Supporting Information

Palladium-Catalyzed Reaction of Cyclobutanone *O*-Benzoyloximes via C–C Bond Cleavage

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General Method. ¹H NMR spectra were obtained in CDCl₃ or C₆D₆ at 300 or 400 MHz with Me₄Si as an internal standard. ¹³C NMR spectra were obtained at 75.5 or 100 MHz. ³¹P NMR spectra were obtained at 161.9 MHz.

Materials. Commercially available organic and inorganic compounds were used without further purification except for solvent, which was distilled by the known method before use.¹⁵ Pd (dba)₂ (dba = dibenzylideneacetone) was synthesized by the literature

method.¹⁶ Cyclobutanone *O*-benzoyloximes were prepared according to the reported procedures from the corresponding cyclobutanone oximes.^{4f} Cyclobutanones were obtained from the reduction of 2,2-dichlorocyclobutanones which were prepared from the corresponding alkenes and trichloroacetyl chloride.¹⁶ (*R*)-MeO-MOP and (*S*)-H-MOP⁹ were prepared according to the reported procedures.

Typical Procedure for Preparation of 3-Phenylcyclobutanone *O*-Benzoyloxime (1). To a mixture of hydroxylamine hydrochloride (12 mmol), sodium acetate (15 mmol), ethanol (7 mL) and water (3 mL) in a 30-mL two-necked flask was added 3-phenylcyclobutanone (10 mmol) and the mixture was stirred at 100 °C for 12 h. The reaction mixture was cooled down to room temperature and then ethanol was removed under reduced pressure and the resulting mixture was extracted with diethyl ether. The organic layer was washed with water and dried over MgSO₄. The solvent was removed under vacuum and the residue was subjected to column chromatography on SiO₂ with EtOAc/hexane (2/8) as an eluent to give 3-phenylcyclobutanone oxime (99%). To a mixture of 3-phenylcyclobutanone oxime (5 mmol), triethylamine (10 mmol) and CH₂Cl₂ (10 mL) in a 30-mL two-necked flask was added benzoyl chloride (7 mmol) at 0 °C. After 6 h, water was added to the above solution, and the mixture was diluted with diethyl ether. The organic layer was washed with water and dried over MgSO₄. The solvent was removed under vacuum and the residue was subjected to column chromatography on SiO₂ with EtOAc/hexane (1/9) as an eluent to give **1** (4.3 mmol, 86%) as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ 3.20–3.32 (m, 2H), 3.54–3.77 (m, 3H), 7.24–7.30 (m, 3H), 7.34–7.42 (m, 2H), 7.47 (t, *J* = 7.3 Hz, 2H), 7.60 (t, *J* = 7.3 Hz, 1H), 8.05–8.09 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 32.5, 39.4, 39.5, 126.3, 126.9, 128.5, 128.7, 128.9, 129.6, 133.2, 143.0, 163.9, 166.0. Anal. Calcd for C₁₇H₁₅NO₂: C, 76.96; H, 5.70; N, 5.71. Found: C, 77.17; H, 5.67; N, 5.70.

(E)-Bicyclo[4.2.0]octan-7-one O-benzoyloxime [(E)-4]. Generally, it is possible to distinguish syn and anti oximes by ^{13}C NMR, as the chemical shift of α -carbon syn to OH appears upfield than that of the

carbon anti to OH at the same position. Thus, we estimated the configuration of (E)-4 and (Z)-4 according to this trend. Colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 1.30–2.00 (m, 8H), 2.40–2.52 (m, 1H), 2.73 (ddd, $J = 16.1, 5.1, 2.4$ Hz, 1H), 3.02 (ddd, $J = 16.1, 8.6, 1.7$ Hz, 1H), 3.40–3.50 (m, 1H), 7.43–7.48 (m, 2H), 7.55–7.61 (m, 1H), 8.01–8.05 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.0, 21.6, 23.7, 25.8, 26.7, 35.8, 44.2, 128.4, 129.1, 129.4, 133.0, 164.2, 170.5. Anal. Calcd for $\text{C}_{15}\text{H}_{17}\text{NO}_2$: C, 74.05; H, 7.04; N, 5.76. Found: C, 73.84; H, 7.27; N, 5.75.

(Z)-Bicyclo[4.2.0]octan-7-one O-benzoyloxime [(Z)-4]. White solid; mp 73.0–73.2 °C; ^1H NMR (400 MHz, CDCl_3) δ 1.25–1.75 (m, 6H), 1.80–2.05 (m, 2H), 2.42–2.52 (m, 1H), 2.79 (ddd, $J = 16.1, 4.4, 2.4$ Hz, 1H), 3.05 (dd, $J = 16.1, 8.3$ Hz, 1H), 3.37–3.44 (m, 1H), 7.46 (t, $J = 7.3$ Hz, 2H), 7.58 (t, $J = 7.3$ Hz, 1H), 8.05 (d, $J = 7.3$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.4, 21.6, 23.4, 26.8, 27.8, 37.1, 43.1, 128.4, 129.4, 129.6, 133.1, 164.2, 171.0. Anal. Calcd for $\text{C}_{15}\text{H}_{17}\text{NO}_2$: C, 74.05; H, 7.04; N, 5.76. Found: C, 74.11; H, 7.09; N, 5.73.

3,3-Dibutylcyclobutanone O-benzoyloxime (19). Colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 0.93 (t, $J = 7.3$ Hz, 6H), 1.17–1.39 (m, 8H), 1.50–1.56 (m, 4H), 2.76 (d, $J = 3.9$ Hz, 2H), 2.78 (d, $J = 3.9$ Hz, 2H), 7.45 (t, $J = 7.8$ Hz, 2H), 7.58 (t, $J = 7.8$ Hz, 1H), 8.05 (d, $J = 7.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 14.1, 23.1,

26.5, 36.7, 37.3, 42.2, 42.2, 128.5, 129.2, 129.6, 133.2, 164.1, 167.0. Anal. Calcd for $C_{19}H_{27}NO_2$: C, 75.71; H, 9.03; N, 4.65. Found: C, 75.71; H, 8.82; N, 4.56.

