Supporting Information

Gold-Catalyzed Cyclizations of Alkyne-Propargylic Acetates to 2,3-Bisalkyledenedecycloakanones and Their Related Benzene Derivatives

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Characterization of new compounds 2a-d, 4a-f, and 5h-i are therein.

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1. General experimental procedure:

To a mixture of AuCl(PPh$_3$) (80.0 mg, 0.16 mmol) and AgSbF$_6$ (54.0 mg, 0.16 mmol) in dry acetonitrile (0.5 mL) was added a solution of yne-propargylic acetate 1 (100.0 mg, 3.2 mmol) in dry dichloromethane (0.5 mL) at 0°C under argon atmosphere. The resulting mixture was stirred for 5–10h at 25–80 °C. Upon completion of the reaction, the solvent was removed under vacuum and the crude product was subjected for flash column chromatography(SiO$_2$, n-hexane/EtOAc = 10:1) to afford the pure products.
2. Spectroscopic data of compounds

**Spectroscopic data of compound 2a:** $^1$H NMR (400 MHz, CDCl$_3$) $\delta$

7.41 (s, 1H), 7.11 (s, 1H), 2.90 (t, $J = 7.2$ Hz, 4H), 2.85 (t, $J = 7.8$ Hz, 2H), 2.74 (t, $J = 7.6$ Hz, 2H), 2.09 (quint, $J = 7.2$ Hz, 2H), 1.68 (quint, $J = 7.2$ Hz, 2H), 1.52 (m, 2H), 1.38 (m, 4H), 0.94 (t, $J = 7.2$ Hz, 3H), 0.92 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 205.79, 147.74, 141.63, 140.93, 137.20, 127.06, 124.18, 42.11, 34.67, 33.10, 32.61, 26.91, 25.62, 23.05, 22.71, 14.23, 14.18; FTIR (NaCl, cm$^{-1}$) 1684, 1559, 1541, 1507; HRMS (ES) calcd for C$_{18}$H$_{26}$O Na$^+$ (M+Na$^+$) 281.1876, found 281.1883.

**Spectroscopic data of compound 2b:** $^1$H NMR (400 MHz, CDCl$_3$) $\delta$

7.27 (s, 1H), 6.93 (s, 1H), 2.85 (t, $J = 7.2$ Hz, 2H), 2.80-2.70 (m, 6H), 1.82-1.78 (m, 4H), 1.72-1.64 (m, 2H), 1.56-1.48 (m, 2H), 0.94 (t, $J = 7.2$ Hz, 3H), 0.91 (t, $J = 7.6$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 205.17, 140.70, 139.90, 136.16, 134.46, 131.89, 129.49, 41.78, 34.52, 29.55, 29.35, 29.19, 29.93, 23.41, 23.23, 23.10, 22.71, 14.25, 14.21; FTIR (NaCl, cm$^{-1}$) 1684, 1559, 1458; HRMS (ES) calcd for C$_{19}$H$_{28}$O Na$^+$ (M+Na$^+$) 295.2132, found 295.2033.

**Spectroscopic data of compound 2c:** $^1$H NMR (400 MHz, CDCl$_3$) $\delta$

7.30 (s, 1H), 6.97 (s, 1H), 2.86 (t, $J = 7.2$ Hz, 2H), 2.82-2.71 (m, 5H), 1.89-1.81 (m, 2H), 1.72-1.61 (m, 6H), 1.57-1.49 (m, 3H), 1.44-1.34 (m, 5H), 0.95 (t, $J = 7.2$ Hz, 3H), 0.92 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 205.23, 146.89, 140.71, 140.64, 136.28, 132.11, 129.27, 41.82, 36.83, 36.55, 34.46, 33.50, 32.85, 29.93, 28.52, 28.33, 26.85, 23.12, 22.71, 14.24, 14.20; FTIR (NaCl, cm$^{-1}$) 1683, 1609, 1557; HRMS (ES) calcd for C$_{20}$H$_{30}$ONa$^+$ (M+Na$^+$) 309.2189; found 309.2194.

