Supporting Information

Highly Efficient [2+2] Cyclizations of Allenynes under Microwave Irradiation: Construction of Fused Bicyclic Compounds

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General pathways leading to allenyne substrates, cycloaddition procedures, and characterization data for the substrates **1a-j** and the products **2a-j** are therein.

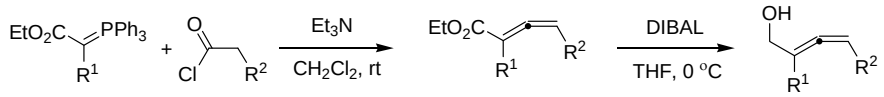
General:

Solvents were reagent grade. All chemicals were purchased from Aldrich Chemical Co. Reactions were normally carried out under argon atmosphere in flame-dried new glassware. Samsung Domestic Microwave oven, Mono mode 300 was used. All products were purified by flash column chromatography using silica gel 60 (70-230 mesh, Merck) and/or by a Young-Lin M930 HPLC employing a Nova-Pak silica preparative column and a UV detector. The purified products were identified with ^{1}H and ^{13}C NMR spectral data obtained from a Varian Mercury 400 MHz NMR spectrometer using $CHCl_3$ or tetramethylsilane as an internal standard.

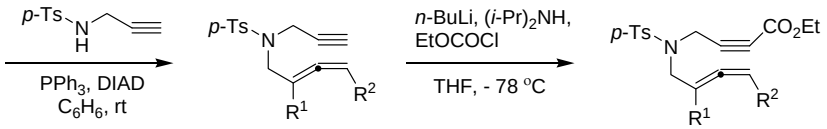
Preparation of Used Various Allenynes 1a-j:

The allene compounds used in this study were synthesized by the following methods and fully derived through the well-known procedure such as an alkylation, Mitsunobu-type reaction, reduction, and so on.

1) Syntheses of Allenynes 1a-c[1]: (by Wittig-type reaction[2])

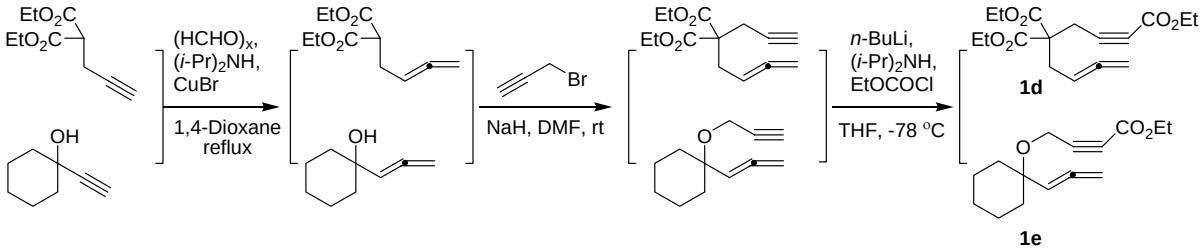


(R^1 = H or Me) (R^2 = H or Me)

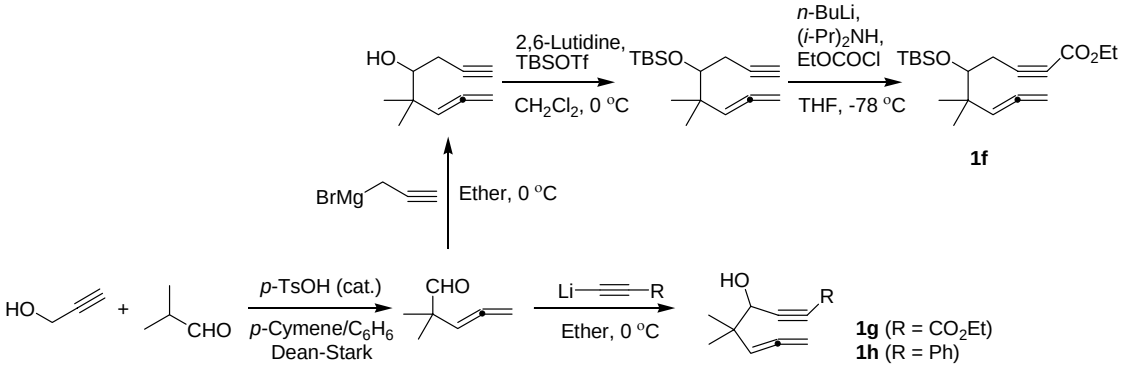


1a (R^1 = H, R^2 = H)
1b (R^1 = Me, R^2 = H)
1c (R^1 = H, R^2 = Me)

