Palladium-Catalyzed Intramolecular Hydroalkylation of Unactivated Olefins with Dialkyl Ketones

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Supporting Information

Experimental procedures, analytical and spectroscopic data for new compounds and cyclohexanones (15 pages).

Experimental

General Methods. Reactions were performed under an atmosphere of nitrogen employing standard Schlenk techniques unless otherwise stated. NMR were obtained on a Varian spectrometer operating at 400 MHz for ^1H NMR and 100 MHz for ^{13}C NMR in CDCl_3 unless otherwise noted. IR spectra were obtained on a Bomen MB-100 FT IR spectrometer. Gas chromatography was performed on a HP 5890 gas chromatography equipped with a 25 m polydimethylsiloxane capillary column. Flash column chromatography was performed employing 200-400 mesh silica gel (EM); all compounds were isolated as colorless oils unless otherwise noted. Elemental analyses were performed by Complete Analysis Laboratories (Parsippany, NJ). Thin layer chromatography (TLC) was performed on SiO_2 plates eluting with a 5:1 mixture of hexanes–EtOAc unless noted otherwise. CH_2Cl_2 was distilled from CaH_2 prior to use. 1,4-Dioxane (Aldrich, anhydrous), DMSO (Aldrich, anhydrous) and $\text{PdCl}_2(\text{CH}_3\text{CN})_2$ (Strem) were used as received.

Alkenyl Ketones

3-Butenyl benzyl ketone (Table 1, entry 1),¹ benzyl 4-pentenyl ketone (Table 1, entry 9),² 3-butenyl heptyl ketone (Table 2, entry 2),³ 3-butenyl pentyl ketone (Table 2, entry 3),³ 3-butenyl phenethyl ketone (Table 2, entry 4),⁴ 1-(4-methoxyphenyl)-5-hexen-2-one (Table 2, entry 6),⁵ 3-butenyl cyclohexyl ketone (Table 2, entry 7),⁶ methyl 5-oxo-8-nonenate (Table 2, entry 8),⁷ were prepared by published procedures.

1-(4-Bromophenyl)-5-hexen-2-one (Table 1, entry 2). *n*-BuLi (2.5 M in hexanes, 2.4 mL, 6.0 mmol) was added dropwise to a solution of 2-but-3-enyl-[1,3]dithiane (1.04 g, 6.0 mmol) in THF (25 mL) at $-20\text{ }^\circ\text{C}$ and stirred for 2 h. The resulting solution was treated with a solution of 1-bromomethyl-4-bromobenzene (1.50 g, 6.0 mmol) in THF (5 mL) at $-78\text{ }^\circ\text{C}$, stirred for 10 min and warmed slowly to room temperature. The resulting mixture was treated with saturated aqueous NH_4Cl , extracted with ether, washed with brine, dried (MgSO_4), and concentrated under vacuum. The residue was filtered through a plug of silica gel and eluted with hexanes: Et_2O (50:1). The

resulting solution was concentrated under vacuum to give 2-(3-butenyl)-2-(4-bromobenzyl)-[1,3]-dithiane as a pale yellow oil (1.52 g) that was used in the next step without further purification.

A solution of crude 2-(3-butenyl)-2-(4-bromobenzyl)-[1,3]-dithiane (1.52 g) in acetonitrile (15 mL) was added to a mixture of N-chlorosuccinamide (3.21 g, 24.0 mmol) and silver nitrate (4.59 g, 24.0 mmol) in acetonitrile/water (4:1, 100 mL) at room temperature and stirred for 30 min. Saturated aqueous Na₂SO₃, saturated aqueous Na₂CO₃, and brine were added sequentially to the resulting white suspension. The layers were separated and the aqueous layer was extracted (dichloromethane:hexanes = 1:1), dried (MgSO₄), and concentrated under vacuum. The residue was chromatographed (hexanes–Et₂O = 50:1→30:1) to give 1-(4-bromophenyl)-5-hexen-2-one (0.66 g, 44%). TLC: *R_f* = 0.52. ¹H NMR: δ 7.46-7.43 (m, 2 H), 7.08-7.06 (m, 2 H), 5.76 (tdd, *J* = 5.6, 10.2, 17.0 Hz, 1 H), 5.03-4.95 (m, 2 H), 3.65 (s, 2 H), 2.55 (t, *J* = 7.4 Hz, 2 H), 2.34-2.28 (m, 2 H). ¹³C{¹H} NMR: δ 207.0, 137.1, 133.4, 132.1, 131.5, 121.4, 115.8, 49.6, 41.6, 28.0. IR (neat, cm⁻¹): 3077, 2976, 2917, 1897, 1713, 1640, 1590, 1487, 1405, 1358, 1262, 1186, 1107, 1070, 1012, 914, 797. Anal. calcd (found) for C₁₂H₁₃BrO: C, 56.94 (57.06); H, 5.18 (4.99).

1-(3-Trifluoromethylphenyl)-5-hexen-2-one (Table 1, entry 3). 1-(3-Trifluoromethylphenyl)-5-hexen-2-one was obtained in 43% overall yield as a yellow oil from reaction of 3-trifluoro-methylphenyl acetaldehyde and 3-butenyl magnesium bromide followed by oxidation using a procedure similar to that used to synthesize 1,1-diphenyl-hex-5-en-2-one.

For 1-(3-Trifluoromethylphenyl)-5-hexen-2-ol: TLC: *R_f* = 0.32. ¹H NMR: δ 7.50-7.48 (m, 2 H), 7.44-7.41 (m, 2 H), 5.84 (tdd, *J* = 6.4, 10.4, 17.0 Hz, 1 H), 5.08-4.98 (m, 2 H), 3.89-3.83 (m, 1 H), 2.86 (dd, *J* = 4.4, 13.6, 1 H), 2.73 (dd, *J* = 8.2, 13.8 Hz, 1 H), 2.31-2.13 (m, 2 H), 1.72-1.55 (m, 3 H). ¹³C{¹H} NMR: δ 140.0, 138.5, 133.2, 131.1 (q, *J* = 31.7 Hz), 129.2, 126.2 (q, *J* = 3.4 Hz), 124.5 (q, *J* = 270.6 Hz), 123.6 (q, *J* = 3.5 Hz), 115.4, 72.3, 44.0, 36.3, 30.4. IR (neat, cm⁻¹): 3376, 3077, 2924, 1641, 1449, 1331, 1201, 1164, 1125, 1074, 915, 798, 703. Anal. calcd. (found) for C₁₃H₁₅F₃O: H, 6.19 (6.28); C, 63.93 (63.68).