**Spectroscopic data of compound 2d:** $^1$H NMR (400 MHz, CDCl$_3$) $\delta$

7.82-7.79 (m, 2H), 7.56 (m, 1H), 7.46-7.42 (m, 2H), 7.03 (s, 1H), 7.01 (s, 1H), 2.81 (dd, $J = 7.0$, 2.0 Hz, 2H), 2.74 (dd, $J = 7.2$, 2.0 Hz, 2H), 2.59 (t, $J = 8.0$ Hz, 2H), 1.84 (m, 2H), 1.70-1.60 (m, 4H), 1.53-1.45 (m, 2H), 1.29-1.22 (m, 2H), 0.82 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 199.01, 145.89, 140.12, 139.72, 138.38, 135.86, 132.78, 130.93, 130.11, 129.40, 128.27, 36.67, 36.10, 34.02, 32.61, 28.24, 28.15, 22.61, 13.82; FTIR (NaCl, cm$^{-1}$) 1663, 1596; HRMS (ES) calcd for C$_{22}$H$_{26}$O Na$^+$ (M+Na$^+$) 329.1876, found 329.1886.

**Spectroscopic data of compound 4a:** $^1$H NMR (400 MHz, CDCl$_3$) $\delta$

5.99 (s, 1H), 5.81-5.70 (m, 1H), 5.03-4.98 (m, 3H), 4.81 (q, $J = 1.2$Hz,
Spectroscopic data of compound 4b: $^1$H NMR (400 MHz, CDCl$_3$) δ 5.21 (s, 1H), 4.82 (s, 1H), 4.18 (q, $J = 7.2$ Hz, 4H), 3.00 (s, 1H), 2.83 (s, 1H), 2.07 (s, 2H), 1.23 (t, $J = 7.2$ Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 197.06, 170.30, 146.64, 139.74, 135.01, 117.80, 62.11, 55.10, 46.53, 39.94, 24.34, 22.90, 14.25; FTIR (NaCl, cm$^{-1}$) 1750, 1686; HRMS (ES) calcd for C$_{16}$H$_{22}$O$_5$Na$^+$ (M+Na$^+$) 317.1365, found 317.1361.

Spectroscopic data of compound 4c: $^1$H NMR (400 MHz, CDCl$_3$) δ 6.73 (t, $J = 7.6$ Hz, 1H), 5.23 (d, $J = 1.2$ Hz, 1H), 4.95 (s, 1H), 4.19 (q, $J = 7.2$ Hz, 4H), 2.96 (s, 2H), 2.85 (s, 1H), 2.35 (m, 2H), 1.23 (t, $J = 7.2$ Hz, 6H), 1.08 (t, $J = 6.8$ Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 195.67, 169.92, 144.16, 137.16, 136.13, 117.39, 61.91, 54.47, 44.36, 39.70, 22.89, 14.00, 13.73; FTIR (NaCl, cm$^{-1}$) 1740, 1692; HRMS (ES) calcd for C$_{16}$H$_{22}$O$_5$Na$^+$ (M+Na$^+$) 317.1365, found 317.1369.

Spectroscopic data of compound 4d: $^1$H NMR (400 MHz, CDCl$_3$) δ 6.50 (t, $J = 7.6$ Hz, 1H), 5.18 (d, $J = 1.2$ Hz, 1H), 4.97 (s, 1H), 4.19 (q, $J = 7.2$ Hz, 4H), 2.97 (s, 2H), 2.86 (m, 1H), 2.85 (s, 2H), 1.23 (t, $J = 7.2$ Hz, 6H), 1.04 (d, $J = 6.8$ Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 196.32, 170.16, 148.63, 137.64, 135.13, 116.98, 62.16, 54.72, 44.68, 40.02, 27.93, 22.73, 14.25 FTIR (NaCl, cm$^{-1}$) 1755, 1685; HRMS (ES) calcd for C$_{17}$H$_{24}$O$_5$Na$^+$ (M+Na$^+$) 331.1521, found 331.1527.

