Palladium catalyzed cross-coupling of acetates of Baylis-Hillman adducts and potassium organotrifluoroborates

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Supporting Information:

General Considerations: All glassware was dried in an oven at 120 °C and flushed with dry nitrogen. Reactions were carried out under a nitrogen atmosphere. All reagents were purchased from Aldrich Chemical Company and used as received. Products were purified by flash chromatography using silica gel (60 Å, 230 400 mesh) with hexanes and ethyl acetate (Hexanes:EtOAc 20:1) as eluent. Elemental Analyses were performed by Atlantic Microlabs Inc., Norcross, GA. ¹H NMR and ¹³C NMR were recorded in CDCl₃ on a Bruker 250 MHz instrument with chemical shifts reported relative to TMS.

2-(4'-Methylbenzyl)-3-phenylacrylic acid methyl ester:

¹H NMR (CDCl₃) 7.91 (s, 1H), 7.34 7.05 (m, 9H), 3.91 (s, 2H), 3.71 (s, 3H), 2.29 (s, 3H); ¹³C NMR (CDCl₃) 166.5, 140.8, 136.1, 135.9, 135.4, 135.2, 129.1, 128.6, 128.4, 128.1, 127.6, 51.6, 32.6, 20.6.

2-(4'-Chlorophenyl)-2-(4'-methylbenzyl)acrylic acid methyl ester:

M. P. 58 °C, ¹H NMR (CDCl₃) 7.84 (s, 1H), 7.30 7.05 (m, 8H), 3.87 (s, 2H), 3.73 (s, 3H), 2.29 (s, 3H); ¹³C NMR (CDCl₃) 168.3, 139.3, 135.7, 135.6, 134.6, 133.6, 131.3, 130.4, 129.2, 128.8, 127.5, 52.0, 32.6, 20.9. Anal. Calcd. for C₁₈H₁₇ClO₂: C, 71.88; H, 5.70. Found: C, 71.69; H, 5.76.

2-(4'-Methylbenzyl)-3-naphthalene-1-ylacrylic acid methyl ester:

M. P. 94 °C, ¹H NMR (CDCl₃) 8.35 (s, 1H), 8.0 6.9 (m, 11H), 3.77 (s, 2H), 3.74 (s, 3H), 2.23 (s, 3H); ¹³C NMR (CDCl₃) 168.1, 138.7, 136.4, 135.2, 133.4, 133.3, 132.5, 131.4, 128.9, 128.7, 128.4, 127.8, 126.3, 126.0, 125.8, 125.0, 124.4, 51.8, 33.0, 20.8. Anal. Calcd. for C₂₂H₂₀O₂: C, 83.51; H, 6.37. Found: C, 83.26; H, 6.33.

2-(4'-Methylbenzyl)-3-octylacrylic acid methyl ester:

¹H NMR (CDCl₃) 7.07 (s, 4H), 6.92 (t, *J* = 6 Hz, 1H), 3.67 (s, 3H), 3.63 (s, 2H), 2.28 (s, 3H); 2.21 (m, 1H); 1.60 1.20 (m, 12H); 0.90 (t, *J* = 8 Hz, 3H). ¹³C NMR (CDCl₃) 168.1, 144.2, 136.6, 135.2, 130.8, 128.8, 128.0, 51.6, 31.8, 29.3, 29.1, 28.8, 28.6, 22.6, 20.9, 14.0. Anal. Calcd. for C₂₁H₃₂O₂: C, 79.70; H, 10.19. Found: C, 79.37; H, 10.09.

2-(4'-Methylbenzyl)-3-phenylacrylonitrile:

¹H NMR (CDCl₃) 7.68 (m, 2H), 7.36 (m, 3H), 7.15, (s, 4H), 6.91 (s, 1H); 3.62 (s, 2H), 2.31 (s, 3H); ¹³C NMR (CDCl₃) 143.7, 136.8, 133.8, 133.3, 129.9, 129.5, 128.6, 116.6, 110.9, 41.7, 21.0.

3-(4'-Chlorophenyl)-2-(4'-methylbenzyl)acrylonitrile:

M.P. 41 42 °C, ¹H NMR (CDCl₃) 7.58 (d, *J* = 9 Hz, 2H), 7.27 (d, *J* = 9 Hz, 2H), 7.12 (s, 4H), 6.83 (s, 1H); 3.58 (s, 2H), 2.30 (s, 3H); ¹³C NMR (CDCl₃) 142.0, 136.8, 135.6, 132.9, 131.8, 129.6, 129.4, 128.7, 128.5, 116.2, 111.5, 41.4, 20.8. Anal. Calcd. for C₁₇H₁₄ClN: C, 76.26; H, 5.27; N, 5.23. Found: C, 76.32; H, 5.35; N, 5.17.

3-(4'-Methoxyphenyl)-2-(4'-methylbenzyl)acrylonitrile:

¹H NMR (CDCl₃) 7.66 (d, *J* = 9 Hz, 2H), 7.10 (s, 4H), 6.63 (d, *J* = 9 Hz, 2H), 6.62 (s, 1H), 3.71 (s, 3H), 3.53 (s, 2H), 2.28 (s, 3H); ¹³C NMR (CDCl₃) 160.6, 143.1, 136.4, 133.5, 130.1, 129.2,

128.4, 126.0, 119.0, 113.8, 107.5, 54.9, 41.4, 20.8. Anal. Calcd. for $C_{18}H_{17}NO$; C, 82.10; H, 6.51; N, 5.32. Found: C, 82.20; H, 6.53; N, 5.40.

