Supporting Information for:

Nickel-Catalyzed Addition of Pyridine-N-oxides Across Alkynes

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General. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique or in a dry box under an argon atmosphere. Flash column chromatography was performed using Kanto Chemical silica gel (spherical, 40–50 µm). Analytical thin layer chromatography (TLC) was performed on Merck Kieselgel 60 F254 (0.25 mm) plates. Visualization was accomplished with UV light (254 nm) and/or an aqueous alkaline KMnO4 solution followed by heating.

Apparatus. Proton and carbon nuclear magnetic resonance spectra (1H and 13C NMR) were recorded on a Varian Mercury 400 (1H NMR, 400 MHz; 13C NMR 101 MHz) spectrometer with solvent resonance as the internal standard (1H NMR, CHCl3 at 7.26 ppm; 13C NMR, CDCl3 at 77.0 ppm). 1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, br = broad, m = multiplet), coupling constants (Hz), and integration. Mass spectra were obtained with a JEOL JMS-700 (EI) or JEOL JMS-HMS-HX110A (FAB+) spectrometer. GC analysis was performed on a Shimadzu GC 2010 equipped with a DB-5 column (30 m x 0.53 mm, pressure = 31.7 kPa, detector = FID, 290 °C) with helium gas as a carrier.

Chemicals. Unless otherwise noted, commercially available reagents were used without purification. All alkynes were distilled before use. Anhydrous toluene purchased from Kanto Chemical was degassed vigorously with argon for 20 min and further purified by passage through activated alumina under positive argon pressure as described by Grubbs et al.1

Representative procedure for the synthesis of pyridine-N-oxides.2 Pyridine was dissolved in dry CH2Cl2 and then m-chloroperbenzoic acid (1.2 equiv) was added in one portion. The reaction mixture was stirred at room temperature overnight, concentrated in vacuo, and purified by flash chromatography on silica gel (CH2Cl2–acetone as an eluent).

Nickel-catalyzed hydroheteroarylation of alkynes using pyridine-N-oxides. A general Procedure. A pyridine-N-oxide (1.0 mmol) and an alkyne (1.5 mmol) were added sequentially to a solution of Ni(cod)2 (28 mg, 0.10 mmol) and PCyp3 (24 mg, 0.10 mmol) in toluene (2.5 mL) in a dry box. The vial was taken outside the dry box and heated at 35 °C for the time specified in Table 1. The resulting mixture was filtered through a pad of silica gel, concentrated in vacuo, and purified by flash chromatography on silica gel to give the corresponding hydroheteroarylation products in yields listed in Table 1.

(E)-2-(4-Octen-4-yl)pyridine-N-oxide [(E)-3aa]. A colorless oil, Rf 0.40 (CH2Cl2–acetone = 3:2); 1H NMR (400 MHz, CDCl3) δ 8.18 (dt, J = 6.2, 1.0 Hz, 1H), 7.24–7.06 (m, 3H), 5.62 (t, J = 7.2 Hz, 1H), 2.65 (t, J = 7.3 Hz, 2H), 2.20 (q, J = 7.4 Hz, 2H), 1.49 (sext, J = 7.4 Hz, 2H), 1.26 (sext, J = 7.5 Hz, 2H), 0.97 (t, J = 7.3 Hz, 3H), 0.86 (t, J = 7.3 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 152.8, 139.7, 136.3, 135.0, 127.2, 125.4, 123.9, 30.1, 29.5, 22.7, 22.0, 14.12, 14.07; Anal. Calcd for C13H19NO: C, 76.06; H, 9.33.
(Z)-2-(4-Octen-4-yl)pyridine-N-oxide [(Z)-3aa]. A colorless oil, R<sub>f</sub> 0.40 (CH<sub>2</sub>Cl<sub>2</sub>-acetone = 3:2), <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.27 (d, J = 5.9 Hz, 1H), 7.25–7.08 (m, 3H), 5.73 (t, J = 7.5 Hz, 1H), 2.70–2.30 (br, 2H), 1.87 (q, J = 7.3 Hz, 2H), 1.43–1.20 (m, 4H), 0.89 (t, J = 7.3 Hz, 3H), 0.86 (t, J = 7.3 Hz, 3H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.8, 140.1, 134.1, 132.5, 127.6, 124.9, 124.1, 36.5, 31.4, 22.8, 21.6, 14.0, 13.8.