For 1-(3-Trifluoromethylphenyl)-5-hexen-2-one: TLC: *R_f* = 0.54. ¹H NMR: δ 7.53 (br d, *J* = 7.6 Hz, 1 H), 7.47-7.43 (m, 2 H), 7.38 (br d, *J* = 8.0 Hz, 1 H), 5.77 (tdd, *J* = 6.4, 10.4,

16.8 Hz, 1 H), 5.04-4.96 (m, 2 H), 3.77 (s, 2 H), 2.59 (t, $J = 7.4$ Hz, 2 H), 2.36-2.31 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 206.6, 137.0, 135.3, 133.3, 131.3 (q, $J = 32.0$ Hz), 129.4, 126.5 (q, $J = 3.4$ Hz), 124.4 (q, $J = 271.0$ Hz), 124.2 (q, $J = 3.4$ Hz), 115.8, 49.7, 41.8, 27.9. IR (neat, cm^{-1}): 2918, 1718, 1332, 1164, 1124, 1075, 914, 702. Anal. calcd. (found) for $\text{C}_{13}\text{H}_{13}\text{F}_3\text{O}$: H, 5.41 (5.34); C, 64.46 (64.25).

1-(2-oxo-5-hexenyl)naphthalene (Table 1, entry 4). 1-(2-oxo-5-hexenyl)-naphthalene was obtained in 70% overall yield from reaction of naphthalen-1-yl-acetaldehyde and 3-butenyl magnesium bromide, followed by oxidation using a procedure similar to that used to synthesize 1,1-diphenyl-5-hexen-2-one.

For 1-naphthalen-1-yl-5-hexen-2-ol: TLC: $R_f = 0.42$. ^1H NMR: δ 8.05 (m, 1 H), 7.88 (m, 1 H), 7.77 (m, 1 H), 7.56-7.48 (m, 2 H), 7.45-7.37 (m, 2 H), 5.88 (tdd, $J = 6.8, 10.4, 17$ Hz, 1 H), 5.11-4.99 (m, 2 H), 4.02 (m, 1 H), 3.36 (dd, $J = 4.2, 13.8$ Hz, 1 H), 3.08 (dd, $J = 8.6, 13.8$ Hz, 1 H), 2.38-2.18 (m, 2 H), 1.77-1.71 (m, 2 H), 1.58 (s, 1 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 138.7, 134.8, 134.2, 132.4, 129.1, 127.9, 127.6, 126.2, 125.9, 125.7, 124.1, 115.1, 71.7, 41.4, 36.5, 30.4. Anal. calcd. (found) for $\text{C}_{16}\text{H}_{18}\text{O}$: H, 8.02 (7.94); C, 84.91 (84.77).

For 1-(2-oxo-5-hexenyl)naphthalene: TLC: $R_f = 0.53$. ^1H NMR: δ 7.92-7.87 (m, 2 H), 7.82 (d, $J = 8$ Hz, 1 H), 7.56-7.44 (m, 3 H), 7.40 (d, $J = 6.8$ Hz, 1 H), 5.74 (tdd, $J = 6.4, 10.4, 17.2$ Hz, 1 H), 5.00-4.92 (m, 2 H), 4.13 (s, 2 H), 2.53 (t, $J = 7.2$ Hz, 2 H), 2.32-2.27 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 208.1, 137.2, 134.1, 132.4, 131.2, 129.0, 128.5, 128.3, 126.7, 126.1, 125.8, 124.1, 115.5, 48.7, 40.8, 27.9. Anal. calcd. (found) for $\text{C}_{16}\text{H}_{16}\text{O}$: H, 7.19 (7.03); C, 85.68 (85.81).

Benzyl (Z)-3-hexenyl ketone (Table 1, entry 5). Benzyl (Z)-3-hexenyl ketone was obtained in 17% overall yield from reaction of (Z)-4-heptenal with benzyl magnesium chloride, followed by oxidation of employing a procedure similar to that used to synthesize 1,1-diphenyl-hex-5-en-2-one. TLC: $R_f = 0.72$. ^1H NMR: δ 7.35-7.31 (m, 2 H), 7.28-7.24 (m, 1 H), 7.21-7.19 (m, 2 H), 5.40-5.33 (m, 1 H), 5.26-5.19 (m, 1 H), 3.68 (s, 2 H), 2.49 (t, $J = 7.0$ Hz, 2 H), 2.31-2.25 (m, 2 H), 2.06-1.98 (m, 2 H), 0.93 (t, $J = 7.6$ Hz, 3 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 208.2, 134.6, 133.2,

129.7, 129.0, 127.4, 127.3, 50.6, 42.2, 21.9, 20.8, 14.6. IR (neat, cm^{-1}): 3062, 3028, 2963, 2932, 2876, 1958, 1885, 1713, 1603, 1495, 1453, 1359, 1314, 1260, 1070, 1030, 970. Anal. calcd (found) for $\text{C}_{14}\text{H}_{18}\text{O}$: C, 83.12 (83.02); H, 8.97 (8.91).

Benzyl (*E*)-3-hexenyl ketone (Table 1, entry 6). Benzyl (*E*)-3-hexenyl ketone was synthesized in 39% overall yield from reaction of phenylacetaldehyde and (*E*)-3-hexenyl magnesium bromide, followed by oxidation employing a procedure similar to that used to synthesize 1,1-diphenyl-hex-5-en-2-one.

