Rhodium Catalyzed Intramolecular C-H Insertion of α -Aryl- α -diazo Ketones

Douglass F. Taber and Weiwei Tian*

Department of Chemistry and Biochemistry, University of Delaware, Newark, DE, 19716

taberdf@udel.edu

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General:

^1H NMR and ^{13}C NMR spectra were recorded, as solutions in deuteriochloroform (CDCl_3) unless otherwise indicated, at 400 MHz and 100 MHz, respectively. ^{13}C multiplicities were determined with the aid of a JVERT pulse sequence, differentiating the signals for methyl and methine carbons as "d" from methylene and quaternary carbons as "u". The infrared (IR) spectra were determined as neat oils. R_f values indicated refer to thin layer chromatography (TLC) on 2.5 x 10 cm, 250 μm analytical plates coated with silica gel GF, unless otherwise noted, and developed in the solvent system indicated. All glassware was oven dried and rinsed with dry solvent before use. THF and diethyl ether were distilled from sodium metal/benzophenone ketyl under dry nitrogen. Toluene and acetonitrile were distilled from calcium hydride under dry nitrogen. MTBE is methyl tert-butyl ether and PE is petroleum ether. All reactions were conducted under N_2 and stirred magnetically.

1-(4-Bromophenylmethyl)oxirane (4c):¹ In a round bottom flask, 1,4-dibromobenzene (10.0 g, 42.4 mmol), Mg (0.93 g, 38.5 mmol), iodine (trace) and 200 mL of THF were combined. The reaction was exothermic, reached reflux after 10 min. Then the reaction mixture was kept at reflux by heating until the Mg disappeared (about 30 min). After cooling to 0 °C, allyl bromide (4.66 g, 38.5 mmol) was added dropwise. The cooling bath was removed, and the mixture was stirred for 2 h. After quenching with saturated aqueous NH₄Cl, the reaction mixture was partitioned between MTBE and, sequentially, water, saturated aqueous NaHCO₃ and brine. The combined organic extract was dried (Na₂SO₄) and concentrated *in vacuo*. The residue was subject to bulb to bulb distillation (90 °C, 0.1 Torr) to afford the alkene (7.25 g) as a colorless oil.

In a round bottom flask, the alkene (7.25 g), THF (100 mL), H₂O (50 mL) and NBS (10.3 g, 57.8 mmol) were added sequentially. After 4 h at rt, the reaction mixture was partitioned between MTBE and, sequentially, water, saturated aqueous NaHCO₃ and brine. The combined organic extract was dried (Na₂SO₄) and concentrated *in vacuo*. Into the residue, 200 mL of acetone was added followed by 53.2 g of K₂CO₃ (385 mmol). The reaction mixture was stirred at rt for 1 day. Then it was partitioned between MTBE and, sequentially, water, saturated aqueous NaHCO₃ and brine. The combined organic extract was dried (Na₂SO₄) and concentrated *in vacuo*. The residue was subject to bulb to bulb distillation (150 °C, 0.1 Torr) to afford the epoxide **4c** (4.37 g, 20.5 mmol, 53% yield) as a colorless oil. TLC *R_f* (PE/MTBE = 6/4) = 0.60; ¹H NMR δ 2.51 (1H, dd, *J* = 2.4, 4.8 Hz), 2.76-2.82 (3H, m), 3.08-3.14 (1H, m), 7.10-7.14 (2H, m), 7.40-7.43 (2H, m); ¹³C NMR δ u 38.1, 46.8, 120.7, 136.2; d 52.2, 130.9, 131.7.

1-(4-Bromophenyl)-2-decanone (1a):² The ketone **1a** was synthesized by the same procedure as ketone **1e** in 40% yield. ¹H NMR δ 0.87 (3H, t, J = 7.0Hz), 1.22-1.28 (10H, m), 1.52-1.57(2H, m), 2.43 (2H, t, J = 7.4Hz), 3.60 (2H, s), 7.07 (2H, d, J = 8.4Hz), 7.44 (2H, d, J = 8.4 Hz); ¹³C NMR δ u 22.8, 23.9, 29.3, 29.5, 32.0, 42.4, 49.4, 121.2, 133.5, 208.0; d 14.3, 131.3, 131.9.