2,6-Di(4-octen-4-yl)pyridine-N-oxide (3’aα, EE/others = 94:6). A colorless oil, R<sub>f</sub> 0.70 (CH<sub>2</sub>Cl<sub>2</sub>-acetone = 3:2), <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.12–7.05 (m, 3H), 5.70–5.64 (m, 0.08H), 5.61 (t, J = 7.2 Hz, 1.92H), 2.65 (t, J = 7.8 Hz, 4H), 2.21 (q, J = 7.4 Hz, 4H), 1.48 (sext, J = 7.2 Hz, 4H), 1.25 (sext, J = 7.5 Hz, 4H), 0.97 (m, 6H), 0.85 (m, 6H); <sup>13</sup>C NMR [for (E,E)-3’aα, 101 MHz, CDCl<sub>3</sub>] δ 152.8, 136.8, 134.1, 125.2, 124.4, 30.1, 29.5, 22.6, 21.8, 14.1, 14.0; HMRS (FAB+) Calcd for C<sub>21</sub>H<sub>35</sub>N=O: M<sup>+</sup>, 315.2562. Found: m/z 315.2569.

6-Methyl-2-(4-octen-4-yl)pyridine-N-oxide (3ba, E/Z = 93:7). A colorless oil, R<sub>f</sub> 0.45 (CH<sub>2</sub>Cl<sub>2</sub>-acetone = 3:2), <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.20–6.93 (m, 3H), 5.65 (t, J = 7.5, 0.07H), 5.57 (t, J = 7.2 Hz, 0.93H), 2.61 (t, J = 7.8 Hz, 2H), 2.51 (s, 0.21H), 2.49 (s, 2.79H), 2.19 (q, J = 7.4 Hz, 2H), 1.45 (sext, J = 7.4 Hz, 2H), 1.24 (sext, J = 7.5 Hz, 2H), 0.95 (t, J = 7.5 Hz, 3H), 0.84 (t, J = 7.4 Hz, 3H); <sup>13</sup>C NMR [for (E)-3ba, 101 MHz, CDCl<sub>3</sub>] δ 152.7, 148.8, 136.7, 133.9, 124.6, 124.5, 124.3, 29.9, 29.6, 22.5, 21.8, 18.1, 14.0, 13.9; HMRS (FAB+) Calcd for C<sub>14</sub>H<sub>21</sub>N=O: M<sup>+</sup>, 219.1623. Found: m/z 219.1613.

5,6-Dimethyl-2-(4-octen-4-yl)pyridine-N-oxide (3ca, E/Z = 97:3). A colorless oil, R<sub>f</sub> 0.40 (CH<sub>2</sub>Cl<sub>2</sub>-acetone = 3:2), <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.97 (d, J = 8.0 Hz, 1H), 6.93 (d, J = 8.4 Hz, 1H), 6.5 (t, J = 7.3 Hz, 0.03H), 5.54 (t, J = 7.3 Hz, 0.97H), 2.63 (t, J = 7.8 Hz, 2H), 2.52 (s, 0.09H), 2.5 (s, 2.91H), 2.34 (s, 0.09H), 2.32 (s, 2.91H), 2.20 (q, J = 7.3 Hz, 2H), 1.47 (sext, J = 7.4 Hz, 2H), 1.25 (sext, J = 7.5 Hz, 2H), 0.97 (t, J = 7.4 Hz, 3H), 0.86 (t, J = 7.4 Hz, 3H); <sup>13</sup>C NMR [for (E)-3ca, 101 MHz, CDCl<sub>3</sub>] δ 150.2, 147.8, 136.9, 133.4, 132.3, 126.2, 123.0, 29.9, 29.7, 19.2, 18.5, 14.0, 13.9; Anal. Calcd for C<sub>15</sub>H<sub>23</sub>N=O: C, 77.21; H, 9.93. Found: C, 76.96; H, 10.20.

