Intermolecular Coupling of Alkenes to Heterocycles *via* Rhodium Catalyzed C-H Bond Activation

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I. Spectroscopic and Analytical Data

General Experimental

Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. All solvents were distilled under nitrogen from the following drying agents immediately prior to use: ether and tetrahydrofuran (THF) from sodium/benzophenone ketyl, and dichloromethane (CH_2Cl_2) from calcium hydride. All reagent solutions were handled under an inert nitrogen atmosphere using syringe and cannula techniques. All reactions unless otherwise noted were carried out in flame or oven-dried glassware under an inert nitrogen atmosphere. Reaction progress was monitored with thin layer chromatography on Merck 60 F₂₅₄ 0.25 micron silica plates. Unless otherwise specified, extracts were dried over Na_2SO_4 and solvents were removed with a rotary evaporator. Flash column chromatography was carried out using Merck 60 230-400 mesh silica gel. All infrared (IR) spectra were recorded on a Mattson Galaxy 3000 Fourier Transform spectrometer on thin film on NaCl plates or KBr pellets and only partial data are listed. ^1H NMR spectra were obtained on a Bruker AMX-400, AM-500 FT, or AMX-300 spectrometers. Unless otherwise noted, all NMR spectra were obtained in CDCl_3 ; chemical shifts are reported in ppm relative to internal standard CDCl_3 . Coupling constants are reported in Hz. Elemental analyses were performed by M-H-W Laboratories (Phoenix, AZ) and the University of California, Berkeley elemental analysis facility. Mass Spectrometry was performed by the University of California, Berkeley mass spectrometry facility on group instruments. Analytical data and synthetic protocols have been reported for the following compounds in the literature: compound **1**,ⁱ $[\text{RhCl}(\text{coe})_2]_2$,ⁱⁱ *tert*-butyldiphenylsilyl 5-hexen-1-yl ether,ⁱⁱⁱ and 4, 4, 5, 5-tetramethyl-2-vinyl-[1,3] dioxolane.^{iv}

General procedure coupling of heterocycles to alkenes

Method A. In air to an oven dried vessel with a Kontes stopper was added $[\text{RhCl}(\text{coe})_2]_2$ (5 mol% Rh) (coe = *cis*-cyclooctene), tricyclohexylphosphine (7.5 mol%),

lutidinium chloride (5 mol%) , and the heterocycle. The vessel was then placed under nitrogen. Under a positive pressure of nitrogen, THF (diluted to 0.1M in heterocycle) and the alkene (5 equiv) were added to the vessel. The vessel was closed with the Kontes stopper and then was subjected to three successive freeze pump thaw cycles. The vessel was then heated for 15 h at 150 °C. The reaction mixture was then dry loaded onto SiO₂ and purified by flash chromatography using EtOAc/hexanes.

Method B. In air to an oven dried vessel with a Kontes stopper was added [RhCl(coe)₂]₂ (5 mol% Rh) (coe = *cis*-cyclooctene), tricyclohexylphosphine (7.5 mol%), and lutidinium chloride (5 mol%). The vessel was then placed under nitrogen. Under a positive pressure of nitrogen, THF, the alkene (5 equiv), and the heterocycle (0.1M) were added to the vessel. The vessel was closed with the Kontes stopper and then was subjected to three successive freeze pump thaw cycles. The vessel was then heated for 15 h at 150 °C. The reaction mixture was then dry loaded onto SiO₂ and purified by flash chromatography using EtOAc/hexanes.

Method C. In air to an oven dried vessel with a Kontes stopper was added [RhCl(coe)₂]₂ (10 mol% Rh) (coe = *cis*-cyclooctene), tricyclohexylphosphine (15 mol%), lutidinium chloride (5 mol%) , and the heterocycle. The vessel was then placed under nitrogen. Under a positive pressure of nitrogen, THF (diluted to 0.1M in heterocycle) and alkene (5 equiv) were added to the vessel. The vessel was closed with the Kontes stopper and then was subjected to three successive freeze pump thaw cycles. The vessel was then heated for 15 h at 150 °C. The reaction mixture was then dry loaded onto SiO₂ and purified by flash chromatography using EtOAc/hexanes.