For *trans*-1-phenyl-5-octen-2-ol: TLC: $R_f = 0.42$. ^1H NMR: δ 7.34-7.30 (m, 2 H), 7.26-7.21 (m, 3 H), 5.55-5.48 (m, 1 H), 5.46-5.38 (m, 1 H), 3.87-3.81 (m, 1 H), 2.83 (dd, $J = 4.4$, 13.6 Hz, 1 H), 2.66 (dd, $J = 7.8$, 13.6 Hz, 1 H), 2.24-2.07 (m, 2 H), 2.04-1.97 (m, 2 H), 1.62-1.57 (m, 3 H), 0.97 (t, $J = 7.6$ Hz, 3 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 138.9, 133.1, 129.8, 128.8, 126.8, 72.6, 44.3, 36.8, 29.3, 25.9, 14.2. IR (neat, cm^{-1}): 3386, 3083, 3060, 3025, 2959, 2930, 2870, 2848, 1601, 1494, 1451, 1079, 966. Anal. calcd (found) for $\text{C}_{14}\text{H}_{20}\text{O}$: C, 82.30 (82.18); H, 9.87 (9.69).

For benzyl (*E*)-3-hexenyl ketone. TLC: $R_f = 0.71$. ^1H NMR: δ 7.30 (m, 2 H), 7.26-7.21 (m, 1 H), 7.17 (m, 2 H), 5.45-5.38 (m, 1 H), 5.33-5.25 (m, 1 H), 3.65 (s, 2 H), 2.48 (t, $J = 7.4$ Hz, 2 H), 2.24-2.18 (m, 2 H), 1.97-1.89 (m, 2 H), 0.90 (t, $J = 7.6$ Hz, 3 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 208.2, 134.6, 133.4, 129.7, 129.0, 127.5, 127.3, 50.6, 42.4, 27.0, 25.8, 14.1. IR (neat, cm^{-1}): 3062, 3028, 2961, 2930, 2872, 2848, 1943, 1868, 1713, 1602, 1495, 1454, 1407, 1361, 1188, 1076. Anal. calcd (found) for $\text{C}_{14}\text{H}_{18}\text{O}$: C, 83.12 (82.94); H, 8.97 (9.02).

1,1-Diphenyl-5-hexen-2-one (Table 1, entry 7). Diphenyl acetaldehyde (2.16 g, 11.0 mmol) was added to a solution of 3-butenyl magnesium bromide (0.5 M in THF, 24.2 mL, 12.1 mmol) in THF (30 mL) at room temperature. The solution was refluxed for 12 h and then quenched by addition of saturated aqueous NH_4Cl . The resulting mixture was extracted with ether, and the combined ether extracts were washed with brine, dried (MgSO_4), and concentrated under vacuum. The resulting residue was chromatographed (hexanes–EtOAc = 40:1) to give 1,1-diphenyl-5-hexen-2-ol (0.66 g, 24 %).

A solution of DMSO (0.46 mL, 6.4 mmol) in dichloromethane (2 mL) was added dropwise to a solution of oxalyl chloride (0.27 mL, 3.08 mmol) in dichloromethane (10 mL) at -78°C and stirred for 5 min. 1,1-Diphenyl-5-hexen-2-ol (0.648 g, 2.57 mmol) was added and the resulting solution was stirred for 1.5 h. Triethylamine (1.79 mL, 12.8 mmol) was added, and the reaction mixture was warmed to room temperature, poured into water, and extracted with dichloromethane. The combined dichloromethane extracts were dried (MgSO_4), concentrated and chromatographed (hexanes–EtOAc = 20:1) to give 1,1-diphenyl-5-hexen-2-one as a yellow oil (0.52 g, 81%).

For 1,1-diphenyl-5-hexen-2-ol: TLC: R_f = 0.40. ^1H NMR: δ 7.38–7.36 (m, 2 H), 7.32–7.26 (m, 6 H), 7.23–7.16 (m, 2 H), 5.76 (tdd, J = 6.6, 10.4, 17.2 Hz, 1 H), 5.02–4.91 (m, 2 H), 4.35 (dt, J = 2.9, 8.8 Hz, 1 H), 3.87 (d, J = 8.4 Hz, 2 H), 2.32–2.25 (m, 1 H), 2.20–2.10 (m, 1 H), 1.63–1.54 (m, 2 H), 1.53–1.43 (m, 1 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 142.3, 141.4, 138.4, 128.8, 128.6, 128.2, 126.8, 126.5, 114.8, 73.1, 58.9, 34.2, 30.1. IR (neat, cm^{-1}): 3442, 3061, 3026, 2975, 2916, 2849, 1494, 1450, 1118, 1078, 913, 702. Anal. calcd. (found) for $\text{C}_{13}\text{H}_{20}\text{O}$: H, 7.99 (7.80); C, 85.67 (85.46).

For 1,1-diphenyl-hex-5-en-2-one: TLC: R_f = 0.57. ^1H NMR: δ 7.33–7.29 (m, 4 H), 7.26–7.21 (m, 6 H), 5.75 (tdd, J = 6.6, 10.4, 17.0 Hz, 1 H), 5.12 (s, 1 H), 5.00–4.92 (m, 2 H), 2.64 (t, J = 7.4 Hz, 2 H), 2.35–2.30 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 207.7, 138.3, 136.9, 128.9, 128.7, 127.2, 115.4, 64.2, 41.9, 27.9. IR (neat, cm^{-1}): 3061, 3026, 1717, 1598, 1494, 1451, 1277, 1079, 1032, 1000, 916, 745, 701. Anal. calcd. (found) for $\text{C}_{13}\text{H}_{18}\text{O}$: H, 7.25 (7.22); C, 86.36 (86.33).

1-(4-Fluoro-phenyl)-6-hepten-2-one (Table 1, entry 9) Was synthesized in 56% overall yield from reaction of 4-fluorobenzyl aldehyde and 4-pentenyl magnesium bromide, followed by oxidation employing a procedure similar to that used to synthesize 1,1-diphenyl-5-hexen-2-one.

For 1-(4-fluorophenyl)-6-hepten-2-ol: TLC: R_f = 0.38. ^1H NMR: δ 7.19–7.15 (m, 2 H), 7.03–6.97 (m, 2 H), 5.81 (tdd, J = 6.6, 10.4 17.2 Hz, 1 H), 5.04–4.94 (m, 2 H), 3.82–3.76 (m, 1 H), 7.80 (dd, J = 4.0, 13.6 Hz, 1 H), 2.63 (dd, J = 8.4, 13.6 Hz, 1 H), 2.11–2.06 (m, 2 H), 1.65–1.43 (m, 5 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 162.0 (d, J = 242.8 Hz), 138.9, 134.5, 131.1 (d, J = 7.6 Hz), 115.7

(d, $J = 20.8$ Hz), 115.0, 72.9, 43.5, 36.6, 34.0, 25.3. Anal. calcd. (found) for $C_{13}H_{15}FO$: H, 8.23 (8.32); C, 74.97 (75.10).

