Supporting Information

Synthesis of Triazoles from Nonactivated Terminal Alkynes via the Three Component Coupling Reaction using a Pd(0)-Cu(I) Bimetallic Catalyst

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Experimental Procedure

Representative procedure for the synthesis of 2-allyl-1,2,3-triazole under the Pd(0)-Cu(I) bimetallic catalyst. To an AcOEt solution (1 mL) of phenylacetylene **1a** (55 μ L, 0.5 mmol), Pd₂(dba)₃·CHCl₃ (13.0 mg, 0.0125 mmol), P(OPh)₃ (26 μ L, 0.1 mmol) and CuCl(PPh₃)₃ (44.3 mg, 0.05 mmol) were added allyl methyl carbonate **2a** (0.07 mL, 0.6 mmol) and TMSN₃ (0.08 mL, 0.6 mmol) under an Ar atmosphere. The solution was stirred at 100 °C for 10 h. The reaction mixture was cooled to room temperature and filtered through a short Florisil pad and concentrated. The residue was purified by column chromatography (silica gel, hexane/AcOEt 50:1 to 20:1) to afford 2-allyl-4-phenyl-1,2,3-triazole **2a** in 83% yield (77.0 mg).

Procedure for the ruthenium-catalyzed isomerization of 2-allyl-4-phenyl-1,2,3-triazole (2a**) to 4-phenyl-2-(1-propenyl)-1,2,3-triazole (**2a'**).** A toluene solution (1.2 mL, 0.5 M) of 2-allyl-4-phenyl-1,2,3-triazole **2a** (114.8 mg, 0.62 mmol) and a catalytic amount of HRuCl(CO)(PPh₃)₃ (29.5 mg, 0.031 mmol) was heated at 120 °C for 3 h under Ar atmosphere. Then, the solution was cooled to room temperature and the reaction mixture was filtered through a short Florisil column. The eluent was concentrated and the residue was purified by column chromatography (silica gel, hexane/AcOEt 100:1) to afford 4-phenyl-2-(1-propenyl)-1,2,3-triazole **2a'** in 94% yield (107.4 mg). The ratio of *trans* and *cis* isomers (84/16) was determined by the ¹HNMR spectrum.

Procedure for the ozonolysis of 4-phenyl-2-(1-propenyl)-1,2,3-triazole (2a'**) leading to 4-phenyl-2H-1,2,3-triazole (**3a**).** 4-Phenyl-2-(1-propenyl)-1,2,3-triazole **2a'** (75.9 mg, 0.41 mmol) was dissolved in a combined solution of CH₂Cl₂ (3 mL) and MeOH (2 mL). The reaction mixture was cooled to -78 °C and O₃ was passed through until the color of the solution turned slight blue. Then, Me₂S (30 μ L, 0.41 mmol) was added to the solution and the mixture was gradually warmed up to room temperature. After

concentration of the mixture, the residue was purified by column chromatography (silica gel, hexane/AcOEt 10:1 to 2:1) to give 4-phenyl-2*H*-1,2,3-triazole **3a** in 88% yield (52.1 mg).

Analytical data

2-Allyl-4-phenyl-1,2,3-triazole (2a): yellow oil; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 5.07 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.31 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.33 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.12 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.46-7.31 (3H, m), 7.81-7.76 (2H, m), 7.86 (1H, s); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 57.34, 119.19, 125.85, 128.36, 128.81, 130.31, 131.12, 131.61, 147.88; IR (neat) 1645, 1473, 1458, 979, 918, 769, 692 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{11}\text{H}_{11}\text{N}_3$ (M^+) 185.0953. Found 185.0955.