Method D. In air to an oven dried vessel with a Kontes stopper was added [RhCl(coe)₂]₂ (10 mol% Rh) (coe = *cis*-cyclooctene), tricyclohexylphosphine (15 mol%), and lutidinium chloride (5 mol%). The vessel was then placed under nitrogen. Under a positive pressure of nitrogen, THF, the alkene (5 equiv), and the heterocycle (0.1M) were

added to the vessel. The vessel was closed with the Kontes stopper and then was subjected to three successive freeze pump thaw cycles. The vessel was then heated for 15 h at 150 °C. The reaction mixture was then dry loaded onto SiO₂ and purified by flash chromatography using EtOAc/hexanes.

2-(3, 3-Dimethyl-butyl)-1*H*-benzimidazole (4). Reaction of benzimidazole (154 mg, 1.31 mmol) with neo-hexene (810 µL, 6.30 mmol) according to method **A** yielded the product (254 mg, 96%) as a white solid after column chromatography (10-25% EtOAc/hexanes). Before dry loading the crude reaction solution onto SiO₂, triethylamine was added to neutralize the lutidinium chloride. The crude solution was then dry loaded on to SiO₂ and purified by column chromatography. IR(KBr): 3057.2 (br, s), 1415.8, 1273.5, 736.7 cm⁻¹. ¹H NMR (CD₃OD, 500 MHz): δ 0.99 (s, 9H), 1.8-1.65 (m, 2H), 2.9-2.8 (m, 2H), 7.2-7.1 (m, 2H), 7.5-7.4 (m, 2H). ¹³C NMR (CD₃OD, 125 MHz): δ 156.1, 121.6, 41.9, 29.8, 28.0, 24.0 (Note: two carbons were not observed due the slow exchange of the N-H proton, which causes these signals to be broadened). Anal. Calcd for C₁₃H₁₈N₂: C, 77.18; H, 8.97; N, 13.85. Found: C, 77.04; H, 9.06; N, 13.77. EI: Calc'd for C₁₃H₁₈N₂ (M⁺): 202. Found: 202.

2-(3, 3-Dimethyl-butyl)-benzothiazole (5). Reaction of benzothiazole (145 µL, 1.28 mmol) with neo-hexene (810 µL, 6.30 mmol) according to method **B** yielded the product (272 mg, 97%) as a brown/orange solid after column chromatography (2.5% EtOAc/hexanes). Before dry loading the crude reaction solution onto SiO₂, triethylamine was added to neutralize the lutidinium chloride. The crude solution was then dry loaded on to SiO₂ and purified by column chromatography. IR(NaCl): 2954.7, 2905.1, 2864.9, 758.4, 728.6 cm⁻¹. ¹H NMR (500 MHz): δ 1.00 (s, 9H), 1.9-1.7 (m, 2H), 3.2-3.0 (m, 2H), 7.34 (app. triplet, 1H, *J* = 7.5), 7.44 (app. triplet, 1H, *J* = 7.5), 7.83 (d, 1H, *J* = 8), 7.96 (d, 1H, *J* = 8). ¹³C NMR (125 MHz): δ 173.0, 153.2, 135.1, 125.8, 124.5, 122.4, 121.4,

43.7, 30.6, 30.1, 29.1. Anal. Calcd for C₁₃H₁₇NS: C, 71.18; H, 7.81; N, 6.39. Found: C, 71.19; H, 7.89; N, 6.52.

2-(3, 3-Dimethyl-butyl)-1H-benzoxazole (6). Reaction of benzoxazole (154 mg, 1.27 mmol) with neo-hexene (810 μ L, 6.30 mmol) according to method **A** yielded the product (199 mg, 77%) as a white solid after column chromatography 2.5-10% EtOAc/hexanes). Before dry loading the crude reaction solution onto SiO₂, triethylamine was added to neutralize the lutidinium chloride. The crude solution was then dry loaded on to SiO₂ and purified by column chromatography. IR(NaCl): 3055.5, 2955.7, 2867.1, 1572.7, 1455.3, 1242.8, 744.8 cm⁻¹. ¹H NMR (500 MHz): δ 0.97 (s, 9H), 1.9-1.8 (m, 2H), 3.0-2.8 (m, 2H), 7.3-7.2 (m, 2H), 7.5-7.4 (m, 1H), 7.7-7.6 (m, 1H). ¹³C NMR (125 MHz): δ 167.9, 150.7, 141.3, 124.3, 124.0, 119.4, 110.1, 40.5, 30.3, 29.0, 24.3. Anal. Calcd for C₁₃H₁₇NO: C, 76.81; H, 8.43; N, 6.89 Found: C, 76.42; H, 8.80; N, 6.95.