2) Synthesis of Allenynes 1d and 1e[1]: (by Crabbé reaction[3])



3) Syntheses of Allenynes 1f-h[1]: (by condensation of propargyl alcohol and isobutyraldehyde[4])



1g (R = CO_2Et)
1h (R = Ph)

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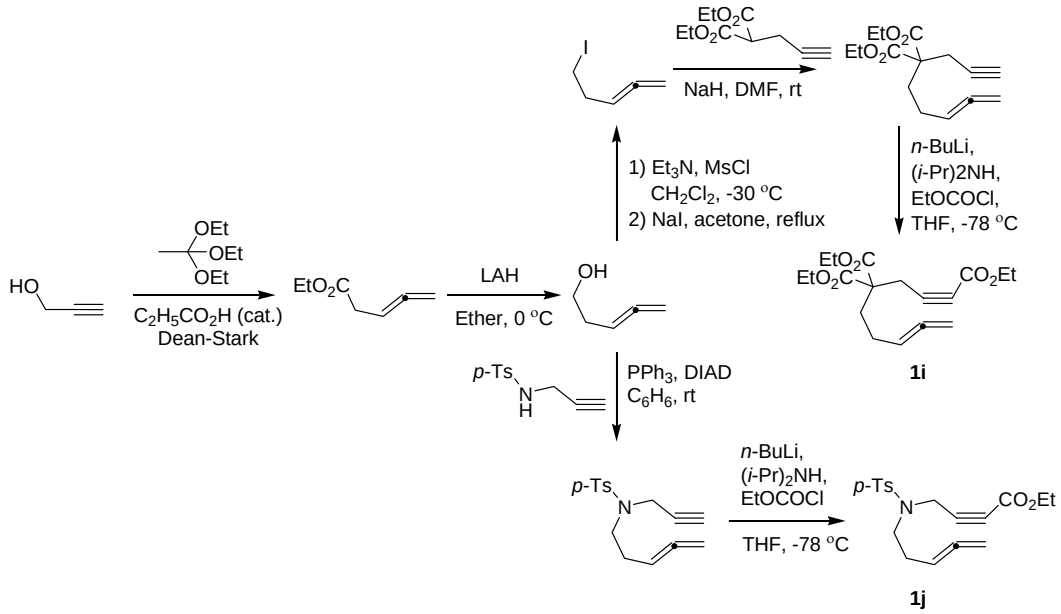
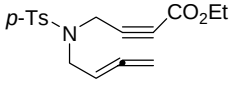
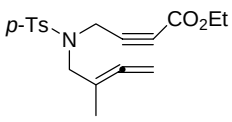
1. C. H. Oh, D. I. Park, S. H. Jung, V. R. Reddy, A. K. Gupta, and Y. M. Kim *Synlett* 2005, 2092.
2. R. W. Lang and H.–J. Hansen, *Org. Synth.* 1984, 202.
3. (a) P. Crabbé, H. Fillion, D. André and J. –L. Luche, *J. Chem. Soc., Chem. Commun.* 1979, 859. (b) S. Searles, Y. Li, B. Nassim, M. -T Robert Lopes, P. T.Tran and P. Crabbé, *J. Chem. Soc., Perkin Trans. 1* 1984, 747.
4. Using the condensation of allyl alcohol and isobutylaldehyde, see: R. G. Salomon, S. Ghosh, *Org. Synth. Coll.*; vol. VII, p177.