1-Cyclohexyl-3-phenyl-2-propanone (1b):³ The ketone **1b** was synthesized by the same procedure as ketone **1e** in 71% yield. TLC *R_f* (PE/MTBE = 8/2) = 0.62; ¹H NMR δ 0.79-0.90 (2H, m), 1.07-1.14 (1H, m), 1.19-1.29 (2H, m), 1.61-1.66 (5H, m), 1.79-1.85(1H, m), 2.30 (2H, d, J = 7.2 Hz), 3.65 (2H, s), 7.17-7.32 (5H, m); ¹³C NMR δ u 26.1, 26.2, 33.1, 49.7, 50.8, 134.3, 208.2; d 33.8, 127.0, 128.7, 129.5.

1-(4-Bromophenyl)-5-methyl-2-hexanone (1c): The ketone **1c** was synthesized by the same procedure as ketone **1e** in 60% yield. TLC *R_f* (PE/MTBE = 8/2) = 0.54; ¹H NMR δ 0.85 (6H, d, J = 6.4Hz), 1.41-1.51 (3H, m), 2.44 (2H, t, J = 6.4 Hz), 3.63 (2H, s), 7.05 (2H, d, J = 8.0 Hz), 7.42 (2H, d, J = 8.0 Hz); ¹³C NMR δ u 32.6, 40.4, 49.2, 121.0, 133.4, 207.9; d 22.4, 27.7, 131.3, 131.8; IR (film, cm⁻¹) 2956, 1716, 1488, 1070, 1012; HRMS calcd for C₁₃H₁₇O⁷⁹Br (M) 268.0463, obsd 268.0459.

1-(3-Methoxyphenyl)-2-octanone (1d): The ketone **1d** was synthesized by the same procedure as ketone **1e** in 89% yield. TLC *R_f* (PE/MTBE = 8/2) = 0.34; ¹H NMR δ 0.86 (3H, t, J = 6.8 Hz), 1.20-1.30 (6H, m), 1.50-1.56 (2H, m), 2.43 (2H, t, J = 7.2 Hz), 3.64 (2 H, s), 3.79 (3H, s), 6.70-6.82 (3H, m), 7.24 (1H, t, J = 8.0 Hz); ¹³C NMR δ u 22.6, 23.8, 28.9, 31.7, 42.0, 50.3, 136.0, 160.0, 208.6; d 14.1, 55.3, 112.5, 115.1, 121.9, 129.8; IR (film, cm⁻¹) 2930, 2857, 1712, 1490, 779; HRMS calcd for C₁₅H₂₂O₂ (M⁺) 234.1620, obsd 234.1615.

1-(4-Bromophenyl)-6-nonen-2-one (1f): The ketone **1f** was synthesized by the same procedure as ketone **1e** in 76% yield. TLC *R_f* (PE/MTBE = 8/2) = 0.61; ¹H NMR δ 0.93 (3H, t, J = 7.6 Hz), 1.57-1.65 (2H, m), 1.94-2.03 (4H, m), 2.44 (2H, t, J = 7.2 Hz), 3.62 (2H, s), 5.22-5.25 (1H, m), 5.36-5.39 (1H, m), 7.05 (2H, d, J = 8.4 Hz), 7.42 (2H, d, J = 8.4 Hz); ¹³C NMR δ u 20.6, 23.7, 26.4, 41.6, 49.3, 121.1, 133.4, 207.7; d 14.5, 128.0, 131.3, 131.8, 132.8; IR (film, cm⁻¹) 2929, 1713, 1488, 1174; HRMS calcd for C₁₅H₂₀BrO (M+H) 295.0698, obsd 295.0688.