4,6-Dimethyl-2-(4-octen-4-yl)pyridine-N-oxide (3da, E/Z = 94:6). A colorless oil, R<sub>f</sub> 0.30 (CH<sub>2</sub>Cl<sub>2</sub>-acetone = 3:2), <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.97 (d, J = 2.2 Hz, 1H), 6.85 (d, J = 2.8 Hz, 1H), 5.65 (t, J = 7.3, 0.06H), 5.57 (t, J = 7.2 Hz, 0.94H), 2.64 (t, J = 7.8 Hz, 2H), 2.51 (s, 0.18H), 2.49 (s, 2.82H), 2.31 (s, 0.18), 2.29 (s, 2.82H), 2.20 (q, J = 7.3 Hz, 2H), 1.48 (sext, J = 7.4 Hz, 2H), 1.27 (sext, J = 7.5 Hz, 2H), 0.98 (t, J = 7.4 Hz, 3H), 0.87 (t, J = 7.3 Hz, 3H); <sup>13</sup>C NMR [for (E)-3da, 101 MHz, CDCl<sub>3</sub>] δ 152.1, 148.1, 137.0, 135.6, 133.9, 125.4, 125.2, 30.1, 29.8, 22.8, 22.0, 20.3, 18.2, 14.20, 14.16; HMRS (FAB+) Calcd for C<sub>15</sub>H<sub>23</sub>N=O: M<sup>+</sup>, 233.1780. Found: m/z 233.1776.

(E)-3,6-Dimethyl-2-(4-octen-4-yl)pyridine-N-oxide (3ea). A colorless oil, R<sub>f</sub> 0.50 (CH<sub>2</sub>Cl<sub>2</sub>-acetone = 3:2), <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.01 (d, J = 8.1 Hz, 1H), 7.00 (d, J = 8.1 Hz, 1H), 5.38 (t, J = 7.3 Hz, 1H), 2.54–2.41 (m, 2H), 2.45 (s, 3H), 2.24 (s, 3H), 2.32–2.22 (m, 2H), 1.56–1.36 (m, 2H), 1.34–1.18 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H), 0.88 (t, J = 7.2 Hz, 3H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 151.9, 146.0, 133.8, 133.0, 132.3, 126.6,
123.2, 31.1, 30.0, 22.7, 22.1, 19.9, 18.0, 14.6, 14.2; HMRS (FAB+) Calcd for C_{15}H_{23}NO: M^+, 233.1780. Found: m/z 233.1786.

(E)-3-(Methoxycarbonyl)-6-methyl-2-(4-octen-4-yl)pyridine-N-oxide (3fa). A colorless oil, R_f 0.60 (CH$_2$Cl$_2$-acetone = 3:2), $^1$H NMR (400 MHz, CDCl$_3$) δ 7.36 (d, J = 8.0 Hz, 1H), 7.20 (d, J = 8.0 Hz, 1H), 5.40 (t, J = 7.2 Hz, 1H), 3.80 (s, 3H), 2.52 (s, 3H), 2.74–2.30 (m, 2H), 2.24–2.08 (m, 2H), 1.48–1.30 (m, 4H), 0.94 (t, J = 6.6 Hz, 3H), 0.91 (t, J = 7.1 Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 165.3, 152.3, 151.2, 134.4, 134.0, 129.6, 124.3, 123.7, 52.5, 31.2, 30.3, 22.5, 21.7, 18.6, 14.7, 14.1; Anal. Calcd for C$_{16}$H$_{22}$NO: C, 68.99; H, 8.29. Found: C, 69.29; H, 8.36.