4) Syntheses of Allenynes 1i and 1j[1]: (by Orthoester-Claisen Rearrangement[5])





Spectroscopic data of compound 1a: FT-IR (neat, cm^{-1}) 2984, 2929, 2874, 2240, 1956, 1715, 1598, 1495, 1446, 1352, 1248, 1164, 1094; ^1H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 7.6 Hz, 2H), 5.03 (quintet, J = 6.8 Hz, 1H), 4.80 (dt, J = 6.8, 2.4 Hz, 2H), 4.27 (s, 2H), 4.16 (q, J = 7.2 Hz, 2H), 3.86 (dt, J = 7.2, 2.4 Hz, 2H), 2.42 (s, 3H), 1.28 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.86, 152.56, 143.91, 135.47, 129.71, 127.58, 85.11, 80.05, 77.11, 76.73, 61.98, 46.15, 35.87, 21.50, 13.94; HRMS FAB calcd *m/z* for $C_{17}H_{20}NO_4S^+$ (M+H) 334.1113, obsd 334.1114.



Spectroscopic data of compound 1b: FT-IR (neat, cm^{-1}) 2984, 2926, 2873, 2238, 1962, 1714, 1598, 1494, 1446, 1347, 1253, 1163, 1093; ^1H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 4.67-4.70 (m, 2H), 4.22 (s, 2H), 4.14 (q, J = 7.2 Hz, 2H), 3.76 (t, J = 2.4 Hz, 2H), 2.42 (s, 3H), 1.71 (t, J = 2.8 Hz, 3H), 1.27 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.59, 152.52, 143.82, 135.41, 129.64, 127.57, 93.04, 79.94, 77.09, 75.43, 61.91, 50.47, 35.58, 21.47, 15.74, 13.92; HRMS FAB calcd *m/z* for $C_{18}H_{22}NO_4S^+$ (M+H) 348.1270, obsd 348.1265.

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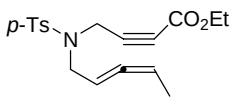
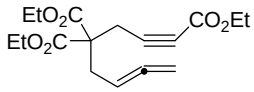
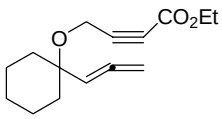
5. (a) J. K. Crandall and G. L. Tindell, *J. Chem. Soc., Chem. Commun.* 1970, 1411. (b) W. G. Dauben and G. Shapiro, *J. Org. Chem.* 1984, **49**, 4252. (c) M. A. Henderson and C. H. Heathcock, *J. Org. Chem.* 1988, **53**, 4736.



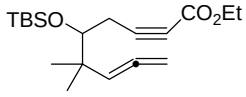
Spectroscopic data of compound 1c: FT-IR (neat, cm^{-1}) 2983, 2928, 2872, 2239, 1968, 1718, 1598, 1495, 1448, 1356, 1252, 1163, 1094; ^1H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, J = 8.4 Hz, 2H), 7.31 (d, J = 8.4 Hz, 2H), 5.14-5.22 (m, 1H), 4.91-4.98 (m, 1H), 4.28 (s, 2H), 4.15 (q, J = 7.2 Hz, 2H), 3.75-3.87 (m, 2H), 2.42 (s, 3H), 1.64 (dd, J = 7.2, 3.2 Hz, 3H), 1.27 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.63, 152.52, 143.81, 135.38, 129.64, 127.52, 87.66, 84.87, 80.06, 76.97, 61.90, 46.66, 35.63, 21.43, 13.89; HRMS FAB calcd *m/z* for $C_{18}H_{22}NO_4S^+$ (M+H) 348.1270, obsd 348.1271.



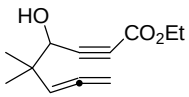
Spectroscopic data of compound 1d: FT-IR (neat, cm^{-1}) 2984, 2939, 2907, 2875, 2240, 1956, 1738, 1720, 1466, 1446, 1367, 1254, 1206, 1082; ^1H NMR (400 MHz, $CDCl_3$) δ 4.95 (quintet, J = 7.6 Hz, 1H), 4.70 (dt, J = 6.8, 2.0 Hz, 2H), 4.22 (q, J = 6.8 Hz, 4H), 4.20 (q, J = 6.8 Hz, 2H), 3.00 (s, 2H), 2.77 (dt, J = 7.6, 2.4 Hz, 2H), 1.29 (t, J = 7.2 Hz, 3H), 1.27 (t, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.14, 168.03, 153.20, 83.51, 83.23, 75.51, 75.00, 61.93, 61.82, 56.69, 31.84, 22.83, 13.95; HRMS FAB calcd *m/z* for $C_{17}H_{23}O_6^+$ (M+H) 323.1495, obsd 323.1499.