1-(4-Bromophenyl)-1-diazo-2-decanone (2a): The diazo ketone **2a** was synthesized by the same procedure as diazo ketone **2e** in 99% yield. TLC R_f (PE/MTBE = 8/2) = 0.47; ^1H NMR δ 0.88 (3H, t, J = 6.8 Hz), 1.19-1.36 (10H, m), 1.64-1.78 (2H, m), 2.58 (2H, t, J = 7.6 Hz), 7.40 (2H, d, J = 8.8 Hz), 7.51 (2H, d, J = 8.8 Hz); ^{13}C NMR δ u 22.8, 24.7, 29.2, 19.3, 29.4, 31.9, 39.3, 71.6, 120.5, 124.9, 192.7; d 14.2, 127.0, 132.2; IR (film, cm^{-1}) 2918, 2072, 1641, 1189, 810; HRMS calcd for $\text{C}_{16}\text{H}_{21}^{79}\text{BrO}$ ($\text{M}-\text{N}_2$) 308.0776, obsd 308.0772.

3-Cyclohexyl-1-diazo-1-phenyl-2-propanone (2b):⁴ The diazo ketone **2b** was synthesized by the same procedure as diazo ketone **2e** (except the reaction time is 20 h) in 89% yield. TLC R_f (PE/MTBE = 8/2) = 0.62; ^1H NMR δ 0.93-1.04 (2H, m), 1.09-1.20 (1H, m), 1.21-1.34 (2H, m), 1.62-1.79 (5H, m), 1.87-1.97 (1H, m), 2.46 (2H, d, J = 7.2 Hz), 7.20-7.27 (1H, m), 7.41 (2H, t, J = 8.0 Hz), 7.50-7.53 (2H, m); ^{13}C NMR δ u 26.2, 26.3, 33.2, 46.8, 125.7, 192.8; d 35.3, 127.0, 129.1.

1-(4-Bromophenyl)-1-diazo-5-methyl-2-hexanone (2c): The diazo ketone **2c** was synthesized by the same procedure as diazo ketone **2e** in 95% yield. TLC R_f (PE/MTBE = 8/2) = 0.65; ^1H NMR δ 0.92 (6H, d, J = 6.4 Hz), 1.57-1.63 (3H, m), 2.56-2.61 (2H, m), 7.39-7.42 (2H, m), 7.49-7.53 (2H, m); ^{13}C NMR δ u 33.7, 37.5, 120.7, 124.9, 193.0; d 22.5, 28.0, 127.2, 132.3; IR (film, cm^{-1}) 2953, 2074, 1644, 1199, 819; HRMS calcd for $\text{C}_{13}\text{H}_{15}\text{O}^{79}\text{Br}$ ($\text{M}-\text{N}_2$) 266.0306, obsd 266.0295.

1-Diazo-1-(3-methoxyphenyl)-2-octanone (2d): The diazo ketone **2d** was synthesized by the same procedure as diazo ketone **2e** in 95% yield. TLC R_f (PE/MTBE = 8/2) = 0.64; ^1H NMR δ 0.88 (3H, t, J = 6.8 Hz), 1.25-1.37 (6H, m), 1.65-1.73 (2H, m), 2.58 (2H, t, J = 7.2 Hz), 3.81 (3H, s), 6.76-6.80 (1H, m), 6.98-7.01 (1H, m), 7.21 (1H, m), 7.30 (1H, t, J = 8 Hz); ^{13}C NMR δ u 22.6, 24.7, 29.0, 31.6, 39.3, 127.1, 160.1, 193.1; d 14.1, 55.4, 111.5, 112.7, 117.7, 129.9; IR (film, cm^{-1}) 2931, 2072, 1651, 1236, 775; HRMS calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2$ ($\text{M}-\text{N}_2$) 232.1463, obsd 232.1462.