(E)-1-(4-Octen-4-yl)-isoquinoline-N-oxide (3ga). A colorless oil, R_f 0.40 (CH$_2$Cl$_2$-acetone = 1:1), $^1$H NMR (400 MHz, CDCl$_3$) δ 8.13 (d, J = 7.1, 1H), 7.94 (d, J = 7.8 Hz, 1H), 7.30 (d, J = 7.4 Hz, 1H), 7.59–7.48 (m, 3H), 5.58 (t, J = 7.2 Hz, 1H), 2.80–2.57 (m, 2H), 2.48–2.28 (m, 2H), 1.64–1.48 (m, 2H), 1.38–1.16 (m, 2H), 1.03 (t, J = 7.3 Hz, 3H), 0.87 (t, J = 7.3 Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 148.9, 136.9, 135.9, 132.1, 129.5, 129.0, 128.5, 127.8, 126.6, 125.8, 122.3, 30.6, 30.3, 22.8, 21.9, 14.6, 14.2; HMRS (FAB+) Calcd for C$_{17}$H$_{21}$NO: M^+, 255.1623. Found: m/z 255.1618.

(E)-1,3-Di(4-octen-4-yl)isoquinoline-N-oxide (3’ga). A colorless oil, R_f 0.75 (CH$_2$Cl$_2$-acetone = 1:1), $^1$H NMR (400 MHz, CDCl$_3$) δ 7.93–7.87 (m, 1H), 7.74–7.67 (m, 1H), 7.53–7.44 (m, 3H), 5.69 (t, J = 7.2 Hz, 1H), 5.57 (t, J = 7.2 Hz, 1H), 2.84–2.54 (m, 4H), 2.50–2.16 (m, 4H), 1.68–1.42 (m, 4H), 1.38–1.16 (m, 4H), 1.10–0.94 (m, 6H), 0.92–0.80 (m, 6H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 150.2, 148.9, 136.9, 135.4, 134.2, 132.6, 129.2, 128.7, 127.5, 127.4, 126.3, 125.6, 122.3, 30.6, 30.4, 30.3, 22.84, 22.76, 21.83, 21.81, 14.6, 14.3, 14.2; HMRS (FAB+) Calcd for C$_{25}$H$_{35}$NO: M^+, 365.2719. Found: m/z 365.2709.

(E)-6-Methyl-2-(4-methyl-2-penten-2-yl)pyridine-N-oxide (3bb). A colorless oil, R_f 0.60 (CH$_2$Cl$_2$-acetone = 3:2), $^1$H NMR (400 MHz, CDCl$_3$) δ 7.17–7.04 (m, 3H), 5.50 (dq, J = 9.2, 1.3 Hz, 1H), 2.80–2.64 (m, 1H), 2.51 (s, 3H), 2.07 (d, J = 1.5 Hz, 3H), 1.05 (d, J = 6.8 Hz, 6H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 153.7, 149.0, 141.5, 129.6, 124.7, 124.4, 123.8, 27.4, 22.6, 18.2, 14.4; HMRS (FAB+) Calcd for C$_{19}$H$_{21}$NO: M^+, 191.1303. Found: m/z 191.1303.

(E)-2-(4,4-Dimethyl-2-penten-2-yl)-6-methylpyridine-N-oxide (3bc). A colorless oil, R_f 0.40 (CH$_2$Cl$_2$-acetone = 3:2), $^1$H NMR (400 MHz, CDCl$_3$) δ 7.15–7.00 (m, 3H), 5.57 (q, J = 1.4 Hz, 1H), 2.49 (s, 3H), 2.12 (d, J = 1.5 Hz, 3H), 1.20 (s, 9H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 154.8, 148.9, 143.7, 130.8, 124.7, 124.2, 123.7, 33.2, 30.5, 18.2, 15.7; Anal. Calcd for C$_{13}$H$_{19}$NO: C, 76.05; H, 9.21. Found: C, 76.06; H, 9.33.