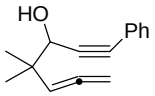
Spectroscopic data of compound 1e: FT-IR (neat, cm^{-1}) 2983, 2936, 2858, 2240, 1954, 1715, 1448, 1368, 1248, 1141; ^1H NMR (400 MHz, $CDCl_3$) δ 5.00 (t, J = 6.8 Hz, 1H), 4.85 (d, J = 6.8 Hz, 2H), 4.22 (q, J = 7.2 Hz, 2H), 4.17 (s, 2H), 1.25-1.73 (m, 10H), 1.30 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.70, 153.34, 94.78, 85.23, 77.16, 76.64, 67.62, 61.94, 50.03, 34.99, 25.48, 22.18, 13.97; HRMS FAB calcd *m/z* for $C_{15}H_{21}O_3^+$ (M+H) 249.1491, obsd 249.1491.



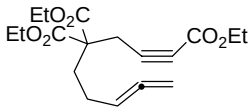
Spectroscopic data of compound 1f: FT-IR (neat, cm^{-1}) 2959, 2931, 2858, 2236, 1955, 1714, 1472, 1388, 1365, 1252, 1078; ^1H NMR (400 MHz, $CDCl_3$) δ 5.12 (t, J = 6.8 Hz, 1H), 4.74 (d, J = 6.8 Hz, 2H), 4.20 (q, J = 7.2 Hz, 2H), 3.61(dd, J = 6.0, 1.6 Hz, 1H), 2.65 (dd, J = 17.6, 4.4 Hz, 1H), 2.34 (dd, J = 17.6, 6.4 Hz, 1H), 1.29 (t, J = 7.2 Hz, 3H), 1.02 (d, J = 4.0 Hz, 6H), 0.91 (s, 9H), 0.16 (s, 3H), 0.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.33, 153.71, 97.21, 88.24, 78.02, 74.76, 69.09, 61.64, 40.08, 25.95, 24.95, 24.21, 23.69, 18.18, 13.99, -3.96, -4.51; HRMS FAB calcd *m/z* for $C_{19}H_{33}O_3Si^+$ (M+H) 337.2199, obsd 337.2198.



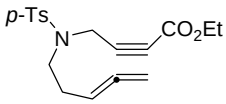
Spectroscopic data of compound 1g: FT-IR (neat, cm^{-1}) 3448, 2972, 2934, 2874, 2235, 1955, 1716, 1466, 1388, 1368, 1248, 1077; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.20 (t, *J* = 6.4 Hz, 1H), 4.84 (d, *J* = 6.8 Hz, 2H), 4.25 (q, *J* =7.2 Hz, 2H), 4.21 (d, *J* = 7.6 Hz, 1H), 2.07 (d, *J* = 7.6 Hz, 1H), 1.32 (t, *J* = 6.8 Hz, 3H), 1.16 (d, *J* = 5.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.67, 153.29, 95.79, 85.98, 77.75, 77.31, 70.22, 62.11, 39.87, 23.74, 23.64, 13.95; HRMS FAB calcd *m/z* for $C_{12}H_{17}O_3^+$ (M+H) 209.1178, obsd 209.1183.



Spectroscopic data of compound 1h: FT-IR (NaCl, cm^{-1}): 3429, 2968, 2872, 2200, 1955, 1667, 1599, 1490, 1467, 1444, 1385, 1047; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.45 (m, 5H), 5.30 (t, *J* = 6.8 Hz, 1H), 4.83 (d, *J* = 6.4 Hz, 2H), 4.31 (d, *J* = 7.2 Hz, 1H), 1.97 (d, *J* = 6.8 Hz, 1H), 1.20 (d, *J* = 5.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.64, 131.65, 128.36, 128.24, 122.59, 96.38, 88.12, 86.06, 76.91, 70.95, 40.13, 23.91, 23.63; HRMS FAB calcd *m/z* for $C_{15}H_{17}O^+$ (M+H) 213.1279, obsd 213.1283.