(6Z)-1-(4-Bromophenyl)-1-diazo-6-nonen-2-one (2f): The diazo ketone **2f** was synthesized by the same procedure as diazo ketone **2e** in 95% yield. TLC R_f (PE/MTBE = 8/2) = 0.74; ^1H NMR δ 0.95 (3H, t, J = 7.6 Hz), 1.73-1.81 (2H, m), 1.98-2.14 (4H, m), 2.59 (2H, t, J = 7.2 Hz), 5.26-5.46 (2H, m), 7.39 (2H, d, J = 8.8 Hz), 7.51 (2H, d, J = 8.8 Hz); ^{13}C NMR δ u 20.8, 24.6, 26.6, 38.6, 120.7, 124.9, 192.5; d 14.5,

127.1, 127.9, 132.3, 133.2; IR (film, cm^{-1}) 2961, 2070, 1652, 1489; HRMS calcd for $\text{C}_{15}\text{H}_{17}^{79}\text{BrO}$ ($\text{M}^+ - \text{N}_2$) 292.0463, obsd 292.0457.

1-Phenyl-octahydro-inden-2-one (3b): The ketone **3b** was synthesized by the same procedure as ketone **3a** (except the addition of **2b** was conducted in 15 min and no DBU was added) in 40% yield. TLC R_f (PE/MTBE = 8/2) = 0.51; ^1H NMR δ 0.80-2.05 (20H, m), 2.34-2.45 (2H, m), 2.53-2.60 (2H, m), 2.96 (1H, d, J = 11.6 Hz), 3.69 (1H, d, J = 7.2 Hz), 7.06-7.35 (10H, m); ^{13}C NMR δ u 20.3, 24.8, 24.9, 26.2, 26.5, 26.7, 30.3, 31.5, 39.2, 45.5, 135.6, 137.6, 216.9, 218.0; d 33.6, 41.4, 42.9, 51.3, 62.8, 63.2, 126.9, 127.0, 128.3, 128.7, 128.7, 129.6; IR (film, cm^{-1}) 2924, 2853, 1741, 1449, 1078; HRMS calcd for $\text{C}_{15}\text{H}_{19}\text{O}$ ($\text{M} + \text{H}$) 215.1436, obsd 215.1434.

2-(4-Bromophenyl)-3,3-dimethylcyclopentanone (3c): The ketone **3c** was synthesized by the same procedure as ketone **3a** (except no DBU added) in 79% yield. TLC R_f (PE/MTBE = 8/2) = 0.31; ^1H NMR δ 0.72 (3H, s), 1.16 (3H, s), 1.86-1.93 (2H, m), 2.41-2.48 (2H, m), 3.16 (1H, s), 6.91-6.94 (2H, m), 7.44-7.47 (2H, m); ^{13}C NMR δ u 35.6, 36.2, 41.5, 121.4, 134.3, 217.5; d 22.5, 28.7, 67.3, 131.5, 132.1; IR (film, cm^{-1}) 2954, 1734, 1488, 1011; HRMS calcd for $\text{C}_{13}\text{H}_{15}\text{O}^{79}\text{Br}$ (M) 266.0306, obsd 266.0303.

2-(3-Methoxyphenyl)-3-propylcyclopentanone (3d): The ketone **3d** was synthesized by the same procedure as ketone **3a** in 71% yield. TLC R_f (PE/MTBE = 8/2) = 0.14; ^1H NMR δ 0.86 (3H, t, J = 7.2 Hz), 1.19-1.60 (6H, m), 2.26-2.35 (2H, m), 2.48-2.55 (1H, m), 2.87 (1H, d, J = 11.2 Hz), 3.79 (3H, s), 6.30 (1H, t, J = 2.0 Hz), 6.68 (1H, d, J = 7.6 Hz), 6.78-6.81 (1H, m), 7.25 (1H, t, J = 8.0 Hz); ^{13}C NMR δ u 20.4, 27.4, 36.9, 38.7, 139.8, 159.9, 218.0; d 14.3, 45.1, 55.3, 63.2, 112.2, 115.0, 121.3, 129.7; IR (film, cm^{-1}) 2956, 1743, 1584, 1256, 778; HRMS calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2$ (M^+) 232.1463, obsd 232.1472.