**Deoxygenation of 3aa and 3ba. A general Procedure.** To a solution of 3 (1.10 mmol) in toluene (5.0 mL), PCl$_3$ (0.11 g, 1.30 mmol) was added dropwise, and the resulting mixture was stirred for 15 min at room temperature. Saturated NaHCO$_3$ was then added carefully, and the whole was stirred for further 5 min. After extraction with CH$_2$Cl$_2$/$\text{H}_2\text{O}$, the organic layers were washed with brine, concentrated in vacuo, and purified by flash silica gel chromatography to afford the corresponding deoxygenated 2-(4-octen-4-yl)pyridines 4.
(E)-2-(4-Octen-4-yl)pyridine (4a). A colorless oil, R, 0.60 (hexane–ethyl acetate = 9/1), \(^1H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.54 (ddd, \(J = 4.8, 1.8, 0.9\) Hz, 1H), 7.58 (ddd, \(J = 8.0, 6.8, 1.9\) Hz, 1H), 7.35 (ddd, \(J = 8.0, 2.0, 1.0\) Hz, 1H), 7.08 (ddd, \(J = 7.4, 4.9, 1.1\) Hz, 1H), 6.21 (t, \(J = 7.4\) Hz, 1H), 2.61 (t, \(J = 7.7\) Hz, 2H), 2.24 (q, \(J = 7.4\) Hz, 2H), 1.51 (sext, \(J = 7.4\) Hz, 2H), 1.42 (sext, \(J = 7.5\) Hz, 2H), 0.98 (t, \(J = 7.3\) Hz, 3H), 0.92 (t, \(J = 7.3\) Hz, 3H); \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta\) 159.8, 148.6, 139.5, 136.0, 132.1, 121.0, 120.2, 30.9, 30.2, 22.9, 22.2, 14.22, 14.18; HMRS (FAB+) Calcd for C\(_{13}\)H\(_{19}\)N: M\(^+\), 189.1517. Found: m/z 189.1525.

(E)-2-(4-Octen-4-yl)-6-methylpyridine (4b). A colorless oil, R, 0.40 (hexane–ethyl acetate = 9/1), \(^1H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.48 (t, \(J = 7.6\) Hz, 1H), 7.13 (d, \(J = 7.9\) Hz, 1H), 6.95 (d, \(J = 7.7\) Hz, 1H), 6.22 (t, \(J = 7.3\) Hz, 1H), 2.60 (t, \(J = 7.6\) Hz, 2H), 2.54 (s, 3H), 2.23 (q, \(J = 7.4\) Hz, 2H), 1.50 (sext, \(J = 7.4\) Hz, 2H), 1.41 (sext, \(J = 7.5\) Hz, 2H), 0.98 (t, \(J = 7.4\) Hz, 3H), 0.92 (t, \(J = 7.3\) Hz, 3H); \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta\) 159.3, 157.1, 139.7, 136.1, 131.6, 120.5, 117.2, 30.9, 30.5, 24.9, 23.0, 22.2, 14.3, 14.2; HMRS (FAB+) Calcd for C\(_{14}\)H\(_{21}\)N: M\(^+\), 203.1674. Found: m/z 203.1676.

Synthesis of (E)-2(4-octen-4-yl)-6-(propen-1-yl)pyridine (5).\(^{3}\) To a solution of 3aa (1.0 mmol) in THF (5 mL) were added allyl(trimethyl)silane (0.40 g, 2.5 mmol) and a 1.0 M solution of Bu\(_4\)NF in THF (0.10 mL, 0.10 mmol) at 0 °C, and the resulting mixture was stirred at 0–5 °C for 2 hours. The whole was diluted with CH\(_2\)Cl\(_2\), and the organic layers were washed with brine, dried over anhydrous Na\(_2\)SO\(_4\), and concentrated in vacuo. The residue was purified by flash chromatography on silica gel (hexane–ethyl acetate as an eluent) to give the title compound (0.12 g, 51%) as a colorless oil, R, 0.65 (hexane–ethyl acetate = 9/1). \(^1H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.51 (t, \(J = 7.7\) Hz, 1H), 7.18 (d, \(J = 7.9\) Hz, 1H), 7.01 (d, \(J = 7.7\) Hz, 1H), 6.79 (dq, \(J = 15.4, 6.8\) Hz, 1H), 15.03 (dq, \(J = 14.1, 1.6\) Hz, 1H), 6.27 (t, \(J = 7.4\) Hz, 1H), 2.63 (t, \(J = 7.7\) Hz, 2H), 2.25 (q, \(J = 7.5\) Hz, 2H), 1.94 (dd, \(J = 6.7, 1.8\) Hz, 3H), 1.58–1.38 (m, 4H), 0.99 (t, \(J = 7.4\) Hz, 3H), 0.94 (t, \(J = 7.3\) Hz, 3H); \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta\) 159.1, 154.6, 139.7, 136.3, 131.6, 131.5, 129.9, 118.2, 118.0, 30.9, 30.3, 23.0, 22.3, 18.4, 14.4, 14.2; HMRS (FAB+) Calcd for C\(_{16}\)H\(_{23}\)N: M\(^+\), 229.1830. Found: m/z 229.1836.