2-Allyl-4-(*p*-chlorophenyl)-1,2,3-triazole (2b): colorless oil; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 5.07 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.31 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.34 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.11 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.42-7.36 (2H, m), 7.75-7.69 (2H, m), 7.82 (1H, s); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 57.41, 119.36, 127.08, 128.81, 129.00, 131.05, 131.45, 134.13, 146.82; IR (neat) 1645, 1475, 1421, 1097, 981, 916, 831 cm^{-1} ; Anal. Calcd for $\text{C}_{11}\text{H}_{10}\text{ClN}_3$: C, 60.15; H, 4.59; N, 19.13. Found: C, 60.29; H, 4.86; N, 18.93; HRMS (EI) Calcd for $\text{C}_{11}\text{H}_{10}\text{ClN}_3$ (M^+) 219.0563. Found 219.0560.

2-Allyl-4-(*p*-(methoxycarbonyl)phenyl)-1,2,3-triazole (2c): white solid; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 3.94 (3H, s), 5.09 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.33 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.35 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.13 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.88-7.84 (2H, m), 7.92 (1H, s), 8.12-8.07 (2H, m); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 52.08, 57.48, 119.44, 125.61, 129.73, 130.12, 131.34, 131.66, 134.57, 146.79, 166.62; IR (KBr) 1718, 1614, 1431, 1273, 1111, 981, 921, 856 cm^{-1} ; Anal. Calcd for $\text{C}_{13}\text{H}_{13}\text{N}_3\text{O}_2$: C, 64.19; H, 5.39; N, 17.27. Found: C, 64.33; H, 5.66; N, 17.11; HRMS (EI) Calcd for $\text{C}_{13}\text{H}_{13}\text{N}_3\text{O}_2$ (M^+) 243.1008. Found 243.1008.

2-Allyl-4-(*p*-methoxyphenyl)-1,2,3-triazole (2d): white solid; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 3.84 (3H, s), 5.06 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.30 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.32 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.12 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 6.98-6.92 (2H, m), 7.74-7.68 (2H, m); 7.77 (1H, s); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 55.22, 57.22, 114.18, 119.06, 122.98, 127.13, 130.55, 131.70, 147.70, 159.73; IR (KBr) 1614, 1488, 1248, 1038, 993, 914, 839 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{12}\text{H}_{13}\text{N}_3\text{O}$ (M^+) 215.1059. Found 215.1058

2-Allyl-4-(*p*-(dimethylamino)phenyl)-1,2,3-triazole (2e): white solid; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 2.99 (6H, s), 5.04 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.29 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.31 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.11 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 6.79-6.73 (2H, m), 7.68-7.62 (2H, m); 7.75 (1H, s); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 40.34, 57.10, 112.34, 118.39, 118.86, 126.75, 130.16, 131.85, 148.29, 150.41; IR (KBr) 1618, 1494, 1363, 1199, 977, 815 cm^{-1} ; Anal. Calcd for $\text{C}_{13}\text{H}_{16}\text{N}_4$: C, 68.39; H, 7.06; N, 24.54. Found: C, 68.30; H, 7.16; N, 24.35; HRMS (EI) Calcd for $\text{C}_{13}\text{H}_{16}\text{N}_4$ (M^+) 228.1375. Found 228.1380.

2-Allyl-4-(1-naphthyl)-1,2,3-triazole (2f): colorless oil; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 5.17 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.36 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.36 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.20 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.56-7.49 (3H, m), 7.71-7.66 (1H, m), 7.90 (1H, s), 7.92-7.86 (2H, m), 8.41-8.35 (1H, m); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 57.39, 119.32, 125.20, 125.33, 125.95, 126.64, 127.15, 127.80, 128.38, 129.01, 130.96, 131.59, 133.81, 134.05, 147.17; IR (neat) 1647, 1593, 1379, 1361, 1018, 997, 802, 777 cm^{-1} ; Anal. Calcd for $\text{C}_{15}\text{H}_{13}\text{N}_3$: C, 76.571; H, 5.568; N, 17.858. Found: C, 76.521; H, 5.788; N, 17.858; HRMS (EI) Calcd for $\text{C}_{15}\text{H}_{13}\text{N}_3$ (M^+) 235.1109. Found 235.1111.