For 1-(4-fluorophenyl)-6-hepten-2-one: TLC: $R_f = 0.44$. 1H NMR: δ 7.17-7.12 (m, 2 H), 7.04-6.98 (m, 2 H), 5.72 (tdd, $J = 6.6, 10.4, 17.0$ Hz, 1 H), 4.99-4.93 (m, 1 H), 3.65 (s, 2 H), 2.46 (t, $J = 7.2$ Hz, 2 H), 2.04-1.99 (m, 2 H), 1.66 (quintet, $J = 7.2$ Hz, 2 H). $^{13}C\{^1H\}$ NMR: δ 208.2, 162.3 (d, $J = 243.9$ Hz), 138.1, 131.2 (d, $J = 7.9$ Hz), 130.3, 115.8 (d, $J = 21.2$ Hz), 115.6, 49.4, 41.5, 33.3, 23.0. Anal. calcd (found) for $C_{13}H_{15}FO$: H, 7.33 (7.28); C, 75.70 (75.61).

1-(4-Bromophenyl)-6-hepten-2-one (Table 1, entry 10). 1-(4-Bromophenyl)-6-hepten-2-one was obtained in 33% overall yield as a pale yellow oil from reaction of 2-(4-pentenyl)-[1,3]dithiane and 1-bromomethyl-4-fluorobenzene followed by deprotection employing a procedure similar to that used to synthesize 1-(4-bromophenyl)-5-hexen-2-one. TLC: $R_f = 0.56$. 1H NMR: δ 7.46-7.42 (m, 2 H), 7.08-7.05 (m, 2 H), 5.73 (tdd, $J = 6.8, 10.6, 17.0$ Hz, 1 H), 5.00-4.93 (m, 2 H), 3.63 (s, 2 H), 2.46 (t, $J = 7.4$ Hz, 2 H), 2.04-1.99 (m, 2 H), 1.66 (quintet, $J = 7.2$ Hz, 2 H). $^{13}C\{^1H\}$ NMR: δ 207.8, 138.1, 133.5, 132.1, 131.4, 121.3, 115.6, 49.6, 41.6, 33.3, 23.0. Anal. calcd. (found) for $C_{13}H_{15}BrO$: H, 5.66 (5.65); C, 58.44 (58.59).

1-Naphthalen-2-yl-hept-6-en-2-one (Table 1, entry 11). 1-Naphthalen-2-yl-hept-6-en-2-one was synthesized in 64% overall yield from reaction of naphthalen-2-yl-acetaldehyde with 4-pentenyl magnesium bromide, followed by oxidation employing a procedure similar to that used to synthesize 1,1-diphenyl-5-hexen-2-one.

For 1-naphthalen-2-yl-hept-6-en-2-ol: TLC: $R_f = 0.34$. 1H NMR: δ 7.85-7.81 (m, 3 H), 7.68 (s, 1 H), 7.52-7.45 (m, 2 H), 7.37 (m, 1 H), 5.85 (tdd, $J = 6.8, 10.4, 17.0$ Hz, 1 H), 5.08-4.97 (m, 2 H), 3.95-3.89 (m, 1 H), 3.00 (dd, $J = 4.0, 13.6$ Hz, 1 H), 2.82 (dd, $J = 8.4, 13.4$ Hz, 1 H), 2.14-2.09 (m, 2 H), 1.70-1.48 (m, 5 H). $^{13}C\{^1H\}$ NMR: δ 138.9, 136.3, 133.8, 132.5, 128.4, 128.1, 128.0, 127.9, 127.7, 126.3, 125.7, 114.9, 72.6, 44.5, 36.5, 33.9, 25.3. Anal. calcd. (found) for $C_{17}H_{20}O$: H, 8.39 (8.26); C, 84.96 (84.87).

For 1-Naphthalen-2-yl-hept-6-en-2-one: TLC: $R_f = 0.42$. 1H NMR: δ 7.85-7.80 (m, 3 H), 7.68 (s, 1 H), 7.51-7.45 (m, 2 H), 7.33 (dd, $J = 2.0, 8.4$, 1 H), 5.73 (tdd, $J = 6.8, 10.4, 16.8$ Hz,

1 H), 4.99-4.93 (m, 2 H), 3.85 (s, 2 H), 2.51 (t, $J = 7.4$ Hz, 2 H), 2.04-1.99 (m, 2 H), 1.68 (quintet, $J = 7.2$ Hz, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 208.6, 138.2, 133.9, 132.7, 132.2, 128.7, 128.4, 128.0, 127.9, 127.8, 126.5, 126.1, 115.5, 50.7, 41.5, 33.3, 23.0. IR (neat, cm^{-1}): 3053, 2935, 1712, 1411, 1313, 992, 952, 904, 828, 757. Anal. calcd. (found) for $\text{C}_{17}\text{H}_{18}\text{O}$: H, 7.61 (7.79); C, 85.67 (85.76).