2-(3, 3-Dimethyl-butyl)-4, 5-dimethyl-thiazole (7). Reaction of 4, 5-dimethylthiazole (140 μ L, 1.32 mmol) with neo-hexene (810 μ L, 6.30 mmol) according to method **B** yielded the product (257 mg, 99%) as a red/orange liquid after column chromatography (5% EtOAc/hexanes). Before dry loading the crude reaction solution onto SiO₂, triethylamine was added to neutralize the lutidinium chloride. The crude solution was then dry loaded on to SiO₂ and purified by column chromatography. IR(NaCl): 2954.0, 2866.1, 1477.8, 1365.0, 1121.5 cm⁻¹. ¹H NMR (500 MHz): δ 0.93 (s, 9H), 1.7-1.6 (m, 2H), 2.26 (s, 3H), 2.28 (s, 3H), 2.9-2.8 (m, 2H). ¹³C NMR (125 MHz): δ 167.5, 147.0, 124.7, 44.3, 30.5, 29.1, 29.0, 14.5, 11.2. Anal. Calcd for C₁₁H₁₉NS: C, 66.95; H, 9.70; N, 7.10 Found: C, 66.61; H, 9.58; N, 7.22.

8-(3, 3-Dimethyl-butyl)-7H-purine (8). Reaction of purine (152 mg, 1.27 mmol) with neo-hexene (810 μ L, 6.30 mmol) according to method **A** (with the exception that the reaction was only heated for 4.5 h) yielded the product (197 mg, 76%) as a white solid after column chromatography (50-100% EtOAc/hexanes). IR(KBr): 2926.5 (br, s),

1616.7, 1592.1, 920.8, 648.6, 614.1 cm^{-1} . ^1H NMR (CD_3OD , 500 MHz): δ 1.00 (s, 9H), 1.8-1.7 (m, 2H), 3.0-2.9 (m, 2H), 8.83 (s, 1H), 8.90 (s, 1H). ^{13}C NMR (125 MHz): δ 151.3, 41.3, 29.9, 27.9, 24.6 (Note: two carbons were not observed due the slow exchange of the N-H proton, which causes these signals to be broadened). Anal. Calcd for $\text{C}_{11}\text{H}_{16}\text{N}_4$: C, 64.68; H, 7.89; N, 27.43. Found: C, 64.48; H, 7.91; N, 27.04. EI: Calcd for $\text{C}_{11}\text{H}_{16}\text{N}_4$ (M^+): 204. Found: 204.

6, 8-Bis-(3, 3-Dimethyl-butyl)-7H-purine (9). Reaction of purine (152 mg, 1.27 mmol) with neo-hexene (810 μL , 6.30 mmol) according to method **A** (with the exception that the reaction was only heated for 4.5 h) yielded the product (61 mg, 17%) as a white solid after column chromatography (50-100% EtOAc/hexanes). IR(KBr): 2960.2 (br, s), 1594.9, 983.4, 653.1 cm^{-1} . ^1H NMR (400 MHz): δ 1.00 (s, 9H), 1.02 (s, 9H), 1.9-1.7 (m, 4H), 3.1-3.0 (m, 2H), 3.3-3.1 (m, 2H), 8.84 (s, 1H). ^{13}C NMR (100 MHz): δ 162.5, 157.9, 152.5, 150.2, 133.3, 42.2, 41.9, 30.7, 30.6, 29.3, 29.2, 28.6, 25.9. Anal. Calcd for $\text{C}_{17}\text{H}_{28}\text{N}_4$: C, 70.79; H, 9.78; N, 19.42. Found: C, 70.88; H, 9.86; N, 19.50.