2-Allyl-4-hexyl-1,2,3-triazole (2g): colorless oil; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 0.88 (3H, bt), 1.40-1.25 (6H, m), 1.70-1.59 (2H, m), 2.66 (2H, t, $J = 7.0$), 4.97 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.24 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.28 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.06 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.37 (1H, s); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 14.00, 22.49, 25.44, 28.86, 29.26, 31.49, 56.98, 118.91, 131.85, 132.68, 148.96; IR (neat) 1647, 1456, 1379, 993, 918, 792 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{11}\text{H}_{19}\text{N}_3$ (M^+) 193.1579. Found 193.1579.

2-Allyl-4-(*t*-butyl)-1,2,3-triazole (2h): colorless oil; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 1.32 (9H, s), 4.98 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.22 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.27 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.07 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.40 (1H, s); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 30.34, 30.73, 56.97, 118.66, 130.53, 131.98, 157.77; IR (neat) 1647, 1461, 1363, 1309, 997, 918, 787 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_9\text{H}_{15}\text{N}_3$ (M^+) 165.1266. Found 165.1268.

2-Allyl-4-(*N*-methyl-*N*-phenylsulfonylamino)methyl-1,2,3-triazole (2i): colorless oil; $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 2.71 (3H, s), 4.32 (2H, s), 4.95 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.23 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.29 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.01 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.50 (1H, s), 7.65-7.49 (3H, m), 7.84-7.79 (2H, m); $^{13}\text{C-NMR}$ (75.4 MHz, CDCl_3) δ 34.60, 45.01, 57.13, 119.25, 127.29, 129.02, 131.23, 132.70, 133.69, 137.24, 143.21; IR (neat) 1647, 1446, 1342, 1166, 985, 923, 794, 736,

692 cm^{-1} ; Anal. Calcd for $\text{C}_{13}\text{H}_{16}\text{N}_4\text{SO}_2$: C, 53.407; H, 5.515; N, 19.163, S, 10.967. Found: C, 53.447, H, 5.547; N, 19.045, S, 10.98.

2-Allyl-4-(benzyloxy)lmethyl-1,2,3-triazole (2j): colorless oil; ^1H -NMR (300 MHz, CDCl_3) δ 4.58 (2H, s), 4.63 (2H, s), 5.02 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.28 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.31 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.07 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.37-7.28 (5H, m), 7.61 (1H, s); ^{13}C -NMR (75.4 MHz, CDCl_3) δ 57.22, 63.16, 72.46, 119.30, 127.74, 127.86, 128.38, 131.47, 133.71, 137.64, 145.39; IR (neat) 1647, 1496, 1454, 1348, 1098, 1045, 995, 794, 738, 698 cm^{-1} ; Anal. Calcd for $\text{C}_{13}\text{H}_{15}\text{N}_3$: C, 68.101; H, 6.593; N, 18.326. Found: C, 68.216; H, 6.727; N, 18.193.

2-Allyl-4-(1-cyclohexenyl)-1,2,3-triazole (2k): colorless oil; ^1H -NMR (300 MHz, CDCl_3) δ 1.80-1.62 (4H, m), 2.28-2.15 (2H, m), 2.45-2.37 (2H, m), 4.98 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.25 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.28 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.06 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 6.38-6.34 (1H, m), 7.56 (1H, s); ^{13}C -NMR (75.4 MHz, CDCl_3) δ 22.07, 22.34, 25.27, 26.05, 57.07, 118.89, 125.92, 127.67, 130.16, 131.79, 149.65; IR (neat) 1645, 1506, 1435, 993, 922, 833, 786 cm^{-1} ; Anal. Calcd for $\text{C}_{11}\text{H}_{15}\text{N}_3$: C, 69.809; H, 7.988; N, 22.202. Found: C, 70.004; H, 8.132; N, 22.038; HRMS (EI) Calcd for $\text{C}_{11}\text{H}_{15}\text{N}_3$ (M^+) 189.1266. Found 189.1262.