1-Cyclohexyl-5-hexen-2-one (Table 2, entry 5). A solution of cyclohexylacetaldehyde (3.5 g, 28 mmol) in ether (10 mL) was added slowly to a solution of 3-butenyl magnesium bromide (0.5 N in THF, 70 mL, 35 mmol) at 0 °C. The resulting mixture was stirred for 1h, quenched by addition of HCl (1 N, 20 mL), and extracted with ether (2×50 mL). The combined organic fractions were dried (MgSO_4) and distilled (75-78 °C/60 mTorr) to give 1-cyclohexyl-5-hexen-2-ol (3.2 g, 17.6 mmol, 63%) that was 80% pure by GC and used in the next step without further purification. A solution of DMSO (0.8 mL, 11.6 mmol) in CH_2Cl_2 (5 mL) was added to a solution of oxalyl chloride (1.0 mL, 11.5 mmol) in CH_2Cl_2 (15 mL) at -78 °C, stirred for 5 min. A solution of 1-cyclohexyl-5-hexen-2-ol (2.0 g, 11.0 mmol) in CH_2Cl_2 (5 mL) was added slowly to the above solution and the resulting white suspension was stirred for 2 h, quenched with water (15 mL), and extracted with CH_2Cl_2 (2×40 mL). The combined organic fractions were dried (MgSO_4), concentrated, and distilled (45 °C/15-20 mTorr) to give 1-cyclohexyl-5-hexen-2-one (1.5 g, 76%). TLC: $R_f = 0.64$. ^1H NMR: δ 5.78 (tdd, $J = 6.4, 16.8$ Hz, 1 H), 5.01 (qd, $J = 1.6, 17.2$ Hz, 1 H), 4.95 (tdd, $J = 1.2, 1.6, 10.0$ Hz, 1 H), 2.46 (t, $J = 7.2$ Hz, 2 H), 2.27-2.32 (m, 2 H), 2.25 (d, $J = 6.8$ Hz, 2 H), 1.76-1.86 (m, 1 H), 1.60-1.69 (m, 5 H), 1.19-1.31 (m, 2 H), 1.06-1.17 (m, 1 H), 0.85-0.95 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 210.3, 137.4, 115.3, 50.8, 42.6, 34.1, 33.4, 27.9, 26.4, 26.3. IR (neat, cm^{-1}): 2922, 2850, 1713, 1448. Anal. calcd. (found) for $\text{C}_{12}\text{H}_{20}\text{O}$: H, 11.18 (11.16); C, 79.94 (79.82).

1-Chloro-7-octen-4-one (Table 2, entry 9). 1-Chloro-7-octen-4-one was synthesized in 82% yield as from reaction of 4-chlorobutyryl chloride and 3-butenyl magnesium bromide employing a procedure similar to that used to synthesize 1-phenoxy-7-octen-4-one. TLC: $R_f = 0.45$. ^1H NMR: δ 5.79 (tdd, $J = 6.4, 10.4, 13.2$ Hz, 1 H), 5.03 (qd, $J = 1.6, 17.2$ Hz, 1 H), 4.96-5.00 (m, 1 H), 3.57 (t, $J = 6.4$ Hz, 2 H), 2.61 (t, $J = 7.2$ Hz, 2 H), 2.53 (t, $J = 7.2$ Hz, 2 H), 2.30-

2.36 (m, 2 H), 2.01-2.07 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 209.1, 137.1, 115.6, 44.7, 42.1, 39.5, 27.9, 26.4. IR (neat, cm^{-1}): 3078, 2961, 2922, 1716, 1640, 1411. Anal. calcd. (found) for $\text{C}_8\text{H}_{13}\text{ClO}$: H, 8.16 (8.11); C, 59.81 (59.74).

1-Phenoxy-7-octen-4-one (Table 2, entry 10). A solution of 3-butenyl magnesium bromide (0.5 N in THF, 9 mL) was added over 15 min to a suspension of CuI (0.9 g, 4.7 mmol), and 4-phenoxybutyryl chloride (1.0 g, 5.0 mmol) in THF (15 mL) at $-78\text{ }^\circ\text{C}$. The resulting mixture was warmed slowly to room temperature, quenched with HCl (1 N, 20 mL), and extracted with ether (3 $\times$ 30 mL). The combined organic fractions were dried (MgSO_4), concentrated, and chromatographed (hexanes–ether = 20:1 $\rightarrow$ 5:1) to give 1-phenoxy-6-hepten-3-one (0.89 g, 4.1 mmol, 91%). TLC: R_f = 0.40. ^1H NMR (300 MHz): δ 7.23-7.27 (m, 2 H), 6.85-6.84 (m, 3 H), 5.79 (tdd, J = 6.6, 10.2, 17.1 Hz, 1 H), 4.94-5.05 (m, 2 H), 3.96 (t, J = 6.0 Hz, 2 H), 2.62 (t, J = 6.9 Hz, 2 H), 2.53 (t, J = 7.5 Hz, 2 H), 2.32 (1, J = 6.9 Hz, 2 H), 2.05 (quintet, J = 6.9 Hz, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz): δ 208.9, 158.1, 136.4, 128.7, 120.0, 114.6, 113.7, 66.0, 41.2, 38.4, 27.1, 22.7. IR (neat, cm^{-1}): 3073, 3039, 2935, 1713, 1496. Anal. calcd. (found) for $\text{C}_{14}\text{H}_{18}\text{O}_2$: H, 8.31 (8.36); C, 77.03 (76.95).

1-Methoxy-7-octen-4-one (Table 2, entry 11). 1-Methoxy-7-octen-4-one was synthesized in 28% overall yield from reaction of 4-methoxy butyraldehyde and 3-butenyl magnesium bromide, followed by oxidation employing a procedure similar to that used to synthesize 1,1-diphenyl-5-hexen-2-one.

For 1-Methoxy-7-octen-4-ol: TLC: R_f = 0.23. ^1H NMR: δ 5.81 (tdd, J = 6.8, 10.4, 17.2 Hz, 1 H), 5.01 (qd, J = 1.6, 17.2 Hz, 1 H), 4.93 (tdd, J = 1.2, 2.0, 10.0 Hz, 1 H), 3.55-3.61 (m, 1 H), 3.75-3.41 (m, 2 H), 3.32 (s, 3 H), 2.66 (s, 1 H), 2.05-2.23 (m, 2 H), 1.38-1.71 (m, 6 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 138.9, 114.8, 73.2, 71.1, 58.7, 36.8, 35.0, 30.3, 26.4. IR (neat, cm^{-1}): 3411, 2931, 2250, 907. Anal. calcd (found) for $\text{C}_9\text{H}_{18}\text{O}_2$: H, 11.47 (11.29); C, 68.31 (68.32).

For 1-Methoxy-7-octen-4-one: TLC: R_f = 0.25. ^1H NMR: δ 5.78 (tdd, J = 6.4, 10.0, 17.2 Hz, 1 H), 5.00 (qd, J = 1.6, 17.2 Hz, 1 H), 4.95 (tdd, J = 1.2, 2.0, 10.0 Hz, 1 H), 3.35 (t, J = 6.4 Hz, 2 H), 3.28 (s, 3 H), 2.46-2.51 (m, 4 H), 2.28-2.33 (m, 2 H), 1.82 (tt, J = 6.0, 7.2 Hz, 2 H).