2-(3, 3-Dimethyl-butyl)-1-methyl-benzimidazole (10). Reaction of 1-methylbenzimidazole (132 mg, 1.00 mmol) with neo-hexene (643 μL , 5 mmol) according to method **C** yielded the product (144 mg, 67%) as a white solid after column chromatography (10-30% EtOAc/hexanes). Before dry loading the crude reaction solution onto SiO_2 , triethylamine was added to neutralize the lutidinium chloride. IR(NaCl): 2953.9, 2864.7, 745.0 cm^{-1} . ^1H NMR (500 MHz): δ 1.02 (s, 9H), 1.79-1.75 (m, 2H), 2.85-2.82 (m, 2H), 3.71 (s, 3H), 7.30-7.20 (m, 3H), 7.72-7.70 (m, 1H). ^{13}C NMR (500 MHz): δ 155.8, 142.5, 135.8, 121.9, 121.7, 119.0, 108.7, 41.4, 30.4, 29.6, 29.1, 23.0. Anal. Calcd for $\text{C}_{14}\text{H}_{20}\text{N}_2$: C, 77.73; H, 9.32; N, 12.95. Found: C, 77.39; H, 9.54; N, 12.63.

2-Hexyl-1H-benzimidazole (11). Reaction of benzimidazole (151 mg, 1.28 mmol) with 1-hexene (538 mg, 6.40 mmol) according to method **A** yielded the product

(209 mg, 80%) as a white solid after column chromatography (20-50% EtOAc/hexanes). IR(KBr): 2930.1 (br, s), 1420.7, 1027.0, 750.2 cm^{-1} . ^1H NMR (CD_3OD , 500 MHz): δ 0.87 (t, 3H, $J=6.5$), 1.4-1.2 (m, 6H), 1.85-1.75 (m, 2H), 2.85 (t, 2H, $J=7.5$), 7.19-7.13 (m, 2H), 7.5-7.45 (m, 2H). ^{13}C NMR (CD_3OD , 125 MHz): δ 155.5, 121.6, 31.2, 28.5, 28.3, 27.9, 22.1, 12.9 (Note: two carbons were not observed due the slow exchange of the N-H proton, which causes these signals to be broadened). Anal. Calcd for $\text{C}_{13}\text{H}_{18}\text{N}_2$: C, 77.18; H, 8.97; N, 13.85. Found: C, 77.36; H, 9.03; N, 13.68. EI: Calc'd for $\text{C}_{13}\text{H}_{18}\text{N}_2$ (M^+): 202. Found: 202.

2-[6-(*tert*-Butyl-diphenyl-silanyloxy)-hexyl]-1*H*-benzimidazole (12). Reaction of benzimidazole (70 mg, 0.591 mmol) with *tert*-butyl-hex-5-enyloxy-diphenyl-silane (1 g, 2.95 mmol) according to method **A** yielded the product (207 mg, 77%) as a white solid after column chromatography (25-50% EtOAc/hexanes). IR(KBr): 2926.5 (br, s), 1426.2, 1273.8, 1109.3, 744.4, 701.0 cm^{-1} . ^1H NMR (CD_3OD , 500 MHz): δ 0.99 (s, 9H), 1.36-1.30 (m, 2H), 1.44-1.38 (m, 2H), 1.56-1.51 (m, 2H), 1.82-1.77 (m, 2H), 2.84 (t, 2H, $J=7.5$), 3.62 (t, 2H, $J=6.5$), 7.2-7.15 (m, 2H), 7.4-7.3 (m, 6H), 7.5-7.45 (m, 2H), 7.7-7.6 (m, 4H). ^{13}C NMR (CD_3OD , 125 MHz): δ 155.4, 135.2, 133.6, 129.3, 127.3, 121.7, 63.4, 32.0, 28.5, 28.3, 27.8, 25.9, 25.1, 18.5 (Note: two carbons were not observed due the slow exchange of the N-H proton, which causes these signals to be broadened). Anal. Calcd for $\text{C}_{29}\text{H}_{36}\text{N}_2\text{OSi}$: C, 76.27; H, 7.95; N, 6.13. Found: C, 76.16; H, 8.10; N, 6.06. FAB: Calc'd for $\text{C}_{29}\text{H}_{36}\text{N}_2\text{OSi}$ ($\text{M}+1$): 457. Found: 457.

