR measurement.

(*E*)-2-Diisopropylamino-5-methyl-5-propyl-3-cyanomethylene-1,2-oxaborolane (4a). ¹H NMR (C₆D₆) δ 0.77 (t, *J* = 6.8 Hz, 3H), 0.91 (s, 3H), 1.03-1.44 (m, 16H), 1.83 (d, *J* = 14.8 Hz, 1H), 2.02 (d, *J* = 14.8 Hz, 1H), 2.85-3.20 (br, 1H), 4.12-4.44 (br, 1H), 5.13 (s, 1H); ¹³C NMR (C₆D₆) δ 14.9, 18.1, 23.4, 27.0, 44.8 (br), 45.1, 50.4 (br), 51.6, 80.8, 102.4, 117.9, 167.0 (br); ¹¹B NMR (C₆D₆) δ 27.9; IR (neat) 2215 cm⁻¹. Anal. calcd. for C₁₅H₂₇BN₂O: C, 68.71; H, 10.38; N, 10.68. Found: C, 68.56; H, 10.45; N, 10.67.

(*E*)-2-Diisopropylamino-3-cyanomethylene-1-oxa-2-borasp[iro[4.5]decane (4b). ¹H NMR (C₆D₆) δ 1.00-1.50 (m, 20H), 1.54-1.70 (m, 2H), 1.94 (d, *J* = 2.1 Hz, 2H), 2.80-3.38 (br, 1H), 4.00-4.57 (br, 1H), 5.15 (s, 1H); ¹³C NMR (C₆D₆) δ 23.4, 26.0, 38.6, 45.0 (br), 50.5 (br), 52.1, 80.1, 102.5, 117.9, 166.1 (br); ¹¹B NMR (C₆D₆) δ 28.4; IR (neat) 2211 cm⁻¹. Anal. calcd. for C₁₆H₂₇BN₂O: C, 70.08; H, 9.92; N, 10.22. Found: C, 69.80; H, 9.66; N, 10.03.

(*E*)-2-Diisopropylamino-3-(1-cyanopropylidene)-1-oxa-2-borasp[iro[4.5]decane (4c).

^1H NMR (C_6D_6) δ 0.91 (t, $J = 7.5$ Hz, 3H), 0.99-1.16 (m, 3H), 1.22-1.52 (m, 17H), 1.57-1.73 (m, 2H), 1.90 (q, $J = 7.5$ Hz, 2H), 1.94 (s, 2H), 3.20-4.22 (br, 2H); ^{13}C NMR (C_6D_6) δ 12.9, 23.4, 26.2, 27.8, 38.8, 46.8, 47.4 (br), 79.9, 119.3, 120.0, 157.1 (br); ^{11}B NMR (C_6D_6) δ 28.6; IR (neat) 2207 cm^{-1} . Anal. calcd. for $\text{C}_{18}\text{H}_{31}\text{BN}_2\text{O}$: C, 71.52; H, 10.34; N, 9.27. Found: C, 71.40; H, 10.41; N, 9.12.

(*E*)-2-Diisopropylamino-3-cyanobenzylidene-5,5-dimethyl-1,2-oxaborolane (4d).

mp $87.0\text{--}87.9\text{ }^\circ\text{C}$: ^1H NMR (C_6D_6) δ 0.90 (s, 6H), 1.33 (d, $J = 6.6$ Hz, 12H), 2.27 (s, 2H), 2.95-4.42 (br, 2H), 6.92-7.05 (m, 3H), 7.20-7.26 (m, 2H); ^{13}C NMR (C_6D_6) δ 23.4, 29.3, 48.1 (br), 50.4, 79.0, 119.0, 119.9, 129.05, 129.10, 129.5, 137.1, 160.3 (br); ^{11}B NMR (C_6D_6) δ 29.3; IR (neat) 2207 cm^{-1} . Anal. calcd. for $\text{C}_{19}\text{H}_{27}\text{BN}_2\text{O}$: C, 73.56; H, 8.77; N, 9.03. Found: C, 73.72; H, 8.65; N, 8.95.

(*E*)-2-Diisopropylamino-3-(1-cyanoethylidene)-5-ethyl-1,2-oxaborolane (4e). ^1H NMR (C_6D_6) δ 0.80 (t, $J = 7.2$ Hz, 3H), 1.12-1.40 (m, 14H), 1.48 (s, 3H), 1.66 (dd, $J = 15.2, 6.8$ Hz, 1H), 2.08 (dd, $J = 15.2, 6.0$ Hz, 1H), 2.75-4.54 (br, 2H), 3.64 (quint, $J = 6.0$ Hz, 1H); ^{13}C NMR (C_6D_6) δ 10.2, 20.5, 23.4, 30.9, 41.0, 47.8 (br), 78.2, 111.8, 120.8, 157.4 (br); ^{11}B NMR (C_6D_6) δ 28.7; IR (neat) 2209 cm^{-1} . Anal. calcd. for $\text{C}_{14}\text{H}_{25}\text{BN}_2\text{O}$: C, 67.76; H, 10.15; N, 11.29. Found: C, 67.62; H, 10.19; N, 11.33.