3-Propyl-2-(4-methylphenyl)cyclopentanone (3e): The ketone **3e** was synthesized by the same procedure as ketone **3a** in 42% yield. TLC R_f (PE/MTBE = 8/2) = 0.35; ^1H NMR δ 0.86 (3H, t, J = 7.2 Hz), 1.20-1.60 (6H, m), 2.22-2.31 (2H, m), 2.32 (3H, s), 2.47-2.54 (1H, m), 2.85 (1H, d, J = 12.0 Hz), 6.97 (2H, d, J = 8.0 Hz), 7.14 (2H, d, J = 8.0 Hz); ^{13}C NMR δ u 20.4, 27.4, 36.8, 38.6, 135.1, 136.7, 218.5; d 14.3, 21.2, 45.0, 62.9, 128.8, 129.5; IR (film, cm^{-1}) 2957, 2871, 1743, 1515, 804; HRMS calcd for $\text{C}_{15}\text{H}_{20}\text{O}$ (M^+) 216.1514, obsd 216.1505.

2-(4-Bromophenyl)-3-((1Z)-1-butenyl)cyclopentanone (3f): The ketone **3f** was synthesized by the same procedure as ketone **3a** in 81% yield. TLC R_f (PE/MTBE = 8/2) = 0.39; ^1H NMR δ 0.72 (3H, t, J = 7.6 Hz), 1.60-1.88 (3H, m), 2.19-2.25 (1H, m), 2.31-2.41 (1H, m), 2.54-3.61 (1H, m), 2.98 (1H, d, J = 6.0 Hz), 3.14-3.18 (1H, m), 5.24-5.29 (1H, m), 5.39-5.46 (1H, m), 6.96 (2H, d, J = 8.4 Hz), 7.43 (2H, d, J = 8.4 Hz); ^{13}C NMR δ 21.1, 28.3, 38.4, 121.1, 136.4, 216.7; d 14.3, 43.9, 62.2, 129.9, 130.6, 131.7, 134.4; IR (film, cm^{-1}) 2962, 1741, 1489, 1072, 1011; HRMS calcd for $\text{C}_{15}\text{H}_{17}\text{O}^{79}\text{Br}$ (M^+) 292.0463, obsd 292.0452.

(2R,3R, 6S, 7S)-2,3 -diphenyl-6-(3-Methoxyphenyl)-7-propyl-1,4-dioxaspiro[4.4]nonane and (2R,3R, 6R, 7R)-2,3 -diphenyl-6-(3-Methoxyphenyl)-7-propyl-1,4-dioxaspiro[4.4]nonane (6d and 7d): In a round bottom flask, ketone **3d** (29 mg, 0.13 mmol), R,R-hydrobenzoin (54 mg, 0.25 mmol), triethyl orthoformate (37 mg, 0.25 mmol), p-toluenesulfonic acid monohydrate (trace) and 1.5 mL of dichloromethane were combined. The mixture was stirred overnight at rt. Then the reaction mixture was directly chromatographed to afford a mixture of **6d** and **7d** as a yellow oil (32 mg, 0.08 mmol, 60% yield). TLC R_f (PE/MTBE = 8/2) = 0.75; ^1H NMR δ 0.82-0.87 (4.33H, m), 1.50-1.60 (7.89H, m), 2.11-2.22 (2.54H, m), 2.30-2.42 (2.14H, m), 2.48-2.60 (1.04H, m), 2.92 (0.34H, d, J = 12.4 Hz), 3.04 (0.98H, d, J = 12.0 Hz), 3.74 (0.37H, d, J = 8.4 Hz), 3.79 (1.06H, s), 3.82 (3.0H, s), 4.21 (1.00H, d, J = 8.4 Hz), 4.53 (1.35H, m), 6.62-6.67 (1.98H, m), 6.81-6.85 (0.41H, m), 6.90-6.93 (1.05H, m), 6.98-7.33 (17.1H, m); ^{13}C NMR δ 20.8, 20.8, 27.4, 28.1, 36.9, 37.3, 38.4, 118.5, 135.8, 136.1, 136.7, 137.3, 140.0, 140.1, 159.4, 159.5; d 14.4, 14.4, 40.8, 42.9, 55.3, 55.4, 59.7, 61.4, 85.0, 85.2, 85.3, 85.6, 112.4, 112.6, 115.8, 115.8, 122.9, 122.9, 126.4, 126.9, 127.2, 127.5, 128.2, 128.3, 128.3, 128.4, 128.4, 128.8, 128.9; IR (film, cm^{-1}) 2955, 1600, 1456, 1159, 1046; HRMS calcd for $\text{C}_{29}\text{H}_{32}\text{O}_3$ (M^+) 428.2351, obsd 428.2359.