Synthesis of (E)-2-hydroxymethyl-6-(4-octen-4-yl)pyridine (6).\(^{4}\) Trifluoroacetic anhydride (0.24 mL, 1.63 mmol) was added dropwise to 3aa (70 mg, 0.33 mmol). The resulting orange mixture was stirred for 30 min at room temperature, and then reflushed for further 30 min. After cooling the mixture to room temperature, a saturated NaHCO\(_3\) aqueous solution was added slowly until the solution indicated pH 8, then stirred for 5 min. The mixture was extracted with CH\(_2\)Cl\(_2\), and the combined organic layers were washed with brine, dried over anhydrous Na\(_2\)SO\(_4\), and concentrated in vacuo. The residue was purified by flash chromatography on silica gel to afford 6 (58 mg, 81%) as a colorless oil, R, 0.35 (hexane–ethyl acetate = 1:1). \(^1H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.60 (t, \(J = 7.7\) Hz, 1H), 7.30 (d, \(J = 7.9, 1H\)), 7.00 (d, \(J = 7.7\) Hz, 1H), 6.29 (t, \(J = 7.4\) Hz, 1H), 4.73 (s, 2H), 4.60–4.40 (br, 1H), 2.62 (t, \(J = 7.2\) Hz, 2H), 2.25 (q, \(J = 7.0\) Hz, 2H), 1.58–1.38 (m, 4H), 0.99 (t, \(J = 7.3\) Hz, 3H), 0.94 (t, \(J = 7.4\) Hz, 3H); \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta\) 158.2, 156.8, 138.9, 136.8, 132.5, 118.5, 117.6, 63.5, 30.9, 30.2, 23.0, 22.3, 14.3, 14.2; HMRS (FAB+) Calcd for C\(_{14}\)H\(_{21}\)NO: M\(^+\), 219.1623. Found: m/z 219.1615.

References
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 7109-74-3-Pyr+2PrCCPr-clean-H
Mercury-400BB = m400

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
16 repetitions
OBSERVE H1, 399.9480258 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min, 4 sec

(EE/others = 96:4)
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 7109-74-3-PyrNO-2PrCCPr-C
Mercury-400SB "m400"

Relax, delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.195 sec
width 25125.6 Hz
69 repetitions
OBSERVE C13, 100.5670285 MHz
DECOUPLE H1, 399.9504906 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 39 min, 10 sec

(EE/others = 96:4)

[Chemical structure diagram]

[Graph with spectral data]
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 6805-74-3-2mepic-PrCCPr-H
Mercury-400 BR "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
16 repetitions
OBSERVE H1, 399.9480275 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min, 4 sec

3ba
(E/Z = 93:7)
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400SB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.159 sec
width 25125.6 Hz
113 repetitions
OBSERVE Cl3, 100.5670331 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 39 min, 10 sec

3ba
(E/Z = 93:7)
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
16 repetitions
OBSERVE H1, 399.9480254 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min, 4 sec

3da
($\alpha$/$\beta$ = 94:6)
Pulse Sequence: e2psl
Solvent: CDC13
Ambient temperature
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1024 repetitions

OBSERVE C13, 100.5670200 MHz
DECOUPLE H1, 399.9500405 MHz
Power 36 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 56 min, 12 sec

\[
3\text{da} \\ (E/Z = 94:6)
\]
Pulse Sequence: a2pul
Solvent: CDC13
Ambient temperature
Mercury-400BB "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.302 sec
Width 5995.2 Hz
16 repetitions
OBSERVE Hz, 399.9490262 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min, 4 sec