Spiro[3.5]nonan-2-one *O*-benzoyloxime (21). Colorless oil; 1H NMR (400 MHz, $CDCl_3$) δ 1.38–1.62 (m, 10H), 2.77 (d, $J = 3.9$ Hz, 2H), 2.79 (d, $J = 3.9$ Hz, 2H), 7.43–7.48 (m, 2H), 7.55–7.61 (m, 1H), 8.03–8.08 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.2, 25.4, 34.9, 37.3, 42.6, 42.7, 128.5, 129.2, 129.6, 133.2, 164.1, 166.8. Anal. Calcd for $C_{16}H_{19}NO_2$: C, 74.68; H, 7.44; N, 5.44. Found: C, 74.85; H, 7.51; N, 5.34.

3,3-Dibenzylcyclobutanone *O*-benzoyloxime (23). Colorless oil; 1H NMR (300 MHz, $CDCl_3$) δ 2.91 (s, 4H), 2.99 (s, 4H), 7.17 (d, $J = 6.8$ Hz, 4H), 7.24–7.35 (m, 6H), 7.45 (t, $J = 7.7$ Hz, 2H), 7.58 (t, $J = 7.3$ Hz, 1H), 8.02 (d, $J = 7.3$ Hz, 2H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ 38.0, 40.1, 40.2, 43.9, 126.7, 128.4, 129.0, 129.6, 130.1, 133.2, 137.6, 157.8, 163.9, 165.2. Anal. Calcd for $C_{25}H_{23}NO_2$: C, 81.27; H, 6.27; N, 3.79. Found: C, 81.02; H, 6.26; N, 3.78.

Preparation of Allyl Substituted Cyclobutanone *O*-Benzoyloximes. A typical procedure to prepare allyl substituted *O*-benzoyloximes is as follows: to a mixture of allyl substituted oximes, which were synthesized from the corresponding oximes and allyl bromide by the literature methods,^{s1} and triethylamine (5.0 equiv.) in CH_2Cl_2 was added benzoyl chloride (1.2 equiv.) at 0 °C and the mixture was stirred for 2 h. After the general workup, the products were purified by column chromatography on SiO_2 .

Colorless oil; ^1H NMR (300 MHz, C_6D_6) δ 0.68 (d, $J = 6.6$ Hz, 3H), 0.74 (d, $J = 6.6$ Hz, 3H), 0.78 (d, $J = 6.6$ Hz, 3H), 0.81 (d, $J = 6.6$ Hz, 3H), 0.97–1.06 (m, 2H), 1.34–1.46 (m, 2H), 1.35–1.58 (m, 2H), 2.11–2.24 (m, 1H), 2.34–2.46 (m, 1H), 2.38 (d, $J = 17.1$ Hz, 1H), 2.79 (dd, $J = 17.1, 3.0$ Hz, 1H), 2.93–3.03 (m, 1H), 5.04 (d, $J = 10.2$ Hz, 1H), 5.11 (d, $J = 17.1$ Hz, 1H), 6.08–6.23 (m, 1H), 7.00–7.10 (m, 3H), 8.15–8.26 (m, 2H); ^{13}C NMR (75.5 MHz, C_6D_6) δ 22.9, 23.3, 24.7, 25.0, 25.3, 32.1, 39.2, 40.5, 43.6, 47.4, 54.4, 116.2, 128.6, 129.8, 130.4, 132.9, 136.9, 163.5, 170.2. Anal. Calcd for $\text{C}_{22}\text{H}_{31}\text{NO}_2$: C, 77.38; H, 9.15; N, 4.10. Found: C, 77.52; H, 9.36; N, 4.05.

Colorless oil; ^1H NMR (300 MHz, C_6D_6) δ 0.67 (d, $J = 6.4$ Hz, 3H), 0.75 (d, $J = 6.4$ Hz, 3H), 0.77 (d, $J = 6.4$ Hz, 3H), 0.80 (d, $J = 6.4$ Hz, 3H), 1.00–1.18 (m, 2H), 1.33–1.60 (m, 4H), 2.22–2.47 (m, 3H), 2.70 (dd, $J = 16.2, 2.8$ Hz, 1H), 3.09–3.18 (m, 1H), 4.91 (d, $J = 9.9$ Hz, 1H), 5.05 (d, $J = 17.1$ Hz, 1H), 5.86 (dddd, $J = 17.1, 9.9, 6.8, 6.8$ Hz, 1H), 7.01–7.12 (m, 3H), 8.15–8.26 (m, 2H); ^{13}C NMR (75.5 MHz, C_6D_6) δ 22.7, 23.4, 24.7, 25.0, 25.3, 25.4, 31.7, 39.0, 41.1, 44.0, 46.4, 54.7, 116.3, 128.6, 129.9, 130.4, 132.8, 137.3, 163.7, 168.4.

Colorless oil; ^1H NMR (300 MHz, C_6D_6) δ 2.11–2.23 (m, 1H), 2.33 (d, $J = 16.2$ Hz, 1H), 2.43–2.65 (m, 4H), 2.78 (d, $J = 13.8$ Hz, 1H), 2.96 (dd, $J = 16.2, 1.8$ Hz, 1H), 3.16 (td, $J = 7.2, 1.8$ Hz, 1H), 4.98 (d, $J = 9.3$ Hz, 1H), 5.02 (d, $J = 15.6$ Hz, 1H), 5.80–5.94 (m, 1H), 6.77–6.81 (m, 2H), 7.00–7.16 (m, 11H),

8.12–8.16 (m, 2H); ^{13}C NMR (75.5 MHz, C_6D_6) δ 31.5, 38.5, 40.1, 41.8, 43.1, 51.8, 117.1, 127.3, 127.5, 129.2, 129.2, 129.2, 130.4, 131.3, 131.5, 133.5, 137.1, 138.4, 138.8, 164.2, 169.1. Anal. Calcd for $\text{C}_{28}\text{H}_{27}\text{NO}_2$: C, 82.12; H, 6.65; N, 3.42. Found: C, 81.86; H, 6.81; N, 3.44.