(*E*)-2-Diisopropylamino-3-cyanomethylene-5-ethyl-1,2-oxaborolane (4f). ^1H NMR (C_6D_6) δ 0.75 (t, $J = 7.2$ Hz, 3H), 1.00-1.45 (m, 14H), 1.73 (dd, $J = 14.8, 6.8$ Hz, 1H), 1.94 (dd, $J = 14.8, 6.0$ Hz, 1H), 2.88-3.14 (br, 1H), 3.59 (quint, $J = 6.8$ Hz, 1H), 4.14-4.46 (br, 1H), 5.05 (s, 1H); ^{13}C NMR (C_6D_6) δ 10.1, 23.3, 30.5, 44.5 (br), 45.9, 50.5 (br),

78.4, 102.1, 117.9, 165.8 (br); ^{11}B NMR (C_6D_6) δ 28.1; IR (neat) 2216 cm^{-1} . Anal. calcd. for $\text{C}_{13}\text{H}_{23}\text{BN}_2\text{O}$: C, 66.68; H, 9.90; N, 11.96. Found: C, 66.82; H, 10.12; N, 12.03.

(E)-2-Diisopropylamino-3-(1-cyanopropylidene)-1,2-oxaborolane (4g). ^1H NMR (C_6D_6) δ 0.87 (t, $J = 7.5\text{ Hz}$, 3H), 1.25 (d, $J = 6.6\text{ Hz}$, 12H), 1.88 (q, $J = 7.5\text{ Hz}$, 2H), 1.98 (t, $J = 6.6\text{ Hz}$, 2H), 3.57 (t, $J = 6.6\text{ Hz}$, 2H), 3.44-3.89 (br, 2H); ^{13}C NMR (C_6D_6) δ 12.9, 23.4, 27.9, 35.2, 47.7 (br), 66.2, 119.2, 119.8, 156.4 (br); ^{11}B NMR (C_6D_6) δ 29.1; IR (neat) 2209 cm^{-1} . Anal. calcd. For $\text{C}_{13}\text{H}_{23}\text{BN}_2\text{O}$: C, 66.68; H, 9.90; N, 11.96. Found: C, 66.95; H, 9.78; N, 11.74.

(E)-2-(2-Diisopropylamino-[1,2]oxaborolan-3-ylidene)-4-methylpent-3-enenitrile (4h). ^1H NMR (C_6D_6) δ 1.29 (d, $J = 6.9\text{ Hz}$, 12H), 1.44 (d, $J = 1.5\text{ Hz}$, 3H), 1.51 (d, $J = 1.5\text{ Hz}$, 3H), 2.05 (dt, $J = 6.6, 0.6\text{ Hz}$, 2H), 3.49-3.95 (br, 2H), 3.59 (t, $J = 6.6\text{ Hz}$, 2H), 5.47-5.51 (m, 1H); ^{13}C NMR (C_6D_6) δ 19.9, 23.4, 26.2, 37.0, 47.8 (br), 66.3, 114.6, 119.5, 121.4, 140.9, 157.6 (br); ^{11}B NMR (C_6D_6) δ 29.1; IR (neat) 2211 cm^{-1} . Anal. calcd. for $\text{C}_{16}\text{H}_{27}\text{BN}_2\text{O}$: C, 69.24; H, 9.68; N, 10.77. Found: C, 69.12; H, 9.80; N, 10.73.

Synthesis of acetic acid (E)-4-cyano-3-(4,4,5,5-tetramethyl-[1,3,2]dioxaborolan-2-yl)hex-3-enyl ester (5). To a mixture of **4g** (70 mg, 0.30 mmol) and pinacol (43 mg, 0.36 mmol) in THF (1 mL) was added acetic anhydride (34 μL , 0.36 mmol) at room temperature. The mixture was heated with stirring at $40\text{ }^\circ\text{C}$ for 11 h. Evaporation followed by bulb-to-bulb distillation ($230\text{--}280\text{ }^\circ\text{C}/1\text{ mmHg}$) yielded **5** (78 mg, 89%).