2-Allyl-4-isopropenyl-1,2,3-triazole (2l): colorless oil; ^1H -NMR (300 MHz, CDCl_3) δ 2.14 (3H, dd, $J = 1.5, 1.0$ Hz), 5.01 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.14 (1H, dq, $J = 1.5, 1.5$ Hz), 5.27 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.30 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 5.57 (1H, dq, $J = 1.5, 1.0$ Hz), 6.08 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.64 (1H, s); ^{13}C -NMR (75.4 MHz, CDCl_3) δ 20.41, 57.20, 113.23, 119.08, 131.17, 131.67, 133.98, 148.97; IR (neat) 1645, 1508, 1453, 997, 925, 894 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_8\text{H}_{11}\text{N}_3$ (M^+) 149.0953. Found 149.0957.

2-Allyl-4-(1-octynyl)-1,2,3-triazole (2m): yellow oil; ^1H -NMR (300 MHz, CDCl_3) δ 0.89 (3H, bt), 1.49-1.24 (6H, m), 1.65-1.55 (2H, m), 2.41 (2H, t, $J = 7.0$ Hz), 4.99 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.28 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.32 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz), 6.05 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.59 (1H, s); ^{13}C -NMR (75.4 MHz, CDCl_3) δ 13.96, 19.27, 22.43, 28.22, 28.51, 31.25, 57.45, 69.60, 94.40, 119.56, 131.09, 131.75, 136.60; IR (neat) 2248, 1647, 1504, 1467, 995, 933, 786 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{13}\text{H}_{19}\text{N}_3$ (M^+) 217.1579. Found 217.1571.

2-Allyl-4-(*N*-tosyl-3-piperidyl)-1,2,3-triazole (2n): yellow oil; ^1H -NMR (300 MHz, CDCl_3) δ 1.39-1.53 (1H, m), 1.68-1.85 (2H, m), 1.96-2.05 (1H, m), 2.36-2.49 (2H, m), 2.44 (3H, s), 3.02-3.12 (1H, m), 3.64-3.72 (1H, m), 3.83-3.90 (1H, m), 4.97 (2H, ddd, $J = 6.0, 1.0, 1.0$ Hz), 5.24 (1H, ddt, $J = 17.0, 1.0, 1.0$ Hz), 5.29 (1H, ddt, $J = 10.0, 1.0, 1.0$ Hz).

Hz), 6.04 (1H, ddt, $J = 17.0, 10.0, 6.0$ Hz), 7.29-7.35 (2H, m), 7.43 (1H, s), 7.63-7.67 (2H, m); ^{13}C -NMR (75.4 MHz, CDCl_3) δ 21.42, 24.23, 29.51, 33.34, 46.30, 50.71, 57.10, 119.08, 127.55, 129.58, 131.51, 131.89, 133.08, 143.42, 149.04; IR (neat) 1647, 1596, 1465, 1340, 1168, 1005, 945, 815 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{17}\text{H}_{22}\text{N}_4\text{SO}_2$ ($[\text{M}+\text{H}]^+$) 347.1536. Found 347.1531.

4-Phenyl-2-(*trans*-1-propenyl)-1,2,3-triazole (2a'): white solid; ^1H NMR (300 MHz, CDCl_3) δ 1.91 (3H, dd, $J = 7.5, 2.0$ Hz), 6.65 (1H, dq $J = 14.0, 7.5$ Hz), 7.13 (1H, dq, $J = 14.0, 2.0$ Hz), 7.32-7.48 (3H, m), 7.83 (2H, m), 7.90 (1H, s); ^{13}C NMR (75.4 MHz, CDCl_3) δ 13.16, 116.71, 125.93, 128.03, 128.58, 128.83, 131.55, 133.54, 147.95; IR (neat) 1678, 1475, 1396, 977, 767, 692 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{11}\text{H}_{11}\text{N}_3$ (M^+) 185.0953. Found 185.0953.