Spectroscopic data of compound 1i: FT-IR (neat, cm^{-1}): 2984, 2940, 2908, 2874, 2241, 1957, 1732, 1718, 1466, 1447, 1367, 1255, 1188, 1091; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.10 (quintet, *J* = 6.4 Hz, 1H), 4.71 (dt, *J* = 6.8, 3.2 Hz, 2H), 4.17-4.25 (m, 6H), 2.98 (s, 2H), 2.16-2.20 (m, 2H), 1.94-1.99 (m, 2H), 1.29 (t, *J* = 7.2 Hz, 3H), 1.27 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.37, 169.52, 153.21, 88.78, 83.29, 75.70, 75.46, 61.87, 56.26, 31.59, 23.14, 23.07, 13.93; HRMS FAB calcd *m/z* for $C_{18}H_{25}O_6^+$ (M+H) 337.1651, obsd 337.1654.

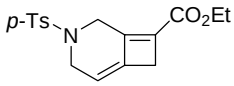


Spectroscopic data of compound 1j: FT-IR (neat, cm^{-1}) 2984, 2928, 2871, 2239, 1956, 1715, 1598, 1448, 1350, 1252, 1163, 1098; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 5.08 (quintet, *J* = 6.8 Hz, 1H), 4.72 (dt, *J* = 6.8, 2.8 Hz, 2H), 4.27 (s, 2H), 4.16 (q, *J* = 6.8 Hz, 2H), 3.27 (dd, *J* = 7.2 Hz, 2H), 2.42 (s, 3H), 2.26-2.32 (m, 2H), 1.28 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.85, 152.45, 143.77, 135.33, 129.63, 127.52, 86.10, 80.07, 77.11, 75.61, 61.96, 46.27, 36.53, 26.85, 21.46, 13.89; HRMS FAB calcd *m/z* for $C_{18}H_{22}NO_4S^+$ (M+H) 348.1270, obsd 348.1270.

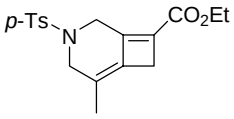
General Procedures for the [2+2] Intramolecular Cyclization of Allenynes:

Microwave method: A mixture of allenyne **1a-j** (1 mmol) and the appropriate solvent (1 mL) were placed in a well dried and screw capped test tube and subjected to microwave irradiation for a set time. After cooling, the solution was concentrated, and the residue was subjected to flash column chromatography to give products **2a-j**.

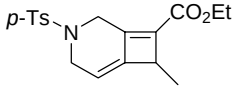
Metal catalyzed Method: In to a well dried 10 mL round bottomed flask, alleneynes **1a-j** (1 mmol), $PdCl_2(PPh_3)_2$ (5 mol%) and dry solvent toluene 2 mL were taken. The reaction mixture was heated to refluxing temperature. After completion of the reaction, cooled to room temperature and subjected for column chromatographic purification to get pure products **2a-j**.



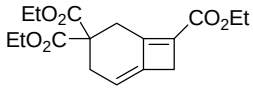
Spectroscopic data of compound 2a: R_f 0.44 (25% EtOAc/hexane); FT-IR (neat, cm^{-1}) 2967, 2924, 1727, 1691, 1625, 1597, 1493, 1368, 1345, 1263, 1092; ^1H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, J = 8.0 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 5.51 (t, J = 3.6 Hz, 1H), 4.22 (q, J = 7.2 Hz, 2H), 3.92 (d, J = 3.6 Hz, 2H), 3.09 (t, J = 2.4 Hz, 2H), 2.42 (s, 3H), 1.32 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.63, 148.67, 143.59, 135.34, 134.11, 129.50, 128.49, 127.40, 113.25, 60.55, 44.47, 43.08, 36.07, 21.64, 14.56; HRMS EI calcd *m/z* for $C_{17}H_{19}NO_4S$ (M^+) 333.1035, obsd 333.1023.