4-(1*H*-Benzimidazol-2-yl)-butyric acid isobutyl ester (13). Reaction of benzimidazole (150 mg, 1.27 mmol) with isobutyl vinylacetate (903 mg, 6.35 mmol) according to method **C** (with the exception that the reaction was heated for only 8 h) yielded the product (288 mg, 87%) as a white solid after column chromatography (40-75% EtOAc/hexanes). IR(KBr): 2947.8 (br, s), 1732.9 9(s), 749.3 cm^{-1} . ^1H NMR (CD_3OD , 500 MHz): δ 0.89 (d, 6H, $J=6.5$), 1.90-1.82 (m, 1H), 2.15-2.10 (m, 2H), 2.41

(t, 2H, $J = 7.5$), 2.93 (t, 2H, $J = 7.5$), 3.80 (d, 2H, $J = 6.5$), 7.18-7.16 (m, 2H), 7.49-7.46 (m, 2H). ^{13}C NMR (CD_3OD , 125 MHz): δ 173.2, 154.4, 138.0 (br), 121.8, 113.9 (br), 70.2, 32.8, 27.6, 27.5, 23.0, 17.9. Anal. Calcd for $\text{C}_{15}\text{H}_{20}\text{N}_2\text{O}_2$: C, 69.20; H, 7.74; N, 10.76. Found: C, 69.14; H, 7.86; N, 10.73. EI: Calc'd for $\text{C}_{15}\text{H}_{20}\text{N}_2\text{O}_2$ (M^+): 260. Found: 260.

2-[2-(4, 4, 5, 5-Tetramethyl-[1, 3]dioxolan-2-yl)-ethyl]-1H-benzimidazole (14).

Reaction of benzimidazole (150 mg, 1.27 mmol) with 4, 4, 5, 5-tetramethyl-2-vinyl-[1, 3]dioxolane (992 mg, 6.35 mmol) according to method C yielded the product (209 mg, 60%) as a white solid after column chromatography (25-75% EtOAc/hexanes). Before dry loading the crude reaction solution onto SiO_2 , triethylamine was added to neutralize the lutidinium chloride. The crude solution was then dry loaded on to SiO_2 and purified by column chromatography. IR(KBr): 2976.2 (br, s), 1547.7, 1164.3, 1131.4, 758.9 cm^{-1} . ^1H NMR ($\text{DMSO}-d_6$, 500 MHz): δ 1.12 (s, 6H), 1.14 (s, 6H), 2.01-1.97 (m, 2H), 2.85 (t, 2H, $J = 8$), 5.04 (t, 1H, $J = 5$), 7.09-7.08 (m, 2H), 7.47-7.40 (m, 2H), 12.20-12.05 (m, 1H). ^{13}C NMR ($\text{DMSO}-d_6$, 125 MHz): δ 154.8, 121.5 (br), 99.6, 81.8, 34.4, 24.5, 24.0, 22.3 (Note: two carbons were not observed due to the slow exchange of the N-H proton, which causes these signals to be broadened). Anal. Calcd for $\text{C}_{16}\text{H}_{22}\text{N}_2\text{O}_2$: C, 70.04; H, 8.08; N, 10.21. Found: C, 69.99; H, 8.33; N, 10.06. FAB: Calc'd for $\text{C}_{16}\text{H}_{22}\text{N}_2\text{O}_2$ ($\text{M}+1$): 275. Found: 275.

2-Phenethyl-1H-benzimidazole (15). Reaction of benzimidazole (100 mg, 0.847 mmol) with styrene (491 μL , 4.24 mmol) according to method C yielded the product (36 mg, 19%) as a white solid after column chromatography (25-50% EtOAc/hexanes). The THF used in this reaction was dried over 4 Å molecular sieves instead of being distilled from Na^0 /benzophenone. GCMS analysis of the crude reaction mixture showed a linear:branched ratio of 25:1. IR(KBr): 2866.8 (br, s), 1420.2, 1280.1, 749.3, 696.2 cm^{-1} . ^1H NMR (CD_3OD , 500 MHz): δ 3.2-3.05 (m, 4H), 7.3-7.1 (m, 7H), 7.5-7.43 (m, 2H). ^{13}C NMR (CD_3OD , 125 MHz): δ 154.5, 140.3, 128.1, 127.9, 125.9, 121.7, 34.0, 30.5 (Note:

two carbons were not observed due the slow exchange of the N-H proton, which causes these signals to be broadened). HRMS: Calc'd for $C_{15}H_{14}N_2$ (M^+): 222.1158. Found: 222.1157.