4-Phenyl-2*H*-1,2,3-triazole (3a): white solid; ^1H NMR (300 MHz, CDCl_3) δ 7.38 (1H, br t, $J = 7.0$ Hz), 7.46 (2H, br t, $J = 7.0$ Hz), 7.83 (2H, br d, $J = 7.0$ Hz), 7.98 (1H, s); ^{13}C NMR (75.4 MHz, CD_3OD) δ 126.93, 127.44, 129.51, 129.98, 131.18, 146.96; IR (KBr) 3150-2500 (br), 1467, 1454, 1083, 765, 694 cm^{-1} ; Anal. Calcd for $\text{C}_8\text{H}_7\text{N}_3$: C, 66.19; H, 4.86; N, 28.95. Found: C, 66.28; H, 4.94; N, 28.74; HRMS (EI) Calcd for $\text{C}_8\text{H}_7\text{N}_3$ (M^+) 145.0640. Found: 145.0639.

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 RGAIN 18

Crystallographic data of 2e

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{13}H_{18}N_4$
Formula Weight	228.30
Crystal Color, Habit	Colorless, Plate
Crystal Dimensions	0.20 X 0.20 X 0.08 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	4908 (6.7 - 55.0°)
Lattice Parameters	a = 6.057(4) Å b = 8.667(6) Å c = 23.73(2) Å V = 1246(1) Å ³
Space Group	P2 ₁ 2 ₁ 2 ₁ (#19)
Z value	4
D _{calc}	1.217 g/cm ³
F ₀₀₀	488.00
$\mu(\text{MoK}\alpha)$	0.76 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku/MSC Mercury CCD
Radiation	MoK α (λ = 0.71070 Å) graphite monochromated
Temperature	-100.0 °C
Voltage, Current	50 kV, 40 mA
Collimator Size	0.5 mm

Detector Aperture	70 mm x 70 mm
Data Images	900 exposures
ω oscillation Range ($\chi=45.0$, $\phi=0.0$)	-77.0 - 103.0°
Exposure Rate	50.0 sec./°
Detector Swing Angle	13.10°
Detector Position	39.96 mm
ω oscillation Range ($\chi=45.0$, $\phi=90.0$)	-77.0 - 103.0°
Exposure Rate	50.0 sec./°
Detector Swing Angle	13.10°
Detector Position	39.96 mm
$2\theta_{max}$	54.9°
No. of Reflections Measured	Total: 11663 Unique: 1666 ($R_{int} = 0.027$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.8937 - 0.9939)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(Fo - Fc)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(F_o)} = [\sigma_o^2(F_o) + \frac{e^2}{4} F_o^2]^{-1}$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations ($I > 2.00\sigma(I)$, $2\theta < 54.93^\circ$)	2427
No. Variables	155
Reflection/Parameter Ratio	15.66
Residuals: R; Rw	0.044 ; 0.054

Residuals: R1	0.044
No. of Reflections to calc R1	2427
Goodness of Fit Indicator	1.38
Max Shift/Error in Final Cycle	0.015
Maximum peak in Final Diff. Map	$0.14 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.14 \text{ e}^-/\text{\AA}^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
N(1)	0.3708(3)	0.4043(2)	0.31235(7)	2.57(3)
N(2)	0.5140(3)	0.3007(2)	0.29398(7)	2.29(3)
N(3)	0.5681(3)	0.1888(2)	0.33033(7)	2.26(3)
N(4)	0.4832(3)	-0.1332(2)	0.57749(7)	2.44(3)
C(1)	0.3237(4)	0.3554(2)	0.36463(9)	2.49(4)
C(2)	0.4465(3)	0.2216(2)	0.37629(8)	1.97(4)
C(3)	0.4542(3)	0.1280(2)	0.42765(8)	1.97(3)
C(4)	0.2973(3)	0.1465(2)	0.47008(9)	2.19(4)
C(5)	0.3054(3)	0.0606(2)	0.51907(8)	2.22(4)
C(6)	0.4726(3)	-0.0496(2)	0.52824(8)	1.98(3)
C(7)	0.6309(3)	-0.0683(2)	0.48532(8)	2.29(4)
C(8)	0.6207(3)	0.0193(2)	0.43637(8)	2.33(4)
C(9)	0.3186(4)	-0.1142(3)	0.62103(9)	3.06(5)
C(10)	0.6516(4)	-0.2489(3)	0.58515(9)	2.88(5)
C(11)	0.6216(4)	0.3138(3)	0.23863(9)	2.56(4)
C(12)	0.8532(3)	0.3731(2)	0.24433(8)	2.39(4)
C(13)	0.9342(4)	0.4876(3)	0.21547(10)	3.10(5)
H(1)	0.1699	0.2273	0.4683	3.1
H(2)	0.1761	0.0792	0.5448	3.1
H(3)	0.7372	-0.1672	0.4944	3.1
H(4)	0.7282	0.0065	0.4040	3.1
H(5)	0.2104	0.4066	0.3897	3.1
H(6)	0.4985	0.3859	0.2172	3.1
H(7)	0.6277	0.2089	0.2240	3.1
H(8)	0.9271	0.3210	0.2737	3.1
H(9)	0.8469	0.5350	0.1862	3.9
H(10)	1.0769	0.5281	0.2230	3.9
H(11)	0.1744	-0.1229	0.6045	3.9
H(12)	0.3350	-0.1913	0.6489	3.9
H(13)	0.3295	-0.0193	0.6374	2.6
H(14)	0.6277	-0.2999	0.6197	3.7
H(15)	0.6692	-0.3277	0.5555	2.6
H(16)	0.7921	-0.1952	0.5874	3.7