$^{13}\text{C}\{^1\text{H}\}$ NMR: δ 210.3, 137.3, 115.3, 71.9, 58.7, 42.0, 39.5, 28.0, 23.9. IR (neat, cm^{-1}): 3078, 2977, 2927, 2828, 1713, 1119. Anal. calcd. (found) for $\text{C}_9\text{H}_{16}\text{O}_2$: H, 10.32(10.18); C, 69.19 (69.31).

Cyclohexanones

2-Phenylcyclohexanone (Table 1, entry 1).⁸ A suspension of benzyl 3-butenyl ketone (87 mg, 0.50 mmol), dichlorodimethylsilane (0.19 mL, 1.50 mmol), $\text{PdCl}_2(\text{MeCN})_2$ (13 mg, 0.05 mmol), and CuCl_2 (67 mg, 0.50 mmol) in 1,4-dioxane (10 mL) was stirred at 70 °C for 18 h. The reaction mixture was cooled to room temperature, filtered through a plug of silica gel, and eluted with ether. The resulting solution was concentrated and the residue was chromatographed (hexanes–ether = 25:1→15:1) to give 2-phenylcyclohexanone (68 mg, 78%) as a pale yellow solid. ^1H NMR: δ 7.33-7.29 (m, 2 H), 7.25-7.21 (m, 1 H), 7.13-7.11 (m, 2 H), 3.59 (dd, J = 5.4, 12.2 Hz, 1 H), 2.54-2.43 (m, 2 H), 2.28-2.22 (m, 1 H), 2.16-2.10 (m, 1 H), 2.04-1.95 (m, 2 H), 1.83-1.77 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 210.6, 139.1, 128.8, 128.7, 127.2, 57.7, 42.5, 35.4, 28.1, 25.6.

A similar procedure was employed for the synthesis of the remaining cyclohexanones found in Table 1, with the exception that SiMe_3Cl was employed in place of SiMe_2Cl_2 .

2-(4-Bromophenyl)cyclohexanone (Table 1, entry 2).⁹ ^1H NMR: δ 7.47-7.44 (m, 2 H), 7.03-7.00 (m, 2 H), 3.57 (dd, J = 5.2, 12.4 Hz, 1 H), 2.56-2.54 (m, 2 H), 2.29-2.23 (m, 1 H), 2.19-2.14 (m, 1 H), 2.02-1.95 (m, 2 H), 1.87-1.78 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 210.0, 138.1, 131.8, 130.6, 121.2, 57.2, 42.5, 35.5, 28.1, 25.7.

2-(3-trifluoromethylphenyl)cyclohexanone. Yellow oil. TLC: R_f = 0.33. ^1H NMR: δ 7.52 (br d, J = 7.6 Hz, 1 H), 7.48 (br t, J = 7.6 Hz, 1 H), 7.40 (s, 1 H), 7.33 (br d, J = 8.0 Hz, 1 H), 3.68 (dd, J = 5.6, 12.4 Hz, 1 H), 2.57-2.44 (m, 2 H), 2.32-2.27 (m, 1 H), 2.22-2.16 (m, 1 H), 2.06-1.96 (m, 2 H), 1.89-1.78 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 209.7, 140.0, 132.5, 130.9 (q, J = 32 Hz), 129.0, 125.7 (q, J = 3.4 Hz), 124.5 (q, J = 208 Hz), 124.8 (q, J = 34 Hz), 57.5, 42.5, 35.6, 28.1, 25.7. IR (neat, cm^{-1}): 2940, 2864, 1711, 1449, 1330, 1163, 1124, 1074, 799, 702. Anal. calcd. (found) for $\text{C}_{13}\text{H}_{13}\text{O}$: C, 64.46 (64.29); H, 5.41 (5.25).

2-Naphthalen-1-yl-cyclohexanone (Table 1, entry 4).¹⁰ ¹H NMR: δ 7.89-7.86 (m, 1 H), 7.80 (d, J = 8.0 Hz, 1 H), 7.75-7.73 (m, 1 H), 7.51-7.45 (m, 3 H), 7.38 (d, J = 7.2 Hz, 1 H), 4.37 (dd, J = 5.2, 12.4 Hz, 1 H), 2.71-2.62 (m, 2 H), 2.46-2.39 (m, 1 H), 2.34-2.24 (m, 2 H), 2.17-2.11 (m, 1 H), 2.01-1.86 (m, 2 H). ¹³C{¹H} NMR: δ 210.2, 135.5, 134.1, 132.0, 129.3, 127.9, 126.1, 125.60, 125.57, 125.54, 123.5, 53.6, 42.9, 34.5, 28.1, 26.2.

***trans*-3-Ethyl-2-phenylcyclohexanone (Table 1, entry 5, 6).** TLC: R_f = 0.47. ¹H NMR: δ 7.36-7.32 (m, 2 H), 7.28-7.24 (m, 1 H), 7.08-7.06 (m, 2 H), 3.31 (d, J = 11.6 Hz, 1 H), 2.57-2.55 (m, 1 H), 2.53-2.51 (m, 1 H), 2.44 (dt, J = 6.0, 13.6 Hz, 1 H), 2.22-2.09 (m, 2 H), 2.03-1.93 (m, 1 H), 1.86-1.74 (m, 1 H), 1.27 (dq, J = 3.2, 7.6, 13.6 Hz, 1 H), 1.08 (m, 1 H), 0.81 (t, J = 7.6 Hz, 3 H). ¹³C{¹H} NMR: δ 210.6, 137.8, 129.7, 128.5, 127.1, 63.6, 46.7, 42.3, 30.4, 27.4, 26.2, 10.9. IR (neat, cm⁻¹): 3087, 3061, 3028, 2961, 2935, 2863, 1947, 1870, 1812, 1713, 1603, 1497, 1454, 1445, 1381, 1358, 1344, 1310, 1280, 1176, 1123. Anal. calcd (found) for C₁₄H₁₈O: C, 83.12 (83.05); H, 8.97 (8.92).