5: ^1H NMR (CDCl_3) δ 1.16 (t, $J = 7.6$ Hz, 3H), 1.31 (s, 12H), 2.02 (s, 3H), 2.35 (q, $J = 7.6$ Hz, 2H), 2.61 (t, $J = 6.8$ Hz, 2H), 4.10 (t, $J = 6.8$ Hz, 2H); ^{13}C NMR (CDCl_3) δ 12.5, 20.8, 24.4, 24.6, 29.9, 62.6, 84.7, 118.6, 128.1, 171.0; ^{11}B NMR (C_6D_6) δ 29.1; IR (neat) 2215, 1740 cm^{-1} . HRFABMS calcd. for $\text{C}_{15}\text{H}_{25}\text{BNO}_4$ ($\text{M}+\text{H}^+$): 294.1877. Found: 294.1878. See below for the ^1H and ^{13}C NMR charts.

Synthesis of (Z)-2-ethyl-5-hydroxy-3-*p*-nitrophenylpent-2-enenitrile (6). To a mixture of $\text{Pd}_2(\text{dba})_3 \cdot \text{benzene}$ (6.0 mg, 0.006 mmol), tri-*t*-butylphosphine (3.6 μL , 0.0144 mmol), potassium fluoride (58 mg, 0.99 mmol), water (11 mg, 0.60 mmol) and *p*-iodonitrobenzene (90 mg, 0.36 mmol) in dioxane (1.0 mL) was added **4g** (70 mg, 0.30 mmol) at room temperature. The mixture was heated with stirring at 80 $^\circ\text{C}$ for 4 h. Water is added to the reaction mixture, and organic material was extracted with ethyl acetate three times. Evaporation followed by preparative TLC (hexane: ethyl acetate = 2:1) yielded **6** (71 mg, 96%). **6:** mp 77.3-77.6 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 1.24 (t, $J = 7.5$ Hz, 3H), 1.72 (br, 1H), 2.47 (q, $J = 7.5$ Hz, 2H), 2.81 (t, $J = 6.3$ Hz, 2H), 3.57 (t, $J = 6.3$ Hz, 2H), 7.51 (d, $J = 9.0$ Hz, 2H), 8.24 (d, $J = 9.0$ Hz, 2H); ^{13}C NMR (CDCl_3) δ 12.8, 24.1, 36.2, 59.6, 117.3, 118.2, 123.9, 129.1, 146.4, 147.8, 152.6; IR (neat) 3261, 2211, cm^{-1} . HRMS calcd. for $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}_3$ (M^+): 264.1004. Found: 264.1004. See below for the ^1H and ^{13}C NMR charts.

Synthesis of (Z)-2-ethyl-5-hydroxy-3-*p*-tolylpent-2-enenitrile (7). By a similar procedure to that of **6**, the title compound **7** was prepared from **4g** (70 mg, 0.30 mmol) and *p*-iodotoluene (79 mg, 0.36 mmol). The reaction was conducted for 10 h, and a

mixture of hexane/ethyl acetate (3:1) was used as eluent for preparative TLC. The product yield was 97% (63 mg). **7**: ^1H NMR (CDCl_3) δ 1.22 (t, $J = 7.6$ Hz, 3H), 1.75 (s, 1H), 2.36 (s, 3H), 2.43 (q, $J = 7.6$ Hz, 2H), 2.78 (t, $J = 6.8$ Hz, 2H), 3.53 (t, $J = 6.8$ Hz, 2H) 7.15-7.28 (m, 4H); ^{13}C NMR (CDCl_3) δ 13.0, 21.1, 24.1, 36.6, 60.0, 114.6, 119.3, 127.7, 129.3, 136.6, 138.9, 154.5; IR (neat) 3430, 2211, cm^{-1} . HRMS calcd. for $\text{C}_{14}\text{H}_{17}\text{NO}$ (M^+): 215.1310. Found: 215.1310. See below for the ^1H and ^{13}C NMR charts.

Synthesis of (E)-2-ethyl-5-hydroxypent-2-enitrile (8). To a mixture of $\text{Rh}(\text{acac})(\text{cod})$ (2.8 mg, 0.009 mmol) and 1,4-bis(diphenylphosphino)butane (3.8 mg, 0.009 mmol) in dioxane (1 mL) were added **4g** (70 mg, 0.03 mmol) and water (21.6 μL , 1.2 mmol) at room temperature. The mixture was stirred at 50 $^\circ\text{C}$ for 20 h. Water is added to the reaction mixture at room temperature and the organic material was extracted with ethyl acetate. Column chromatography on silica gel afforded **8** (36 mg, 96%). **8**: ^1H NMR (CDCl_3) δ 1.12 (t, $J = 7.5$ Hz, 3H), 2.10-2.29 (br, 1H), 2.24 (q, $J = 7.5$ Hz, 2H), 2.42 (q, $J = 6.6$ Hz, 2H), 3.67 (t, $J = 6.6$ Hz, 2H), 6.36, (t, $J = 6.9$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 12.5, 22.0, 31.5, 60.7, 118.3, 119.6, 143.8; IR (neat) 3411, 2217, cm^{-1} . HRCIMS calcd. for $\text{C}_7\text{H}_{12}\text{NO}$ ($\text{M}+\text{H}^+$): 126.0919. Found: 126.0919. See below for the ^1H and ^{13}C NMR charts.