(Z)-1-Allylspiro[3.5]nonan-2-one O-benzoyloxime (13). Colorless oil; ^1H NMR (400 MHz, C_6D_6) δ 0.91–1.32 (m, 10H), 2.14–2.22 (m, 2H), 2.39–2.45 (m, 2H), 2.64 (t, $J = 7.5$ Hz, 1H), 4.99 (d, $J = 11.1$ Hz, 1H), 5.04 (d, $J = 17.1$ Hz, 1H), 5.84–5.94 (m, 1H), 7.03–7.15 (m, 3H), 8.18 (d, $J = 15.6$ Hz, 2H); ^{13}C NMR (100 MHz, C_6D_6) δ 22.9, 23.7, 26.0, 31.1, 38.1, 38.5, 38.9, 40.5, 54.4, 116.1, 128.6, 129.8, 130.4, 132.9, 136.9, 163.4, 169.1. Anal. Calcd for $\text{C}_{19}\text{H}_{23}\text{NO}_2$: C, 76.73; H, 7.80; N, 4.71. Found: C, 76.69; H, 7.90; N, 4.53.

4-Allyltricyclo[4.2.1.0^{2,5}]nonan-3-one O-benzoyloxime (16, E/Z mixture). The structure of this compound is estimated by the observation of different NOE spectra. Colorless oil; ^1H NMR (400 MHz, C_6D_6) δ 0.74–0.81 (m), 0.90–0.96 (m), 1.12–1.25 (m), 1.84–1.87 (m), 2.12–2.21 (m), 2.31–2.33 (m), 2.42–2.54 (m), 2.62–2.68 (m), 2.74–2.77 (m), 2.80–2.84 (m), 4.91–5.02 (m), 5.54–5.65 (m), 5.71–5.84 (m), 6.98–7.13 (m), 8.10–8.18 (m); ^{13}C NMR (100 MHz, C_6D_6) δ 27.2, 27.4, 28.1, 28.5, 33.6, 33.9, 34.8, 36.6, 37.4, 39.0, 39.6, 39.6, 41.5, 42.2, 46.9, 47.6, 50.1, 50.6, 116.7, 116.9, 128.5, 128.7, 129.8, 130.2, 130.3, 132.9, 135.0, 135.3, 163.3, 171.7, 172.1. Anal. Calcd for $\text{C}_{19}\text{H}_{21}\text{NO}_2$: C, 77.26; H, 7.17; N, 4.74. Found: C, 77.07; H, 7.22; N, 4.46.

Typical Procedure for the Reaction of Cyclobutanone *O*-Benzoyloximes (1, 4, 19, 21, 23). To a mixture of Pd₂(dba)₃·CHCl₃ (0.0125 mmol), (*R*)-(+)-BINAP (0.0375 mmol) and THF (2 mL) in a 10-mL two-necked flask were added K₂CO₃ (0.50 mmol), cyclobutanone *O*-benzoyloxime (0.50 mmol) and THF (3 mL) under N₂ and the mixture was stirred at 90 °C (bath temp.) until the reaction had reached completion by monitoring with TLC analysis. The reaction mixture was cooled down to room temperature and then filtered through a pad of Florisil. The filtrate was concentrated under vacuum to give a yellow oil, which was subjected to column chromatography on SiO₂ with diethyl ether-hexane (4/96) as an eluent. All major products were isolated and identified by ¹H and ¹³C NMR, IR and C, H, N combustion analysis.

(*E*)-3-Phenylbut-2-enitrile (3). Colorless oil; IR (neat) 3063, 2214 (CN), 1608, 1575, 1496, 1446, 1381, 1261, 827, 757, 692 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 2.47 (s, 3H), 5.62 (s, 1H), 7.38–7.48 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 20.2 (q), 95.6 (d), 117.6 (s), 125.9 (d), 128.8 (d), 130.3 (d), 138.3 (s), 159.8 (s). Anal. Calcd for C₁₀H₉N: C, 83.88; H, 6.34; N, 9.78. Found: C, 84.10; H, 6.52; N, 9.75.

2-Methylenecyclohexanecarbonitrile (5). Colorless oil; IR (neat) 2941, 2862, 2245 (CN), 1652, 1447, 901 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.47–2.14 (m, 7H), 2.43 (ddd, *J* = 13.9, 5.7, 3.9 Hz, 1H), 3.25 (dd, *J* = 9.3, 4.4 Hz, 1H), 4.93 (s, 1H), 5.09 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 24.2 (t), 27.1 (t), 32.0 (t), 33.5 (t), 35.8 (d), 110.6 (t), 120.1 (s), 142.2 (s). Anal. Calcd for C₈H₁₁N: C, 79.29; H, 9.15; N, 11.56. Found: C, 79.50; H, 9.07; N, 11.42.

Cyclohex-2-enylacetonitrile (6). Colorless oil; IR (neat) 3022, 2927, 2862,

2246 (CN), 1449, 1425, 724, 676 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.27–1.38 (m, 1H), 1.45–1.57 (m, 1H), 1.65–1.74 (m, 1H), 1.83–1.98 (m, 3H),

2.25 (dd, $J = 19.0, 16.6$ Hz, 1H), 2.27 (dd, $J = 17.8, 16.6$ Hz, 1H), 2.37–2.47 (m, 1H), 5.50 (dd, $J = 10.3, 2.4$ Hz, 1H), 5.78 (ddd, $J = 10.3, 5.9, 3.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 20.7 (t), 23.8 (t), 24.8 (t), 28.5 (t), 32.4 (d), 118.8 (s), 127.7 (d), 130.2 (d). Anal. Calcd for $\text{C}_8\text{H}_{11}\text{N}$: C, 79.29; H, 9.15; N, 11.56. Found: C, 79.21; H, 9.37; N, 11.27.