Spectroscopic data of compound 4e: $^1$H NMR (400 MHz, CDCl$_3$) δ 7.56-7.54 (m, 2H), 7.45 (s, 1H), 7.32-7.27 (m, 3H), 5.14 (s, 1H), 5.08 (d, $J = 1.2$ Hz, 1H), 4.20 (q, $J=7.6$, 4H), 3.12 (s, 1H), 2.95 (s, 1H), 1.23 (t, $J = 7.2$ Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 195.46, 169.87, 136.81, 136.36, 134.82, 129.59, 128.85, 128.42, 119.00, 62.02, 54.38, 44.71, 39.34, 13.99; FTIR (NaCl, cm$^{-1}$) 1745, 1680, 1591; HRMS (ES) calcd for C$_{20}$H$_{22}$O$_5$Na$^+$ (M+Na$^+$) 365.1365, found 365.1361.

Spectroscopic data of compound 4f: $^1$H NMR (400 MHz, CDCl$_3$) δ 6.74 (t, $J=7.6$, 1H), 5.83-5.73 (m, 1H), 5.23 (d, $J = 1.2$ Hz, 1H), 5.02-4.92 (m, 3H), 4.19 (q, $J = 7.2$ Hz, 4H), 4.18 (q, $J = 7.2$ Hz, 4H), 2.96 (s, 1H), 2.85 (s, 1H),
2.34 (q, J = 7.2 Hz, 2H), 2.07-2.01 (m, 2H), 1.52-1.37 (m, 4H), 1.23 (t, J = 7.6 Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 195.60, 169.90, 142.64, 138.49, 137.17, 117.46, 114.63, 61.91, 54.46, 44.35, 39.70, 33.43, 29.67, 29.28, 28.61, 28.54, 13.99; FTIR (NaCl, cm$^{-1}$) 1748, 1679; HRMS (ES) calcd for C$_{20}$H$_{28}$O$_5$Na$^+$ (M+Na$^+$) 371.1834, found 371.1840.

Spectroscopic data of compound 5h: $^1$H NMR (400 MHz, CDCl$_3$) $\delta$

7.46-7.41 (m, 3H), 7.39-7.35 (m, 2H), 7.11 (s, 1H), 4.17 (q, J = 6.8 Hz, 4H), 3.53 (s, 2H), 3.38 (t, J = 7.6 Hz, 2H), 3.10 (s, 1H), 2.04 (m, 2H), 1.19 (t, J = 7.2 Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 195.52, 170.68, 148.50, 143.87, 140.92, 138.75, 129.05, 128.39, 128.27, 127.22, 62.63, 55.97, 45.27, 36.13, 35.19, 32.64, 26.20, 14.62; FTIR (NaCl, cm$^{-1}$) 1750, 1729, 1682, 1593; HRMS (ES) calcd for C$_{25}$H$_{26}$O$_5$Na$^+$ (M+Na$^+$) 429.1678, found 429.1680.

Spectroscopic data of compound 5i: $^1$H NMR (400 MHz, CDCl$_3$) $\delta$

7.43 (m, 4H), 7.40-7.34 (m, 1H), 7.10 (s, 1H), 3.39 (t, J = 6.8 Hz, 2H), 2.99 (t, J = 6 Hz, 2H), 2.89 (t, J = 7.6 Hz, 2H), 2.66 (t, J = 6.4 Hz, 2H), 2.13 (q, J = 6.4 Hz, 2H), 2.04 (q, J = 7.6 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 199.53, 147.64, 143.34, 142.51, 141.80, 140.51, 128.36, 128.29, 127.51, 127.32, 40.46, 34.70, 31.89, 30.22, 25.64, 23.36; FTIR (NaCl, cm$^{-1}$) 1678, 1593; HRMS (ES) calcd for C$_{19}$H$_{18}$ONa$^+$ (M+Na$^+$) 285.1255, found 285.1260.
3. $^1$H and $^{13}$C NMR Spectra of Compounds 2a-d, 4a-f, and 5h-i
4b
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NOE of 4c

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