2,2-Diphenylcyclohexanone (Table 1, entry 7).¹¹ ¹H NMR: δ 7.32-7.27 (m, 4 H), 7.25-7.21 (m, 2 H), 7.05-7.02 (m, 4 H), 2.58 (t, J = 5.8 Hz, 2 H), 2.49 (t, J = 6.8 Hz, 2 H), 1.96-1.90 (m, 2 H), 1.85-1.79 (m, 2 H). ¹³C{¹H} NMR: δ 211.3, 142.3, 128.5, 128.3, 126.8, 63.9, 40.7, 39.2, 27.8, 22.1.

***trans*-3-Methyl-2-phenylcyclohexanone (Table 1, entry 8).**¹² ¹H NMR: δ 7.32-7.28 (m, 2 H), 7.24-7.21 (m, 1 H), 7.05-7.03 (m, 2 H), 3.16 (d, J = 11.6 Hz, 1 H), 2.53-2.48 (m, 1 H), 2.41 (dt, J = 5.6, 13.6 Hz, 1 H), 2.15-2.05 (m, 2 H), 2.03-1.96 (m, 1 H), 1.80 (tq, J = 4.0, 13.2 Hz, 1 H), 1.61-1.51 (m, 1 H), 0.79 (d, J = 6.4 Hz, 3 H). ¹³C{¹H} NMR: δ 210.3, 137.8, 129.7, 128.6, 127.1, 65.5, 42.1, 41.0, 34.7, 26.3, 21.6.

***trans*-2-(4-Fluorophenyl)-3-methylcyclohexanone (Table 1, entry 9).** White solid. TLC: R_f = 0.43. ¹H NMR: δ 7.03 (s, 2 H), 7.02 (s, 2 H), 3.20 (d, J = 11.6 Hz, 1 H), 2.55-2.40 (m, 2 H), 2.18-2.00 (m, 3 H), 1.87-1.75 (m, 1 H), 1.64-1.53 (m, 1 H), 0.81 (d, J = 6.4 Hz, 3 H). ¹³C{¹H} NMR: δ 210.1, 162.1 (d, J = 243.2 Hz), 133.4, 131.0 (d, J = 7.9 Hz), 115.4 (d, J = 20.9

Hz), 64.7, 42.1, 41.3, 34.6, 26.3, 21.5. IR (neat, cm^{-1}): 2922, 2863, 1705, 1509, 1220, 1160, 1047, 807. Anal. calcd. (found) for $\text{C}_{13}\text{H}_{15}\text{FO}$: H, 7.33 (7.16); C, 75.70 (75.70).

***trans*-2-(4-Bromophenyl)-3-methylcyclohexanone (Table 1, entry 10).** White solid. mp 91-92 °C. TLC: R_f = 0.42. ^1H NMR: δ 7.47-7.44 (m, 2 H), 6.96-6.93 (m, 2 H), 3.16 (d, J = 11.6 Hz, 1 H), 2.55-2.39 (m, 2 H), 2.18-2.00 (m, 3 H), 1.87-1.75 (m, 1 H), 1.63-1.53 (m, 1 H), 0.81 (d, J = 6.4 Hz, 3 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 209.8, 136.8, 131.7, 131.4, 121.1, 64.9, 42.1, 41.1, 34.6, 26.3, 21.5. IR (neat, cm^{-1}): 2955, 2921, 1707, 1320, 1046, 708. Anal. calcd. (found) for $\text{C}_{13}\text{H}_{15}\text{BrO}$: H, 5.66 (5.58); C, 58.44 (58.40).

***trans*-3-Methyl-2-naphthalen-2-yl-cyclohexanone (Table 1, entry 11).** White solid. mp 87-89 °C. TLC: R_f = 0.42. ^1H NMR: δ 7.83-7.77 (m, 3 H), 7.52 (s, 1 H), 7.47-7.41 (m, 2 H), 7.22 (dd, J = 1.2, 8.4 Hz, 1 H), 3.35 (d, J = 12.0 Hz, 1 H), 2.57-2.42 (m, 2 H), 2.28 (m, 2 H), 2.06-2.01 (m, 1 H), 1.91-1.79 (m, 1 H), 1.66-1.56 (m, 1 H), 0.82 (d, J = 6.4 Hz, 3 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 210.5, 135.5, 133.7, 132.9, 128.7, 128.1, 128.0, 127.4, 126.1, 125.8, 65.6, 42.2, 40.8, 34.6, 26.3, 21.7. IR (neat, cm^{-1}): 3053, 2952, 29.25, 28.66, 1710, 1599, 1508, 1455, 1374, 1318, 1267, 1184, 1121, 1048, 962, 907, 851, 811, 745. Anal. calcd. (found) for $\text{C}_{17}\text{H}_{18}\text{O}$: H, 7.61 (7.85); C, 85.67 (85.44).

2-Hexylcyclohexanone (Table 2, entry 2).¹³ A suspension of $\text{PdCl}_2(\text{MeCN})_2$ (13 mg, 0.05 mmol), CuCl_2 (20 mg, 0.15 mmol), HCl (4 N in dioxane, 13 μL , 0.05 mmol), and 3-butenyl heptyl ketone (91 mg, 0.50 mmol) in dioxane (6 mL) was heated at 70 °C for 12 h in a sealed tube. The resulting mixture was filtered through a short plug of silica gel, eluting with hexanes/ether (1:1, 30 mL). The resulting solution was concentrated and chromatographed (hexanes–ether = 20:1 $\rightarrow$ 10:1) to give 2-hexylcyclohexanone (70 mg, 77%) as a pale yellow oil. ^1H NMR: δ 2.33-2.38 (m, 1 H), 2.20-2.30 (m, 2 H), 1.96-2.11 (m, 2 H), 1.57-1.85 (m, 2 H), 1.30-1.39 (m, 1 H), 1.13-1.29 (m, 9 H), 0.83 (t, J = 6.8 Hz, 3 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 213.8, 50.9, 42.1, 34.0, 31.9, 29.6, 29.6, 28.2, 27.3, 25.0, 22.8, 14.2.

A similar procedure was employed for the synthesis of the remaining cyclohexanones found in Table 2.