2,2-Dibutylcyclopropanecarbonitrile (20). Colorless oil; IR (neat) 2958,

2932, 2862, 2234 (CN), 1467 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.87–0.97 (m, 8H), 1.15 (dd, $J = 8.3, 5.4$ Hz, 1H), 1.22–1.60 (m, 12H); ^{13}C NMR (100 MHz, CDCl_3) δ 9.3 (d), 14.0 (q), 14.0 (q), 21.0 (t), 22.7 (t), 22.8 (t), 28.3 (t), 28.3 (s), 32.4 (t), 34.8 (t), 121.0 (s).

Anal. Calcd for $\text{C}_{12}\text{H}_{21}\text{N}$: C, 80.38; H, 11.81; N, 7.81. Found: C, 80.47; H, 11.64; N, 7.60.

Spiro[2.5]octane-1-carbonitrile (22). Colorless oil; IR (neat) 2931, 2855,

2233 (CN), 1779, 1446, 973, 881, 843, 791 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.92 (dd, $J = 5.4, 4.9$ Hz, 1H), 0.95 (dd, $J = 8.3, 4.9$ Hz, 1H), 1.15

(dd, $J = 8.3, 5.4$ Hz, 1H), 1.18–1.30 (m, 1H), 1.35–1.68 (m, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 9.1 (d), 20.9 (t), 24.9 (t), 25.3 (t), 25.4 (t), 27.7 (s), 32.2 (t), 35.2 (t), 120.8 (s). Anal. Calcd for $\text{C}_9\text{H}_{13}\text{N}$: C, 79.95; H, 9.69; N, 10.36. Found: C, 80.10; H, 9.69; N, 10.06.

2,2-Dibenzylcyclopropanecarbonitrile (24). Colorless oil; ^1H NMR (300

MHz, CDCl_3) δ 1.15 (dd, $J = 8.8, 5.3$ Hz, 1H), 1.26 (dd, $J = 5.3, 5.3$ Hz, 1H),

1.42 (dd, $J = 8.8, 5.3$ Hz, 1H), 2.56 (d, $J = 14.5$ Hz, 1H), 2.71 (d, $J = 14.5$ Hz, 1H), 2.81 (d, $J = 14.9$ Hz, 1H), 3.02 (d, $J = 14.9$ Hz, 1H), 7.01–7.04 (m, 2H), 7.23–7.38 (m, 8H); ^{13}C NMR

(75.5 MHz, CDCl₃) δ 8.1, 18.7, 29.0, 39.4, 39.5, 120.8, 126.8, 126.9, 128.5, 128.6, 129.3, 129.6, 136.7, 137.9. Anal. Calcd for C₁₈H₁₇N: C, 87.41; H, 6.93; N, 5.66. Found: C, 87.15; H, 6.98; N, 5.64.

(2-Benzylindan-2-yl)acetonitrile (25). White solid; mp 93.5–94.2 °C;

¹H NMR (300 MHz, CDCl₃) δ 2.30 (s, 2H), 2.79 (d, *J* = 15.8 Hz, 2H),

3.01 (s, 2H), 3.08 (d, *J* = 15.8 Hz, 2H), 7.14–7.37 (m, 9H); ¹³C NMR

(75.5 MHz, CDCl₃) δ 26.1, 43.5, 43.5, 47.1, 118.8, 125.0, 126.9, 126.9, 128.5, 130.0, 137.3, 140.9. Anal. Calcd for C₁₈H₁₇N: C, 87.41; H, 6.93; N, 5.66. Found: C, 87.12; H, 7.05; N, 5.55.

Typical Procedure for the Reaction of Cyclobutanone *O*-Benzoyloximes (7, 10, 13, 16, 23). To a mixture of Pd(dba)₂ (0.005 mmol), (*R*)-MeO-MOP(0.02 mmol) or (*R*)-*N*-cyclohexyl-*N*-methyl-1-[(*S*)-2-(diphenylphosphino)ferrocenyl]ethylamine (0.01 mmol), K₂CO₃ (0.10 mmol) and THF (0.5 mL) were added allyl substituted cyclobutanone *O*-benzoyloxime (0.10 mmol) and THF (1.5 mL) under N₂ and the mixture was stirred at 90 °C (bath temp.) for 24 h. After completion of the reaction, the reaction mixture was cooled down to room temperature and then filtered through a pad of Florisil. The filtrate was concentrated under vacuum to give a yellow oil, which was subjected to column chromatography on SiO₂. All major products were isolated and identified by ¹H and ¹³C NMR and C, H, N combustion analysis or mass spectroscopy.

2,2-Diisobutyl-4-methylenecyclopentanecarbonitrile (8). Colorless oil;

¹H NMR (300 MHz, CDCl₃) δ 0.84–1.01 (m, 12H), 1.34–1.78 (m, 6H), 2.22

(d, *J* = 16.2 Hz, 1H), 2.41 (d, *J* = 16.2 Hz, 1H), 2.58–2.85 (m, 3H), 4.92 (s,

1H), 4.95 (s, 1H); ¹³C NMR (75.5 MHz, CDCl₃) δ 24.1, 24.6, 24.7, 24.8, 24.9, 25.0, 25.2,

35.0, 38.4, 43.3, 44.1, 108.4, 121.2, 146.5. Anal. Calcd for C₁₅H₂₅N: C, 82.13; H, 11.49; N, 6.39. Found: C, 82.05; H, 11.22; N, 6.58.