$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(1)	0.0319(9)	0.0314(9)	0.0345(8)	0.0058(7)	-0.0018(8)	0.0005(7)
N(2)	0.0286(9)	0.0305(9)	0.0281(8)	0.0008(7)	-0.0005(7)	0.0011(7)
N(3)	0.0296(9)	0.0273(8)	0.0288(8)	0.0020(7)	0.0011(7)	0.0006(7)
N(4)	0.0330(9)	0.0327(9)	0.0269(8)	0.0050(8)	0.0021(7)	-0.0001(7)
C(1)	0.030(1)	0.031(1)	0.0341(9)	0.0061(9)	-0.0004(9)	-0.0025(8)
C(2)	0.0212(9)	0.0252(9)	0.0285(9)	-0.0010(7)	-0.0014(7)	-0.0047(7)
C(3)	0.0248(9)	0.0230(9)	0.0271(8)	-0.0020(7)	-0.0022(7)	-0.0030(7)
C(4)	0.0244(10)	0.0252(10)	0.0335(10)	0.0051(8)	0.0016(8)	-0.0045(8)
C(5)	0.0246(9)	0.0296(10)	0.0300(9)	0.0036(7)	0.0043(8)	-0.0063(8)
C(6)	0.0260(9)	0.0231(9)	0.0262(8)	-0.0024(7)	-0.0006(7)	-0.0056(7)
C(7)	0.0260(10)	0.029(1)	0.0317(9)	0.0065(8)	0.0025(8)	-0.0017(8)
C(8)	0.0266(9)	0.031(1)	0.0306(9)	0.0043(8)	0.0065(8)	-0.0018(8)
C(9)	0.040(1)	0.044(1)	0.032(1)	0.001(1)	0.0074(9)	0.001(1)
C(10)	0.041(1)	0.035(1)	0.034(1)	0.010(1)	0.0013(10)	0.0034(9)
C(11)	0.0345(10)	0.036(1)	0.0273(9)	-0.0037(9)	-0.0012(8)	-0.0011(9)
C(12)	0.0327(9)	0.0307(10)	0.0273(9)	0.0040(8)	-0.0009(9)	-0.0024(8)
C(13)	0.039(1)	0.033(1)	0.046(1)	-0.0007(10)	-0.0012(10)	0.0024(9)