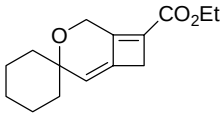
Spectroscopic data of compound 2b: R_f 0.55 (25% EtOAc/hexane); FT-IR (neat, cm^{-1}) 2917, 2841, 1727, 1679, 1623, 1453, 1326, 1234, 1181, 1101; ^1H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 4.19 (q, J = 7.2 Hz, 2H), 3.76 (s, 2H), 3.03 (t, J = 2.4 Hz, 2H), 2.42 (s, 3H), 1.66 (s, 3H), 1.31 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.88, 149.20, 143.52, 134.08, 129.95, 129.44, 127.25, 125.21, 123.17, 60.31, 48.33, 42.68, 34.73, 21.62, 15.56, 14.58; HRMS EI calcd *m/z* for $C_{18}H_{21}NO_4S$ (M^+) 347.1191, obsd 347.1182.



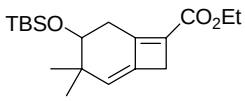
Spectroscopic data of compound 2c: R_f 0.45 (25% EtOAc/hexane); FT-IR (neat, cm^{-1}) 2925, 2837, 1730, 1692, 1628, 1444, 1343, 1267, 1160, 1092; ^1H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 8.4 Hz, 2H), 5.47 (t, J = 3.6 Hz, 1H), 4.20 (q, J = 7.2 Hz, 2H), 3.87-3.40 (m, 3H), 2.41 (s, 3H), 1.31 (t, J = 7.2 Hz, 3H), 1.14 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.55, 147.50, 143.52, 141.68, 134.42, 133.66, 129.52, 127.64, 127.39, 111.46, 60.32, 45.03, 44.29, 42.99, 21.67, 15.69, 14.55; HRMS EI calcd *m/z* for $C_{18}H_{21}NO_4S$ (M^+) 347.1191, obsd 347.1201.



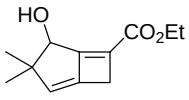
Spectroscopic data of compound 2d: R_f 0.46 (25% EtOAc/hexane); FT-IR (neat, cm^{-1}) 2981, 2883, 1731, 1681, 1622, 1486, 1369, 1298, 1239, 1198, 1101, 1056; ^1H NMR (400 MHz, $CDCl_3$) δ 5.55 (t, J = 4.0 Hz, 1H), 4.18-4.23 (m, 6H), 3.16 (t, J = 2.8 Hz, 2H), 3.07 (t, J = 2.8 Hz, 2H), 2.81 (d, J = 4.0 Hz, 2H), 1.31 (t, J = 7.6 Hz, 3H), 1.23 (t, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.30, 163.38, 152.86, 136.76, 128.50, 114.25, 61.76, 60.12, 35.51, 31.10, 29.65, 14.55, 14.08; HRMS EI calcd *m/z* for $C_{17}H_{22}O_6$ (M^+) 322.1416, obsd 322.1427.



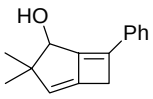
Spectroscopic data of compound 2e: R_f 0.51 (10% EtOAc/hexane); FT-IR (neat, cm^{-1}) 2933, 2857, 2359, 1728, 1445, 1369, 1266, 1092; ^1H NMR (400 MHz, $CDCl_3$) δ 5.66 (s, 1H), 4.53 (t, J = 2.8 Hz, 2H), 4.17 (q, J = 7.2 Hz, 2H), 3.28 (t, J = 2.8 Hz, 2H), 1.65-1.76 (m, 2H), 1.59-1.63 (m, 4H), 1.48-1.51 (m, 4H), 1.26 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.13, 152.70, 133.31, 125.76, 124.95, 77.10, 73.41, 61.70, 36.44, 35.31, 25.49, 21.86, 14.51; HRMS EI calcd *m/z* for $C_{15}H_{20}O_3$ (M^+) 248.1412, obsd 248.1423.