3,3-Diisobutylhepta-4,6-dienenitrile (9). Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 0.89 (d, *J* = 6.6 Hz, 6H), 0.94 (d, *J* = 6.6 Hz, 6H), 1.36–1.45 (m, 2H), 1.50–1.55 (m, 2H), 1.57–1.63 (m, 2H), 2.52 (s, 2H), 5.06 (d, *J* = 10.0 Hz, 1H), 5.17 (d, *J* = 17.0 Hz, 1H), 5.61 (d, *J* = 16.2 Hz, 1H), 5.89 (dd, *J* = 16.2, 10.0 Hz, 1H), 6.31 (ddd, *J* = 17.0, 10.0, 10.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 24.3, 25.0, 25.1, 25.3, 42.2, 48.3, 116.4, 118.1, 128.7, 136.5, 139.9. Anal. Calcd for C₁₅H₂₅N: C, 82.13; H, 11.49; N, 6.39. Found: C, 82.12; H, 11.61; N, 6.15.

2,2-Dibenzyl-4-methylenecyclopentanecarbonitrile (11). Colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 1.95 (d, *J* = 16.2 Hz, 1H), 2.29 (d, *J* = 16.2 Hz, 1H), 2.70 (s, 3H), 2.74 (s, 1H), 2.83–2.92 (m, 3H), 4.96 (s, 1H), 5.03 (s, 1H), 7.04–7.08 (m, 2H), 7.18–7.38 (m, 8H); ¹³C NMR (75.5 MHz, CDCl₃) δ 33.6, 34.7, 38.1, 40.2, 40.6, 49.2, 109.2, 120.1, 126.7, 126.8, 128.2, 128.4, 130.8, 130.9, 136.2, 137.1, 145.4. Anal. Calcd for C₂₁H₂₁N: C, 87.76; H, 7.36; N, 4.87. Found: C, 87.36; H, 7.37; N, 4.86.

3,3-Dibenzylhepta-4,6-dienenitrile (12). White solid; mp 77.8–78.5 °C; ¹H NMR (300 MHz, CDCl₃) δ 2.14 (s, 2H), 2.95 (s, 4H), 5.04 (d, *J* = 9.9 Hz, 1H), 5.05 (d, *J* = 16.7 Hz, 1H), 5.52 (dd, *J* = 15.9 Hz, 9.9 Hz, 1H), 5.70 (d, *J* = 15.9 Hz, 1H), 6.32 (ddd, *J* = 16.7, 9.9, 9.9 Hz, 1H), 7.16–7.31 (m, 10H); ¹³C NMR (75.5 MHz, CDCl₃) δ 23.4, 43.9, 45.4, 117.3, 118.6, 126.9, 128.1, 130.6, 130.8, 136.0, 136.2, 137.0. Anal. Calcd for C₂₁H₂₁N: C, 87.76; H, 7.36; N, 4.87. Found: C, 87.50; H, 7.45; N, 4.76.

3-Methylenespiro[4,5]decane-1-carbonitrile (14). Colorless oil; ^1H

NMR (400 MHz, CDCl_3) δ 1.25–1.45 (m, 4H), 1.47–1.69 (m, 6H), 2.09 (d,

$J = 16.1$ Hz, 1H), 2.53 (d, $J = 16.1$ Hz, 1H), 2.60–2.69 (m, 2H), 2.71–2.81

(m, 1H), 4.92 (s, 1H), 4.95 (s, 1H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 22.6, 23.4, 25.8, 31.8, 34.7, 36.2, 40.0, 42.0, 45.5, 108.5, 120.7, 146.3. Anal. Calcd for $\text{C}_{12}\text{H}_{17}\text{N}$: C, 82.23; H, 9.76; N, 7.99. Found: C, 82.29; H, 9.64; N, 7.81.

(1-Buta-1,3-dienylcyclohexyl)acetonitrile (15). Colorless oil; ^1H NMR

(300 MHz, CDCl_3) δ 1.25–1.58 (m, 8H), 1.67–1.78 (m, 2H), 2.34 (s, 2H),

5.09 (d, $J = 10.1$ Hz, 1H), 5.21 (d, $J = 16.8$ Hz, 1H), 5.61 (d, $J = 15.7$ Hz,

1H), 6.14 (dd, $J = 15.7, 10.1$ Hz, 1H), 6.35 (ddd, $J = 16.8, 10.1, 10.1$ Hz, 1H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 22.0, 25.6, 30.2, 35.4, 38.2, 117.0, 117.8, 131.1, 136.8, 138.6. Anal. Calcd for $\text{C}_{12}\text{H}_{17}\text{N}$: C, 82.23; H, 9.78; N, 7.99. Found: C, 82.10; H, 9.74; N, 7.87.

3-Methyleneoctahydro-4,7-methanoindene-1-carbonitrile (17). The

structure of this compound is estimated by the observation of different

NOE spectra. Colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 1.12–1.23 (m,

3H), 1.41–1.46 (m, 1H), 1.49–1.61 (m, 2H), 2.22–2.28 (m, 2H), 2.49 (d, $J = 8.8$ Hz, 2H), 2.56–2.70 (m, 2H), 3.11 (dt, $J = 8.8, 8.8$ Hz, 1H), 4.93 (d, $J = 1.5$ Hz, 1H), 4.98 (d, $J = 1.5$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 27.8, 29.0, 31.8, 34.7, 39.7, 40.2, 42.9, 48.6, 53.3, 108.0, 121.0, 152.2. Anal. Calcd for $\text{C}_{12}\text{H}_{15}\text{N}$: C, 83.19; H, 8.73; N, 8.08. Found: C, 83.43; H, 8.76; N, 8.09.

3-Buta-1,3-dienylbicyclo[2.2.1]heptane-2-carbonitrile (18). Colorless oil; ^1H NMR (300 MHz, CDCl_3) δ 1.24–1.37 (m, 3H), 1.56–1.64 (m, 2H), 1.75–1.81 (m, 1H), 2.20–2.24 (m, 1H), 2.43 (dd, $J = 9.0, 9.0$ Hz, 1H), 2.63–2.66 (m, 1H), 2.68 (dd, $J = 9.0, 1.8$ Hz, 1H), 5.07 (d, $J = 10.1$ Hz, 1H), 5.17 (d, $J = 16.9$ Hz, 1H), 5.72 (dd, $J = 15.0, 9.0$ Hz, 1H), 6.10 (dd, $J = 15.0, 10.1$ Hz, 1H), 6.36 (ddd, $J = 16.9, 10.1, 10.1$ Hz, 1H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 28.1, 28.9, 35.2, 38.8, 42.4, 42.6, 47.7, 116.9, 120.3, 132.4, 133.7, 136.6. Anal. Calcd for $\text{C}_{12}\text{H}_{15}\text{N}$: C, 83.19; H, 8.73; N, 8.08. Found: C, 83.03; H, 8.77; N, 7.95.