(2*R*,3*R*, 6*S*, 7*S*)-2,3 -Diphenyl-6-(4-bromophenyl)-7-((1*Z*)-1-butenyl)-1,4-dioxaspiro[4.4]nonane and (2*R*,3*R*, 6*R*, 7*R*)-2,3 -Diphenyl-6-(4-bromophenyl)-7-((1*Z*)-1-butenyl)-1,4-dioxaspiro[4.4]nonane (6f and 7f): The ketal **6f** and **7f** were synthesized by the same procedure as ketal **6d** and **7d** in 40% yield. TLC R_f (PE/MTBE = 8/2) = 0.88; ^1H NMR δ 0.78 (1.20H, t, J = 7.2 Hz), 0.88 (3.26H, t, J = 7.6 Hz), 1.55-1.73 (1.76H, m), 1.85-2.15 (4.63H, m), 2.20-2.29 (1.16H, m), 2.35-2.48 (1.82H, m), 3.04 (0.39H, d, J = 12.0 Hz), 3.16 (1.0H, d, J = 12.0 Hz), 3.26-3.33 (0.38H, m), 3.41-3.50 (0.96H, m), 3.75 (0.37H, d, J = 8.4 Hz), 4.23 (1.00H, d, J = 8.4 Hz), 4.53-4.57 (1.40H, m), 5.13-5.22 (1.37H, m), 5.27-5.34 (1.44H, m), 6.58-6.63 (1.96H, m), 6.95-7.53 (21,15H, m); ^{13}C NMR δ u 21.2, 21.3, 28.5, 29.4, 37.3, 38.6, 117.9, 120.9, 135.5, 136.9, 137.0, 137.1; d 14.6, 14.7, 39.6, 42.0, 59.3, 60.9, 85.1, 85.3, 85.4, 85.8, 126.4, 126.9, 127.0, 127.4, 128.3, 128.5, 128.5, 128.5, 128.6, 128.6, 131.0, 131.1, 131.3, 131.8, 132.0, 132.2, 133.0, 133.1; IR (film, cm^{-1}) 2962, 1490, 1303, 1155, 1015; HRMS calcd for $\text{C}_{29}\text{H}_{30}\text{O}_2^{79}\text{Br}$ (MH^+) 489.1429, obsd 489.1416.

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4c

400MHz

CDCl₃

4c

100MHz

CDCl₃

100 MHz
 CDCl₃

1b

1b

100 MHz

CDCl₃

400 MHz

CDCl₃

1c

100 MHz

CDCl₃

1d

400 MHz

CDCl₃

1d

100 MHz

CDCl₃

100 MHz

CDCl₃

2f

2f

100 MHz

CDCl₃

3a

400 MHz

CDCl₃

3a

100 MHz

CDCl₃

3b

3b

100 MHz

CDCl₃

CDCl_3

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