The general temperature factor expression:

$$\exp(-2\pi^2(a^*{}^2U_{11}h^2 + b^*{}^2U_{22}k^2 + c^*{}^2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Table 3. Bond Lengths($\text{\AA}$)

atom	atom	distance	atom	atom	distance
N(1)	N(2)	1.323(3)	N(1)	C(1)	1.342(3)
N(2)	N(3)	1.338(3)	N(2)	C(11)	1.471(3)
N(3)	C(2)	1.346(3)	N(4)	C(6)	1.377(3)
N(4)	C(9)	1.446(3)	N(4)	C(10)	1.442(3)
C(1)	C(2)	1.405(3)	C(2)	C(3)	1.465(3)
C(3)	C(4)	1.393(3)	C(3)	C(8)	1.396(3)
C(4)	C(5)	1.381(3)	C(5)	C(6)	1.409(3)
C(6)	C(7)	1.408(3)	C(7)	C(8)	1.389(3)
C(11)	C(12)	1.500(4)	C(12)	C(13)	1.302(4)
C(1)	H(5)	1.01	C(4)	H(1)	1.04
C(5)	H(2)	1.01	C(7)	H(3)	1.09
C(8)	H(4)	1.01	C(9)	H(11)	0.96
C(9)	H(12)	0.95	C(9)	H(13)	0.91
C(10)	H(14)	0.94	C(10)	H(15)	0.99
C(10)	H(16)	0.97	C(11)	H(6)	1.10
C(11)	H(7)	0.97	C(12)	H(8)	0.94
C(13)	H(9)	0.97	C(13)	H(10)	0.95

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
N(2)	N(1)	C(1)	103.2(2)	N(1)	N(2)	N(3)	116.1(2)
N(1)	N(2)	C(11)	122.2(2)	N(3)	N(2)	C(11)	121.6(2)
N(2)	N(3)	C(2)	103.6(2)	C(6)	N(4)	C(9)	121.0(2)
C(6)	N(4)	C(10)	120.4(2)	C(9)	N(4)	C(10)	118.5(2)
N(1)	C(1)	C(2)	109.3(2)	N(3)	C(2)	C(1)	107.7(2)
N(3)	C(2)	C(3)	122.7(2)	C(1)	C(2)	C(3)	129.6(2)
C(2)	C(3)	C(4)	121.1(2)	C(2)	C(3)	C(8)	121.3(2)
C(4)	C(3)	C(8)	117.6(2)	C(3)	C(4)	C(5)	121.5(2)
C(4)	C(5)	C(6)	121.4(2)	N(4)	C(6)	C(5)	121.4(2)
N(4)	C(6)	C(7)	121.4(2)	C(5)	C(6)	C(7)	117.1(2)
C(6)	C(7)	C(8)	120.8(2)	C(3)	C(8)	C(7)	121.7(2)
N(2)	C(11)	C(12)	111.1(2)	C(11)	C(12)	C(13)	124.5(2)
N(1)	C(1)	H(5)	123.3	C(2)	C(1)	H(5)	127.3
C(3)	C(4)	H(1)	123.6	C(5)	C(4)	H(1)	114.9
C(4)	C(5)	H(2)	113.4	C(6)	C(5)	H(2)	125.0
C(6)	C(7)	H(3)	110.4	C(8)	C(7)	H(3)	128.3
C(3)	C(8)	H(4)	115.2	C(7)	C(8)	H(4)	123.1
N(4)	C(9)	H(11)	109.1	N(4)	C(9)	H(12)	110.3
N(4)	C(9)	H(13)	111.0	H(11)	C(9)	H(12)	109.0
H(11)	C(9)	H(13)	108.1	H(12)	C(9)	H(13)	109.4
N(4)	C(10)	H(14)	109.1	N(4)	C(10)	H(15)	117.9
N(4)	C(10)	H(16)	107.1	H(14)	C(10)	H(15)	108.2
H(14)	C(10)	H(16)	108.2	H(15)	C(10)	H(16)	106.0
N(2)	C(11)	H(6)	99.1	N(2)	C(11)	H(7)	105.3
C(12)	C(11)	H(6)	118.8	C(12)	C(11)	H(7)	108.4
H(6)	C(11)	H(7)	113.1	C(11)	C(12)	H(8)	110.3
C(13)	C(12)	H(8)	125.1	C(12)	C(13)	H(9)	119.8
C(12)	C(13)	H(10)	121.7	H(9)	C(13)	H(10)	118.4