Preparation of Methyl 2-(Methoxycarbonyl)-4-[1-methyl-3-(phenylcarboxyimino)cyclobutyl]butanoate (26).

The procedure for the preparation of the compound **26** is as follows: (a) A mixture of 2-(1-methyl-3-oxo-cyclobutyl)ethyl acetate (**I**) (16 g, 91 mmol), which was synthesized by the literature method,^{s2} methyl orthoformate (30 mL, 260 mmol) and *p*-toluenesulfonic acid monohydrate (95 mg, 0.5 mmol) in methanol (20 mL) was stirred at reflux for 16 h. After completion of the reaction, the solvent was removed in vacuo, and the crude material was purified by column chromatography on SiO_2 to give acetic acid 2-(3,3-dimethoxy-1-methylcyclobutyl)ethyl ester (**II**) (14 g, 65 mmol, 72%). (b) A mixture of **II** (1.1 g, 5.0 mmol) and K_2CO_3 in MeOH was stirred at room temperature for 5 h. After removal of MeOH in vacuo, Et_2O was added to the residue and the mixture was washed with water. The organic layer was dried over Na_2SO_4 and Et_2O was removed in vacuo. (c) Then, pyridine (5 mL) was

added to the residue. To the resulting mixture was added TsCl (1.1 g, 6.0 mmol) at 0 °C. After stirring at room temperature for 6 h, the reaction mixture was quenched with water/ice and extracted twice with Et₂O. The extract was dried over Na₂SO₄ and Et₂O was removed in vacuo. The crude material was purified by column chromatography to afford toluene-4-sulfonic acid 2-(3,3-dimethoxy-1-methylcyclobutyl)ethyl ester (**III**) (1.3 g, 4.1 mmol, 81%). (d) To a two-necked flask containing NaH (0.12 g, 5.0 mmol) in THF (4 mL) was added dimethyl malonate (0.66 g, 5.0 mmol). After stirring at room temperature for 1 h, **III** (0.99 g, 3.0 mmol) in THF (1 mL) was added. The reaction mixture was heated at reflux for 2 h after stirring at room temperature for 12 h. The reaction was quenched with saturated aqueous NH₄Cl. The mixture was extracted twice with Et₂O and the extract was dried over Na₂SO₄. Et₂O was removed in vacuo. (e) To the crude material were added CH₂Cl₂ (20 mL) and K10 montmorillonite (2.0 g). After the reaction mixture was stirred at room temperature for 12 h, the reaction mixture was filtered and then the filtrate was concentrated under vacuum. The crude material was purified by column chromatography to give 2-[2-(1-methyl-3-oxocyclobutyl)ethyl]malonic acid dimethyl ester (**IV**) (0.27 g, 1.0 mmol, 34%). (g) The compound **26** was prepared from **IV** as described above by the literature method (80%).⁶

Typical Procedure for the Reaction of 26. The procedure of the reaction of the compound **26** was the same as that of the reaction of allyl substituted cyclobutanone *O*-benzoyloximes using cyclobutanone *O*-benzoyloximes. The yield of the compound **27** was determined by GLC using pentamethylbenzene as an internal standard.

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3-Cyanomethyl-3-methylcyclopentane-1,1-dicarboxylic acid dimethyl ester (27). Colorless oil; ^1H NMR (300 MHz, CDCl_3)

δ 1.23 (s, 3H), 1.65–1.78 (m, 2H), 2.16–2.47 (m, 4H), 2.40 (s, 2H),

3.74 (s, 6H); ^{13}C NMR (75.5 MHz, CDCl_3), δ 26.5, 29.7, 33.4, 38.4, 41.6, 45.6, 52.9, 53.0, 60.1, 118.3, 172.4, 172.7. Anal. Calcd for $\text{C}_{12}\text{H}_{17}\text{NO}_4$: C, 60.24; H, 7.16; N, 5.85. Found: C, 60.51; H, 7.45; N, 5.62.

Preparation of 3-Methyl-3-(4-phenylbut-3-ynyl)cyclobutanone *O*-Benzoyloxime (28).

The procedure of the preparation of 3-methyl-3-(4-phenylbut-3-ynyl)cyclobutanone *O*-benzoyloxime (**28**) is as follows: (a) To a mixture of phenylacetylene (3.9 mL, 35 mmol) and 1,4-dioxane (70 mL) was added *n*-BuLi (23 mL, 35 mmol, 1.5 M in hexane) dropwise at 0 °C and the mixture was stirred at room temperature for 30 minutes under N_2 . Then, toluene-4-sulfonic acid 2-(3,3-dimethoxy-1-methylcyclobutyl)ethyl ester (**III**) (7.7 g, 20 mmol) in 1,4-dioxane (10 mL) was added and the resulting mixture was heated at refluxing temperature after stirring at room temperature for 30 minutes. After stirring at reflux for 15 h, the mixture

was cooled down to room temperature and quenched with saturated aqueous NH_4Cl . The mixture was extracted twice with Et_2O and the combined organic layer was washed with brine. The Et_2O solution was dried over MgSO_4 and filtered. The filtrate was concentrated under vacuum and the crude material was purified by column chromatography to give [4-(3,3-dimethoxy-1-methylcyclobutyl)but-1-ynyl]benzene (**V**) (3.9 g, 15 mmol, 76%). (b) To **V** (3.9 g, 15 mmol) in CH_2Cl_2 (80 mL) was added K10 montmorillonite (3 g) and the resulting mixture was stirred at room temperature for 15 h. The mixture was filtered and the filtrate was concentrated under vacuum. The crude material was purified by column chromatography to give 3-methyl-3-(4-phenylbut-3-ynyl)cyclobutanone (**VI**) (2.3 g, 11 mmol, 71 %). The compound **28** was prepared from 3-methyl-3-(4-phenylbut-3-ynyl)cyclobutanone (**VI**) by the reported method (56%).⁶