3.49
2.15 5.73
1.00
2.19 3.11
2.21 3.25

(E/Z > 99:1)
Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
Mercury-400MB "H400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
111 repetitions
OBSERVE C13, 100.5670246 MHz
DECOUPLE H1, 359.9500406 MHz
Power 36 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 56 min, 12 sec

\[\text{Mn} \quad \text{N} \quad \text{H} \quad \text{Pr} \]

\[\text{Bea} \quad (E/Z > 99:1)\]
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5998.2 Hz
16 repetitions
OBSPRUE H1, 399.9480256 MHz

DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 36 sec

3ga
(E/Z > 99:1)
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "n400"

Relax. delay 0.801 sec
Pulse 45.0 degrees.
Acq. time 1.199 sec
Width 25125.6 Hz
156 repetitions
OBSERVE c13, 100.5670239 MHz
DECouple H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 56 min, 12 sec

\(\text{3ga} \quad (E/Z > 99:1)\)
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
16 repetitions

OBSERVE  H1, 399.8480255 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 23 sec

\[ \text{3'ga} \quad (\varepsilon/\tau > 99:1) \]
Pulse Sequence: a2pul

$3'ga$

$(E/Z > 99:1)$
Pulse Sequence: a2pul
Solvent: CDCl3
Ambient temperature
Mercury-400SB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
465 repetitions

OBSEERVE C13, 100.5670216 MHz
DECouple H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 56 min, 12 sec

\( \text{Me} \)\(^{13}\text{C}\) \( \text{Me} \)

\[ 3 \text{bb} \text{ (Z/E > 99:1)} \]
Pulse Sequence: a2pul
Solvent: CDCl3
Ambient temperature
File: 6z20-74-3-Pyr-PrCCPh-Colean
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
165 repetitions
OBSERVE C13, 100.5676233 MHz
DECOUPLE H1, 399.9506046 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 56 min, 12 sec


ppm

4aa
Pulse Sequence: s2pul

Solvent: CDC13
Ambient temperature
File: 7119-74-3-deoxygenation-2-picyrN=PrCCPr-H
Mercury-400BB ~m400

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.302 sec
Width 5995.2 Hz
16 repetitions
OBSERVE E1, 399.9480256 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 2 min, 4 sec

4-ha

0.99 1.00 2.24 2.17 2.19 3.29
1.02 1.00 3.09 2.27 3.32
Pulse Sequence: a2pul
Solvent: CDC13
Ambient temperature
File: 7119-74-3-deoxyg-2picHO-PrCCPr-C
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
192 repetitions
OBSERVE C13, 100.3070208 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 35 min, 40 sec
7126-74-3-2picNO-PrCCPr-Me3Si allyl-H

Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
File: 7126-74-3-2picNO-PrCCPr-Me3Si allyl-K
Mercury-400BB "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
15 repetitions
OBSERVE NL, 399.9480258 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min, 4 sec

\[ \text{Diagram Image} \]

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<th>ppm</th>
<th>9</th>
<th>8</th>
<th>7</th>
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<th>5</th>
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<th>3</th>
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<td>2.23</td>
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</table>
Pulse Sequence: s2pul
Solvent: CDCl$_3$
Ambient temperature
File: 7126-74-3-PyrN=PrCCPr+Me3Siallyl-C
Mercury-405BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.195 sec
Width 25125.6 Hz
219 repetitions

OBSERVE C13, 100.5670223 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 56 min, 12 sec

156.096 154.012
139.714 136.293 133.624 131.134 129.311 118.196 117.960
77.320 77.300 76.897
30.302 30.329 32.394 32.331 31.841 14.393 34.233

180 160 140 120 100 80 60 40 20 ppm
Pulse Sequence: s2pol
Solvent: CDC13
Ambient temperature
Mercury-400BB "m400"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
10 repetitions

OBSEERVE H1, 399.9480254 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min, 4 sec