Synthesis of (E)-2-ethyl-3-(2-hydroxyethyl)-6-oxohept-2-enitrile (9). To a mixture of $\text{Rh}(\text{acac})(\text{cod})$ (2.8 mg, 0.009 mmol), dppp (3.7 mg, 0.009 mmol), **4g** (70 mg, 0.30 mmol), methyl vinyl ketone (75 μL , 0.90 mmol), and methanol (27 μL , 0.66 mmol) in dioxane (1.0 mL) was heated at 50 $^\circ\text{C}$ for 22 h. To the mixture cooled to room temperature was added water, and organic material was extracted with AcOEt. Evaporation followed by column chromatography on silica gel (hexane:AcOEt = 2/1 to 1/2) afforded **9** (50 mg, 85%). ^1H NMR (CDCl_3) δ 1.11 (t, $J = 7.5$ Hz, 3H), 2.17 (s,

3H), 2.25 (q, $J = 7.5$ Hz, 2H), 2.43 (t, $J = 6.6$ Hz, 2H), 2.64 (s, 4H), 3.70 (t, $J = 6.6$ Hz, 2H); ^{13}C NMR (CDCl_3) δ 12.9, 23.1, 29.6, 29.7, 30.0, 34.7, 41.6, 60.3, 114.7, 118.5, 154.2; IR (neat) 3348, 2209, 1717, cm^{-1} . HRMS calcd. for $\text{C}_{11}\text{H}_{17}\text{NO}_2$ (M^+): 195.1259. Found: 195.1260. See below for the ^1H and ^{13}C NMR charts.

STANDARD 1H OBSERVE

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Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400HB "varian"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6006.0 Hz
16 repetitions
OBSERVE NL 400.4437670 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 31 sec

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75.4519960 MHz
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13C NMR

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3 600 0673572 MHz

15

1 minute

13C OBSERVE

Date: Dec 20 02
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB 'varian'
 PULSE SEQUENCE
 Relax delay 1.158 sec
 Pulse 50 0 degrees
 Acq time 0.842 sec
 Width 19000 0 Hz
 658 repetitions
 OBSERVE C13 75 451963 MHz
 DECOUPLE H1 300 0064341 MHz
 Power 35 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1 0 Hz
 FT size 32768
 Total time 22 minutes

STANDARD IN OBSERVE

Pulse Sequence: a2pul
 Solvent: CDCl3
 Ambient temperature
 Mercury-400BS "varian"
 Relax. delay 1.500 sec
 Pulse 45.0 degrees
 Acq. time 3.500 sec
 Width 6006.0 Hz
 Spectrometer
 OBSERVATION 400.4437682 MHz
 DATA PROCESSING
 F1 size 65536
 Total time 1 min, 31 sec

STANDARD IN OBSERVE

Date: Feb 8 03
 Solvent: D2O
 Ambient temperature
 SCANN=30000 101010
 PULSE SEQUENCE
 Acq time 3 200 sec
 Pulse delay 1 1002 sec
 Pulse 50 9 degrees
 Repetitions 18
 OBSERVE H1 300 007360 MHz
 DATA PROCESSING
 Total time 1 minute

STANDARD 1H OBSERVE

Date: Dec 20 02
 Solvent: CDCl3
 Name: 1-ethynyl-2-methyl-3-phenyl-4-oxo-5-hydroxy-6-norbornene
 GEMIN: 300889 "verian"
 PULSE SEQUENCE
 Relax delay: 1.502 sec
 Pulse: 90 degrees
 Width: 5000 Hz
 16 repetitions
 OBSERVE: 130.000573572 MHz
 QNP: 130.000573572 MHz
 FT size: 32768
 Total time: 1 minute

13C OBSERVE

Date: Dec 20 02
Solvent: CDCl3
Temperature: 25.000000
GEMIN-3000B "varian"
PULSE SEQUENCE
Relax delay: 1.158 sec
Pulse: 50.000000
Width: 19000.0 Hz
28798 repetitions
Observer: C. L. H. 12/20/02 15:55:50 MHz
Recorder: H. L. 300.0884841 MHz
Power: 35 dB
continuously on
continuously on
DATA PROCESSING
Line broadening: 1.0 Hz
F1 size: 32768
Total time: 16.6 hours