2-Butylcyclohexanone (Table 2, entry 3).¹⁴ ¹H NMR: δ 2.34-2.40 (m, 1 H), 2.21-2.31 (m, 2 H), 2.08-2.12 (m, 1 H), 2.00-2.03 (m, 1 H), 1.61-1.87 (m, 4 H), 0.90-1.60 (m, 6 H), 0.88 (t, J = 7.2 Hz, 3 H). ¹³C{¹H} NMR: δ 213.8, 50.9, 42.1, 34.0, 29.6, 29.3, 28.2, 25.0, 23.0, 14.2.

2-Benzylcyclohexanone (Table 2, entry 4).¹⁵ ¹H NMR: δ 7.25 (t, J = 7.2 Hz, 2 H), 7.17 (d, J = 6.8 Hz, 1 H), 7.13 (d, J = 7.6 Hz, 2 H), 3.21 (dd, J = 6.4, 11.2 Hz, 1 H), 2.49-2.57 (m, 1 H), 1.49-1.71 (m, 2 H), 1.33 (dq, J = 3.6, 12.4 Hz, 1 H). ¹³C{¹H} NMR: δ 212.7, 140.6, 129.3, 128.5, 126.2, 52.7, 42.4, 35.7, 33.6, 28.3, 25.3.

2-Cyclohexylcyclohexanone (Table 2, entry 5).¹⁶ ¹H NMR: δ 2.30-2.36 (m, 1 H), 2.18-2.26 (m, 1 H), 2.02-2.08 (m, 1 H), 1.86-1.94 (m, 2 H), 1.47-1.83 (m, 10 H), 1.24 (tq, J = 3.2, 12.8 Hz, 2 H), 1.09 (tq, J = 2.2, 12.4 Hz, 1 H), 0.96 (dq, J = 3.2, 12.0 Hz, 1 H), 0.80-0.90 (m, 1 H). ¹³C{¹H} NMR: δ 213.8, 56.7, 42.0, 36.2, 31.7, 29.5, 28.1, 26.7, 26.6, 24.2.

2-(4-Methoxyphenyl)cyclohexanone (Table 2, entry 6).¹⁷ ¹H NMR: δ 7.05-7.08 (m, 2 H), 6.86-6.90 (m, 2 H), 3.80 (s, 3 H), 3.56 (dd, J = 5.2, 12.4 Hz, 1 H), 2.40-2.55 (m, 2 H), 2.22-2.29 (m, 1 H), 2.10-2.18 (m, 1 H), 1.95-2.05 (m, 1 H), 1.78-1.88 (m, 2 H). ¹³C{¹H} NMR: δ 210.9, 158.6, 131.1, 129.6, 114.0, 56.8, 55.4, 42.4, 35.5, 28.1, 25.6.

Spiro[5.5]undecan-1-one (Table 2, entries 7 and 12).¹⁸ White crystals. mp 36-39 °C. ¹H NMR: δ 2.38 (t, J = 6.8 Hz, 2 H), 1.79-1.90 (m, 4 H), 1.65-1.74 (m, 4 H), 1.45-1.50 (m, 4 H), 1.29-1.42 (m, 4 H). ¹³C{¹H} NMR: δ 217.1, 49.2, 38.8, 33.9, 28.4, 26.5, 22.2, 20.8.

Methyl 3-(2-oxo-cyclohexyl)propionate (Table 2, entry 8).¹⁹ ¹H NMR: δ 3.61 (s, 3 H), 2.21-2.40 (m, 5 H), 1.96-2.01 (m, 3 H), 1.81-1.86 (m, 1 H), 1.58-1.68 (m, 2 H), 1.45-1.54 (m, 1 H), 1.34 (dq, J = 4.0, 12.4 Hz, 1 H). ¹³C{¹H} NMR: δ 212.7, 174.2, 51.6, 49.9, 42.3, 34.3, 31.8, 28.2, 25.2, 25.0.

2-(2-Chloroethyl)cyclohexanone (Table 2, entry 9). Pale yellow oil. ¹H NMR: δ 3.59 (t, J = 6.4 Hz, 2 H), 2.52-2.60 (m, 1 H), 2.21-2.39 (m, 3 H), 2.04-2.14 (m, 2 H), 1.83-1.88 (m, 1 H), 1.48-1.74 (m, 3 H), 1.34 (dq, J = 3.6, 12.8 Hz, 1 H). ¹³C{¹H} NMR: δ 212.4, 47.6, 43.5, 42.4, 34.3, 32.7, 28.2, 25.4. IR (neat, cm⁻¹): 2934, 2861, 1708, 1448. Anal. calcd. (found) for C₈H₁₃ClO: H, 8.16(8.17); C, 59.81(59.62).

2-Phenoxymethylcyclohexanone (Table 2, entry 10). TLC: $R_f = 0.28$. ^1H NMR: δ 7.21-7.27 (m, 2 H), 3.93-4.04 (m, 2 H), 2.55-2.63 (m, 1 H), 2.23-2.41 (m, 3 H), 2.13-2.19 (m, 1 H), 2.03-2.08 (m, 1H), 1.83-1.88 (m, 1 H), 1.58-1.71 (m, 1 H), 1.40 (dq, $J = 4.0, 12.4$ Hz, 1 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 213.0, 159.1, 120.8, 114.7, 65.8, 47.5, 42.5, 34.6, 29.5, 28.3, 25.4. IR (neat, cm^{-1}): 2933, 2860, 1708, 1244. Anal. calcd. (found) for $\text{C}_{14}\text{H}_{18}\text{O}_2$: H, 8.31(8.10); C, 77.03 (76.95).

2-(2-Methoxyethyl)cyclohexanone (Table 2, entry 11). Pale yellow oil. ^1H NMR: δ 3.35-3.42 (m, 2 H), 3.28 (s, 3 H), 2.42-2.50 (m, 1 H), 2.25-2.40 (m, 2 H), 2.01-2.14 (m, 2 H), 1.78-1.87 (m, 1 H), 1.61-1.72 (m, 2 H), 1.34-1.42 (m, 2 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 213.2, 70.7, 58.7, 47.5, 42.4, 34.5, 29.6, 28.3, 25.3. IR (neat, cm^{-1}): 2932, 2861, 1708, 1118. Anal. calcd. (found) for $\text{C}_9\text{H}_{16}\text{O}_2$: H, 10.32 (10.21); C, 69.19 (69.28).

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