3-(4, 5-Dimethyl-thiazol-2-yl)-propionic acid *tert*-butyl ester (16). Reaction of 4, 5-dimethylthiazole (97% pure, 135 μ L, 1.24 mmol) with *tert*-butyl acrylate (930 μ L, 6.35 mmol) according to method **D** yielded the product (278 mg, 93%) as a red-orange liquid after column chromatography (5-10% EtOAc/hexanes). Before dry loading the crude reaction onto SiO_2 , triethylamine was added to neutralize the lutidinium chloride. The crude solution was then dry loaded on to SiO_2 and purified by column chromatography. IR(NaCl): 2978.9, 2924.5, 1729.1, 1366.88, 1154.65 cm^{-1} . 1H NMR (500 MHz): δ 1.42 (s, 9H), 2.25 (s, 3H), 2.29 (s, 3H), 2.66 (t, 2H, $J=7.5$), 3.14 (t, 2H, $J=7.5$). ^{13}C NMR (125 MHz): δ 171.4, 164.3, 147.3, 125.2, 80.6, 35.1, 28.5, 28.0, 14.5, 11.2. Anal. Calcd for $C_{12}H_{19}NO_2S$: C, 59.72; H, 7.93; N, 5.80. Found: C, 59.75; H, 7.79; N, 6.00.

3-(4, 5-Dimethyl-thiazol-2-yl)-propionitrile(17). Reaction of 4, 5-dimethylthiazole (97% pure, 135 μ L, 1.24 mmol) with acrylonitrile (418 μ L, 6.35 mmol) according to method **D** yielded the product (134 mg, 65% linear product) as a yellow liquid after column chromatography (10-30% EtOAc/hexanes). GCMS analysis of the crude reaction mixture showed a linear:branched ratio of 4.3:1. IR(NaCl): 2922.8, 2864.8, 2249.3, 1439.7, 1134.8 cm^{-1} . 1H NMR (400 MHz): δ 2.27 (s, 3H), 2.30 (s, 3H), 2.80 (t, 2H, $J=7.6$), 3.19 (t, 2H, $J=7.6$). ^{13}C NMR (100 MHz): δ 160.6, 148.2, 126.5, 118.6, 28.9, 17.3, 14.6, 11.2. Anal. Calcd for $C_8H_{10}N_2S$: C, 57.80; H, 6.06; N, 16.85. Found: C, 57.81; H, 6.05; N, 17.04.

III. Additive Table

(a) General Procedure for Additive Screen

In the glove box, to a vial was added compound **1** (0.15M), PCy₃ (0.023M), [RhCl(coe)₂]₂ (0.015M in Rh), and dimethoxytoluene (internal standard) in THF. The solution (0.5 mL) was used to transfer the additive (10 mol%) into a vessel with a Kontes top. If the additive was not soluble then the solution in the reaction vessel was decanted off and was used to transfer the remaining additive into the vessel. This procedure was repeated until the additive was transferred. The reaction mixture was then heated to 105 °C for 15 to 17 h. The reaction mixture was cooled to rt and then the solution was concentrated. The residue was then dissolved in CDCl₃ and analyzed by ¹H NMR spectroscopy.

(b) Table S-1. Complete Additive Table

Additive 10%	% SM	% Product	Time
None	77	<7	15 h
Benzoic Acid	67	10	16 h
TfOH	Decomp	Decomp	16 h
NH ₄ Cl	66	11	16 h
H ₂ O pTSA	57	22	16 h
Pyridinium HCl	75	25	16 h
Pyridinium HBr	64	35	16 h
Lutidinium HI	53	39	15 h
Lutidinium HBr	54	42	15 h
Lutidinium HCL	32	49	17 h
Lutidinium pTSA	50	28	16 h
Bu ₄ NCl	70	8	15 h
PPNCl	71	9	15 h
Cu(OTf) ₂	Decomp	None	15 h

Cu(OTf) ₂ PheBox (in toluene)	Decomp	None	17 h
CuCl ₂	72	6	15 h
ZnCl ₂	72	12	15 h
Al(OTf) ₃	76	Trace	15 h
ScCl ₃	44	33	15 h
Sc(OTf) ₃	85	14	17 h
YCl ₃	19	69	16 h
YBr ₃	47	34	16 h
Mg(OTf) ₂	85	14	16 h
MgBr ₂	12	69	16 h
MgCl ₂	47	27	15 h
MgF ₂	72	-	17 h
MgI ₂	50	18	17 h
LiBr	58	24	16 h
NaBr	77	Trace	16 h
KBr	78	Trace	16 h
CaCl ₂	68	13	16 h

Run at 0.15 M substrate; yields by NMR

IV. Structural Data on **9**

Figure S-1-ORTEP of **9**

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