2-Benzyl-3-phenylacrylic acid methyl ester:

1H NMR ($CDCl_3$) 7.94 (s, 1H), 7.34–7.15 (m, 10H), 3.94 (s, 2H), 3.70 (s, 3H); ^{13}C NMR ($CDCl_3$) 166.4, 140.6, 139.2, 130.5, 129.0, 128.6, 128.4, 128.2, 127.9, 126.4, 125.9, 51.7, 33.0.

2-(4'-Methoxybenzyl)-3-phenylacrylic acid methyl ester:

1H NMR ($CDCl_3$) 7.90 (s, 1H), 7.34–6.80 (m, 9H), 3.88 (s, 2H), 3.78 (s, 3H), 3.74 (s, 3H); ^{13}C NMR ($CDCl_3$) 168.6, 157.9, 140.6, 135.3, 131.2, 131.0, 129.2, 128.7, 128.6, 128.4, 113.9, 55.1, 52.0, 32.2. Anal. Calcd. for $C_{18}H_{18}O_3$; C, 76.57; H, 6.43. Found: C, 76.48; H, 6.48.

3-Phenyl-2-(3-thiophenyl)methylacrylic acid methyl ester:

1H NMR ($CDCl_3$) 7.86 (s, 1H), 7.34–6.85 (m, 8H), 3.88 (s, 2H), 3.73 (s, 3H); ^{13}C NMR ($CDCl_3$) 168.3, 140.2, 139.5, 135.0, 130.6, 129.1, 128.6, 128.4, 127.8, 125.5, 120.5, 51.9, 28.3. Anal. Calcd. for $C_{15}H_{14}O_2S$; C, 69.74; H, 5.46. Found: C, 69.69; H, 5.40.

2-(2'-Methylbenzyl)-3-phenylacrylic acid methyl ester:

1H NMR ($CDCl_3$) 7.99 (s, 1H), 7.24–7.05 (m, 9H), 3.83 (s, 2H), 3.70 (s, 3H), 2.26 (s, 3H); ^{13}C NMR ($CDCl_3$) 168.4, 141.0, 137.2, 135.9, 135.1, 130.2, 129.9, 129.0, 128.5, 128.3, 126.4, 126.3, 125.9, 51.8, 30.7, 19.5. Anal. Calcd. for $C_{18}H_{18}O_2$; C, 81.17; H, 6.81. Found: C, 81.17; H, 6.80.

2-(2',6'-Dimethylbenzyl)-3-phenylacrylic acid methyl ester:

M.P. 68 °C, 1H NMR ($CDCl_3$) 7.63 (s, 1H), 7.36–6.90 (m, 8H), 3.91 (s, 2H), 3.54 (s, 3H), 2.16 (s, 6H); ^{13}C NMR ($CDCl_3$) 168.2, 138.5, 137.1, 135.6, 135.5, 132.1, 129.0, 128.2, 128.0, 127.9, 127.6, 125.8, 51.4, 28.6, 20.4. Anal. Calcd. for $C_{19}H_{20}O_2$; C, 81.40; H, 7.19. Found: C, 81.20; H, 7.26.

2-(2',6'-Difluorobenzyl)-3-phenylacrylic acid methyl ester:

1H NMR ($CDCl_3$) 7.82 (s, 1H), 7.37–6.75 (m, 8H), 3.91 (s, 2H), 3.73 (s, 3H); ^{13}C NMR ($CDCl_3$) 167.0, 163.5, 159.6, 140.5, 135.4, 129.6, 128.9, 128.3, 127.6, 115.2, 111.2, 110.8, 51.8, 21.5. Anal. Calcd. for $C_{17}H_{14}F_2O_2$; C, 70.83; H, 4.89. Found: C, 70.80; H, 4.87.

2-Benzylidene-5-phenylpent-4E-enoic acid methyl ester:

1H NMR ($CDCl_3$) 7.85 (s, 1H), 7.35–7.20 (m, 10H), 6.40 (d, J = 16 Hz, 1H), 6.38–6.20 (m, 1H), 3.76 (s, 3H), 3.57 (d, J = 12 Hz, 1H), 3.45 (d, J = 4 Hz, 1H); ^{13}C NMR ($CDCl_3$) 168.12, 140.3, 137.1, 135.1, 134.2, 132.5, 132.3, 128.5, 128.3, 127.9, 127.6, 127.1, 126.1, 126.0, 125.9, 51.8, 30.7. Anal. Calcd. for $C_{19}H_{18}O_2$; C, 81.99; H, 6.52. Found: C, 81.96; H, 6.56.

2-Benzylidene-5-phenylpent-4Z-enoic acid methyl ester:

1H NMR ($CDCl_3$) 7.72 (s, 1H), 7.31–7.10 (m, 10H), 6.55 (dd, J = 12, 1.5 Hz, 1H), 5.75 (m, 1H), 3.79 (s, 3H), 3.58 (m, 2H); ^{13}C NMR ($CDCl_3$) 168.4, 140.0, 137.1, 134.9, 131.0, 129.7, 129.4, 128.8, 128.4, 128.2, 128.0, 126.7, 52.0, 27.5. Anal. Calcd. for $C_{19}H_{18}O_2$; C, 81.99; H, 6.52. Found: C, 81.85; H, 6.53.

2-Benzylidene-4,5-diphenylpent-4-enoic acid methyl ester:

1H NMR ($CDCl_3$) 7.95 (s, 1H), 7.43–6.90 (m, 15H), 6.37 (s, 1H), 3.77 (s, 3H), 3.67 (s, 1H); ^{13}C NMR ($CDCl_3$) 168.4, 141.6, 141.4, 140.3, 136.9, 135.2, 129.6, 129.0, 128.7, 128.5, 128.3, 128.2, 127.7, 127.0, 126.2, 126.0, 52.0, 37.9. Anal. Calcd. for $C_{25}H_{22}O_2$; C, 84.72; H, 6.26. Found: C, 84.52; H, 6.24.