Typical Procedure for the Reaction of 28 with Alkenes. To a mixture of $\text{Pd}(\text{dba})_2$ (0.0050 mmol), (*R*)-(+)-BINAP (0.0075 mmol), K_2CO_3 (0.10 mmol) and DMF (0.5 mL) were added alkene (1.0 mmol), **28** (0.10 mmol) and DMF (1.5 mL) under N_2 and the resulting mixture was stirred at 90 °C until the reaction completed. After completion of the reaction,

the reaction mixture was cooled down to room temperature and then filtered through a pad of Florisil. The filtrate was concentrated under vacuum to give a yellow oil, which was subjected to column chromatography on SiO₂. All major products were isolated and identified by ¹H and ¹³C NMR and C, H, N combustion analysis or mass spectroscopy.

[1-Methyl-3-(1-phenylnon-2-enylidene)-cyclopentyl]acetonitrile (29c). Colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 0.87 (t, $J = 6.8$ Hz, 3H), 1.14–1.35 (m, 11H), 1.48–1.66 (m, 2H), 1.98–2.07 (m, 2H), 2.09–2.29 (m, 2H), 2.38 (s, 2H), 2.47 (d, $J = 16.6$ Hz, 1H), 2.54 (d, $J = 16.6$ Hz, 1H), 5.11 (ddd, $J = 14.9, 7.3, 7.3$ Hz, 1H), 6.34 (d, $J = 14.9$ Hz, 1H), 7.06–7.10 (m, 2H), 7.22–7.28 (m, 1H), 7.30–7.36 (m, 2H); ^{13}C NMR (67.8 MHz, CDCl_3) δ 14.2, 22.7, 25.4, 29.0, 29.2, 29.5, 30.5, 31.7, 33.2, 37.7, 41.2, 43.2, 118.6, 126.3, 128.1, 129.2, 129.7, 132.6, 134.3, 138.3, 140.6. Anal. Calcd for $\text{C}_{23}\text{H}_{31}\text{N}$: C, 85.92; H, 9.72; N, 4.36. Found: C, 85.64; H, 9.81; N, 4.38.

[3-(4,4-Dimethyl-1-phenylpent-2-enylidene)-1-methylcyclopentyl]acetonitrile (29d). Colorless oil; ^1H NMR (300 MHz, CDCl_3) δ 0.98 (s, 9H), 1.24 (s, 3H), 1.50–1.66 (m, 2H), 2.10–2.25 (m, 2H), 2.39 (s, 2H), 2.47 (d, $J = 17.3$ Hz, 1H), 2.55 (d, $J = 17.3$ Hz, 1H), 5.15 (d, $J = 15.8$ Hz, 1H), 6.25 (d, $J = 15.8$ Hz, 1H), 7.05–7.09 (m, 2H), 7.24–7.38 (m, 3H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 25.4, 29.1, 29.6, 30.5, 33.3, 37.6, 41.1, 43.1, 118.7, 124.5, 126.3, 128.1, 129.4, 134.3, 138.7, 140.7, 143.4. Anal. Calcd for $\text{C}_{21}\text{H}_{27}\text{N}$: C, 85.95; H, 9.27; N, 4.77. Found: C, 85.66; H, 9.30; N, 4.56.

[1-Methyl-3-(1-phenyl-3-trimethylsilylallylidene)cyclopentyl]acetonitrile (29e). White solid; mp 96.2–96.7 °C; ^1H NMR (300 MHz, CDCl_3) δ 0.04 (s, 9H), 1.26 (s, 3H), 1.51–1.70 (m, 2H), 2.14–2.30 (m, 2H), 2.41 (s, 2H), 2.54 (d, $J = 16.8$ Hz, 1H), 2.61 (d, $J = 16.8$ Hz, 1H), 5.33 (d, $J = 18.7$ Hz, 1H), 6.86 (d, $J = 18.7$ Hz, 1H), 7.04–7.08 (m, 2H), 7.25–7.30 (m, 1H), 7.33–7.39 (m, 2H); ^{13}C NMR (75.5 MHz, CDCl_3) δ -

1.1, 25.4, 29.0, 30.9, 37.3, 41.2, 43.0, 118.5, 126.5, 128.2, 129.5, 130.9, 136.2, 140.0, 142.2, 142.6. Anal. Calcd for C₂₀H₂₇NSi: C, 77.61; H, 8.79; N, 4.53. Found: C, 77.51; H, 8.71; N, 4.47.

{3-[4-(Diphenylphosphinoyl)-1-phenylbut-2-enylidene]-1-methylcyclopentyl}acetonitrile (29f). White solid; mp 154.5–155.5 °C; ¹H NMR (400 MHz, CDCl₃) δ 1.19 (s, 3H), 1.48–1.63 (m, 2H), 2.06–2.24 (m, 2H), 2.32 (d, *J* = 17.1 Hz, 1H), 2.34 (s, 2H), 2.38 (d, *J* = 17.1 Hz, 1H), 3.12 (dd, *J* = 15.1, 7.6 Hz, 2H), 5.01 (ddt, *J* = 15.4, 7.6, 7.6 Hz, 1H), 6.37 (dd, *J* = 15.4, 4.4 Hz, 1H), 6.91 (d, *J* = 6.8 Hz, 2H), 7.18–7.27 (m, 3H), 7.43–7.55 (m, 6H), 7.69 (t, *J* = 9.3 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 25.4, 29.1, 30.5, 35.2, 35.9, 37.5, 41.1, 43.2, 118.4, 119.3, 119.4, 126.5, 128.1, 128.4, 128.5, 129.0, 131.0, 131.1, 131.6, 131.7, 132.9, 133.0, 133.7, 133.8, 135.4, 135.5, 139.6, 140.9, 141.0. HRMS (FAB): calcd for C₃₀H₃₁NOP (M+H⁺), 452.2143; found, 452.2141. Anal. Calcd for C₃₀H₃₀NOP: C, 79.80; H, 6.70; N, 3.10. Found: C, 79.21; H, 6.65; N, 3.09.