Spectroscopic data of compound 2f: R_f 0.45 (10% EtOAc/hexane); FT-IR (neat, cm^{-1}) 2956, 2929, 2857, 1713, 1680, 1548, 1468, 1370, 1301, 1238, 1178, 1096; ^1H NMR (400 MHz, $CDCl_3$) δ 5.36 (s, 1H), 4.19 (m, 2H), 3.67 (dd, J = 5.2, 4.8 Hz, 1H), 3.13 (t, J = 3.2 Hz, 2H), 2.67-2.72 (bm, 1H), 2.36-2.48 (m, 1H), 1.29 (t, J = 7.2 Hz, 3H), 1.07 (s, 3H), 0.99 (s, 3H), 0.90 (s, 9H), 0.08 (s, 3H), 0.05 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.65, 156.39, 135.16, 128.53, 127.54, 75.83, 59.95, 39.05, 35.09, 30.39, 27.70, 25.92, 22.18, 18.14, 14.55, -3.83, -4.77; HRMS EI calcd *m/z* for $C_{19}H_{32}O_3Si$ (M^+) 336.2121, obsd 336.2118.

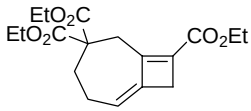


Spectroscopic data of compound 2g: R_f 0.45 (25% EtOAc/hexane); FT-IR (neat, cm^{-1}) 3661, 2977, 1727, 1607, 1548, 1466, 1369, 1248, 1179, 1096; ^1H NMR (400 MHz, $CDCl_3$) δ 5.48 (s, 1H), 4.59-4.60 (m, 1H), 4.21 (m, 2H), 3.23-3.34 (m, 2H), 2.35 (b, 1H), 1.31 (t, J = 6.9 Hz, 3H), 1.18 (s, 3H), 1.14 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.10, 163.00, 139.43, 132.13, 125.08, 78.43, 63.11, 58.12, 32.53, 28.21, 26.23, 25.92; HRMS EI calcd *m/z* for $C_{12}H_{16}O_3$ (M^+) 208.1099, obsd 208.1085.

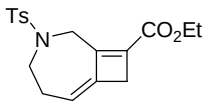


Spectroscopic data of compound 2h: R_f 0.49 (25% EtOAc/hexane); FT-IR (neat, cm^{-1}) 3661, 2977, 2893, 1607, 1548, 1466, 1369, 1248, 1179, 1096; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 6.8 Hz, 1H), 7.34 (t, J = 7.2, 6.8 Hz,

2H), 7.24 (m, 2H), 5.13 (s, 1H), 4.60 (m, 1H), 3.35 (m, 2H), 1.71 (m, 1H), 1.25 (s, 3H), 1.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.43, 140.48, 134.65, 128.97, 128.52, 127.50, 126.18, 124.59, 79.03, 34.45, 28.53, 25.97, 22.90; HRMS EI calcd *m/z* for $C_{15}H_{16}O$ (M^+) 212.1201, obsd 212.1206.



Spectroscopic data of compound 2i: R_f 0.59 (25% EtOAc/hexane); FT-IR (neat, cm^{-1}) 2923, 2887, 1712, 1687, 1598, 1444, 1345, 1160, 1091; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.35 (t, *J* = 3.6 Hz, 1H), 4.13-4.22 (m, 6H), 3.08 (s, 2H), 2.95 (s, 2H), 2.34 (m, 4H), 1.89-1.30 (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.86, 163.99, 155.39, 136.74, 131.02, 121.81, 61.56, 61.42, 59.96, 59.80, 56.36, 36.78, 33.81, 32.01, 27.77; HRMS EI calcd *m/z* for $C_{18}H_{24}O_6$ (M^+) 336.1573, obsd 336.1587.



Spectroscopic data of compound 2j: R_f 0.47 (25% EtOAc/hexane); FT-IR (neat, cm^{-1}) 2981, 2883, 1731, 1681, 1622, 1486, 1369, 1298, 1239, 1198, 1101, 1056; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 7.6 Hz, 2H), 5.43 (t, *J* = 4.4 Hz, 1H), 4.22 (q, *J* = 7.2 Hz, 2H), 3.41 (m, 2H), 2.96 (t, *J* = 2.8 Hz, 2H), 2.53 (m, 2H), 2.42 (s, 3H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.33, 154.18, 143.47, 135.93, 135.77, 130.78, 127.59, 127.04, 121.69, 60.38, 49.60, 49.24, 34.01, 33.15, 29.67, 21.48; HRMS EI calcd *m/z* for $C_{18}H_{21}NO_4S$ (M^+) 347.1191, obsd 347.1195.