Phosphine Mediated [4+2] annulation of Bis(enones): A Lewis-Base Catalyzed “Mock Diels-Alder” Reaction

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2a: mp 89-90°C (ether); ^1H NMR (300 MHz, C_6D_6): 7.41-7.03 (m, 5H), 3.38 (dd, J = 4.5, 13.5 Hz, 1H), 2.40 (dd, J = 8.0, 14.0 Hz, 1H), 2.19-1.98 (m, 3H), 1.69 (dt, J = 6.0, 13.5 Hz, 1H), 1.55 (t, J = 13.5 Hz, 1H), 1.45-0.98 (m, 4H), 0.71 (m, 1H); ^{13}C NMR (75 MHz, C_6D_6): 207.36, 140.80, 129.54, 128.51, 126.20, 52.67, 51.13, 48.12, 44.87, 41.11, 35.55, 32.52, 31.92, 25.89. HRMS calcd. for $\text{C}_{17}\text{H}_{20}\text{O}_2$ ($\text{M}+\text{Na}^+$): 279.1361, found: 279.1353.

2c: mp 88-90°C (methanol); ^1H NMR (300 MHz, C_6D_6): 7.15-7.04 (m, 5H), 3.33 (dd, J = 5.0, 13.5 Hz, 1H), 2.38 (dd, J = 8.0, 14.0 Hz, 1H), 2.15-2.01 (m, 4H), 1.91-1.80 (m, 2H), 1.55-1.07 (m, 7H), 0.86-0.65 (m, 2H); ^{13}C NMR (75 MHz, C_6D_6): 207.67, 140.55, 129.49, 128.52, 126.24, 54.74, 50.67, 48.74, 42.29, 41.56, 37.40, 35.37, 35.12, 27.39, 24.20. HRMS calcd. for $\text{C}_{18}\text{H}_{22}\text{O}_2$ ($\text{M}+\text{Na}^+$): 293.1517, found: 293.1515.

2d: mp 111-113°C (ether); ^1H NMR (300 MHz, C_6D_6): 7.17-7.04 (m, 5H), 3.30 (dd, J = 6.0, 14.0 Hz, 1H), 2.58 (m, 1H), 2.42 (dd, J = 7.5, 14.0 Hz, 1H), 2.27 (m, 1H), 2.16-2.09 (m, 2H), 1.68 (dt, J = 6.0, 14.0 Hz, 1H), 1.55-1.34 (4H, m), 1.14-0.98 (m, 2H), 0.82 (d, J = 7.0 Hz, 3H), 0.89-0.68 (m, 1H); ^{13}C NMR (75 MHz, C_6D_6): 207.67, 206.59, 141.19, 129.26, 128.57, 126.20, 57.62, 57.18, 48.50, 41.18, 37.89, 33.80, 32.58, 32.41, 24.72, 10.40. HRMS calcd. for $\text{C}_{18}\text{H}_{22}\text{O}_2$ ($\text{M}+\text{Na}^+$): 293.1517, found: 293.1518.

2f: mp 70-80°C (ether); ^1H NMR (300 MHz, C_6D_6): 7.18-7.02 (m, 5H), 3.35 (dd, J = 4.5, 14.0 Hz, 1H), 2.40 (dd, J = 8.0, 14.0 Hz, 1H), 2.18-2.05 (m, 3H), 1.74 (dt, J = 6.0, 13.5

Hz, 1H), 1.66-1.23 (m, 5H), 1.08 (m, 1H), 0.91 (d, J = 6.5 Hz, 1H), 0.88 (m, 1H), 0.69 (1H, m); ^{13}C NMR (75 MHz, C_6D_6): 209.41, 207.65, 140.93, 129.53, 128.51, 126.18, 52.82, 51.41, 50.96, 49.80, 41.05, 35.94, 32.59, 30.34, 25.89, 11.80. HRMS calcd. for $\text{C}_{18}\text{H}_{22}\text{O}_2$ ($\text{M}+\text{Na}^+$): 293.1517, found: 293.1516.

2g: mp 105-106°C (ether); ^1H NMR (300 MHz, C_6D_6): 7.18-7.03 (m, 5H), 3.39 (dd, J = 4.5, 14.0 Hz, 1H), 2.37 (dd, J = 8.0, 14.0 Hz, 1H), 2.11-1.88 (m, 3H), 1.79-1.29 (m, 7H), 1.01-0.92 (m, 2H), 0.44 (s, 3H); ^{13}C NMR (75 MHz, C_6D_6): 207.77, 207.68, 140.81, 129.49, 128.52, 126.21, 55.35, 51.40, 43.05, 40.86, 39.46, 35.50, 28.42, 22.26, 17.73. HRMS calcd. for $\text{C}_{18}\text{H}_{22}\text{O}_2$ ($\text{M}+\text{Na}^+$): 293.1517, found: 293.1515.

3a: ^1H NMR (300 MHz, C_6D_6): 7.17-7.01 (m, 5H), 5.88 (s, 1H), 4.73 (s, 1H), 2.91 (m, 1H), 2.76 (t, J = 7.5 Hz, 2H), 2.22-2.01 (m, 5H), 1.84 (dd, J = 7.5, 16.5 Hz, 1H), 1.50-1.17 (m, 3H), 1.05 (m, 1H); ^{13}C NMR (75 MHz, C_6D_6): 206.56, 199.05, 141.59, 141.30, 132.83, 128.59, 128.54, 126.25, 46.27, 44.76, 40.21, 31.18, 29.90, 28.06, 19.21, 12.96. HRMS calcd. For $\text{C}_{17}\text{H}_{20}\text{O}_2$ ($\text{M}+\text{Na}^+$): 279.1361, found: 279.1361.

E-(**3d**): ^1H NMR (300 MHz, C_6D_6): 7.15-6.99 (m, 5H), 6.58 (q, J = 7.0 Hz, 1H), 3.42 (m, 1H), 2.74 (t, J = 8.5 Hz, 2H), 2.39-1.90 (m, 5H), 1.82 (dd, J = 5.5, 17.0 Hz, 1H), 1.36 (d, J = 7.0 Hz, 3H), 1.43-1.22 (m, 4H); ^{13}C NMR (75 MHz, C_6D_6): 206.21, 199.61, 149.17, 141.38, 128.62, 128.59, 126.28, 118.56, 46.71, 44.59, 40.33, 36.91, 29.79, 29.64, 22.09, 21.15. HRMS calcd. for $\text{C}_{18}\text{H}_{22}\text{O}_2$ ($\text{M}+\text{Na}^+$): 293.1517, found: 293.1518.

Z-(**3d**): ^1H NMR (300 MHz, C_6D_6): 7.15-7.01 (m, 5H), 5.34 (q, J = 7.0 Hz, 1H), 2.97 (m, 1H), 2.76 (t, J = 7.0 Hz, 2H), 2.28-2.06 (m, 3H), 2.05-1.92 (m, 2H), 1.81 (d, J = 7.0 Hz, 3H), 1.52-1.16 (m, 4H); HRMS calcd. for $\text{C}_{18}\text{H}_{22}\text{O}_2$ ($\text{M}+\text{Na}^+$): 293.1517, found: 293.1510.