Colorless crystals of the compound **29f** suitable for X-ray analysis were obtained by recrystallization from *n*-hexane. The single crystal was sealed in a Pyrex glass capillary under N₂ atmosphere and used for data collection. All measurements were made on a Rigaku RAXIS imaging plate area detector with graphite monochromated Mo-Kα radiation. The positions of non-hydrogen atoms were determined by direct methods (SIR97) and subsequent Fourier syntheses. An ORTEP drawing of **29f** is shown in Figure S1.

Figure S1. ORTEP drawing of **29f**.

4-(3-Cyanomethyl-3-methylcyclopentylidene)-4-phenylbut-2-enoic acid ethyl ester (29g). Colorless oil; ^1H NMR (300 MHz, CDCl_3) δ 1.24 (s, 3H), 1.25 (t, $J = 7.1$ Hz, 3H), 1.55–1.76 (m, 2H), 2.25–2.34 (m, 2H), 2.42 (s, 2H), 2.68 (s, 2H), 4.16 (q, $J = 7.1$ Hz, 2H), 5.32 (d, $J = 15.4$ Hz, 1H), 7.03–7.09 (m, 2H), 7.28–7.40 (m, 3H), 7.68 (d, $J = 15.4$ Hz, 1H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 14.3, 25.3, 29.0, 31.4, 37.1, 41.3, 43.6, 60.2, 118.2, 119.0, 127.2, 128.7, 129.0, 133.6, 138.5, 143.3, 151.6, 167.5. Anal. Calcd for $\text{C}_{20}\text{H}_{23}\text{NO}_2$: C, 77.64; H, 7.49; N, 4.53. Found: C, 77.38; H, 7.51; N, 4.46.

[1-Methyl-3-(2-oxo-1-phenylpropylidene)cyclopentyl]acetonitrile (29h). Colorless oil; ^1H NMR (300 MHz, CDCl_3) δ 1.21 (s, 3H), 1.55–1.85 (m, 2H), 1.97 (s, 3H), 2.22–2.31 (m, 2H), 2.40 (s, 2H), 2.83 (d, $J = 20.2$ Hz, 1H), 2.90 (d, $J = 20.2$ Hz, 1H), 7.12–7.18 (m, 2H), 7.30–7.44 (m, 3H); ^{13}C NMR (75.5 MHz, CDCl_3) δ 25.3, 29.0, 30.8, 32.8,

36.2, 41.7, 46.8, 118.3, 127.4, 128.9, 129.0, 135.9, 139.8, 158.0, 199.5. Anal. Calcd for C₁₇H₁₉NO: C, 80.60; H, 7.56; N, 5.53. Found: C, 80.31; H, 7.62; N, 5.39.

Typical Procedure for the Reaction of 3,3-Diphenylcyclobutanone *O*-Benzoyloxime (30) with an Equimolar Tetrakis(triphenylphosphine)palladium. The procedure for the reaction of 3,3-diphenylcyclobutanone *O*-benzoyloxime (**30**) with Pd(PPh₃)₄ is as follows: to a mixture of Pd(PPh₃)₄ (0.37 g, 0.32 mmol) in THF was added 3,3-diphenylcyclobutanone *O*-benzoyloxime (**30**) (0.10g, 0.30 mmol) under N₂ and the mixture was heated to 50 °C. After stirring for 4 h at the same temperature, the mixture was concentrated under reduced pressure and the precipitate was washed with Et₂O. The precipitate was dried under vacuum to leave a yellow solid (0.28g).

3,3-Diphenylcyclobutanone *O*-benzoyloxime (30). White Solid; mp 117.0–117.7 °C; ¹H NMR (300 MHz, CDCl₃) δ 3.89 (s, 4H), 7.20–7.37 (m, 10H), 7.46 (dd, *J* = 7.7, 7.7 Hz, 2H), 7.60 (dd, *J* = 7.7, 7.7 Hz, 1H), 8.06 (d, *J* = 7.7 Hz, 2H); ¹³C NMR (75.5 MHz, CDCl₃) δ 45.5, 45.9, 46.0, 126.3, 126.6, 128.5, 128.7, 128.9, 129.6, 133.3, 146.8, 163.8, 164.6. Anal. Calcd for C₂₃H₁₉NO₂: C, 80.92; H, 5.61; N, 4.10. Found: C, 80.95; H, 5.71; N, 4.07.

Preparation of 1-Phenylhept-6-en-3-one *O*-Benzoyloxime (32, *E/Z* mixture). The compound **32** was prepared from 1-phenylhept-6-en-3-one oxime by the same method as that of the preparation of cyclobutanone *O*-benzoyloximes. 1-Phenylhept-6-en-3-one oxime was synthesized by the reported method.^{s1a,4f}

1-Phenylhept-6-en-3-one *O*-benzoyloxime (32, *E/Z* mixture). Colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 2.33–2.51 (m), 2.64 (t, *J* = 7.8 Hz), 2.70–2.86 (m), 2.89–3.02 (m),

5.00–5.14 (m), 5.76–5.92 (m), 7.17–7.34 (m), 7.43–7.50 (m), 7.55–7.63 (m), 8.01–8.10 (m).

Anal. Calcd for $C_{20}H_{21}NO_2$: C, 78.15; H, 6.89; N, 4.56. Found: C, 78.06; H, 7.07; N, 4.56.

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