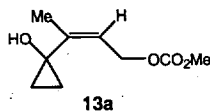
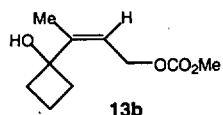


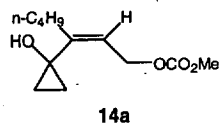
Characterization of compounds



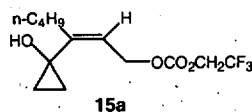
13a: R_f 0.3 (petroleum ether : Et₂O = 1:1). IR (neat) 3420, 2960, 1748, 1728, 1445, 1274, 939 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.42 (t, J = 7.3 Hz, 1H), 4.96 (d, J = 7.3 Hz, 1H), 3.79 (s, 3H), 3.66 (br s, 1H), 1.85 (s, 3H), 0.99 (dd, J = 7.1, 5.1 Hz, 2H), 0.65 (dd, J = 7.1, 5.1 Hz, 2H). ¹³C-NMR (CDCl₃, 75 MHz): δ 156.29, 143.42, 122.24, 64.72, 54.85, 54.59, 21.51, 13.51. Calcd. for C₉H₁₄O₄: C, 58.05; H, 7.58. Found: C, 58.22; H, 7.45.



13b: R_f 0.3 (petroleum ether : Et₂O = 1:1). IR (neat) 3437, 2957, 1749, 1444, 1356, 1274, 1140, 944, 907, 794 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.33 (tq, J = 7.6, 1.2 Hz, 1H), 4.96 (dq, J = 7.6, 0.7 Hz, 2H), 3.78 (s, 3H), 3.23 (s, 1H), 2.1-2.4 (m, 5H), 1.78 (d, J = 1.2 Hz, 3H), 1.65-1.75 (m, 1H). ¹³C-NMR (CDCl₃, 75 MHz): δ 156.17, 146.91, 120.18, 77.11, 64.88, 54.79, 36.02, 20.31, 15.16. Calcd. for C₁₀H₁₆O₄: C, 59.98; H, 8.05. Found: C, 60.07; H, 7.86.

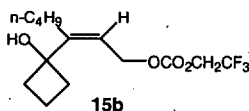


14a: R_f 0.5 (petroleum ether : Et₂O = 1:1). IR (neat) 3480, 2959, 2933, 1749, 1732, 1444, 1367, 1273, 1223, 1020, 942, 795 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.41 (t, J = 7.6 Hz, 1H), 4.99 (d, J = 7.6 Hz, 2H), 3.79 (s, 3H), 3.65 (s, 1H), 2.14 (t, J = 7.3 Hz, 3H), 1.3-1.6 (m, 4H), 0.99 (dd, J = 6.8, 5.4 Hz, 2H), 0.92 (t, J = 7.3 Hz, 3H), 0.62 (dd, J = 6.8, 5.4 Hz, 2H). ¹³C-NMR (CDCl₃, 75 MHz): δ 156.31, 147.38, 121.82, 65.00, 54.92, 54.27, 35.06, 30.06, 22.51, 13.97, 13.51. Calcd. for C₁₂H₂₀O₄: C, 63.14; H, 8.83. Found: C, 63.04; H, 8.68.

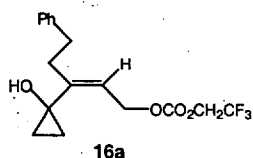


15a: R_f 0.5 (petroleum ether : Et₂O = 1:1). IR (neat) 3453, 2964, 2934, 2864, 1760, 1456, 1417, 1369, 1310, 1247, 1172, 962 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.43 (t, J = 7.3 Hz, 1H), 5.06 (d, J = 7.3 Hz, 2H), 4.52 (q, J = 8.1 Hz, 2H), 3.25 (s, 1H), 2.14 (t, J = 7.6 Hz, 2H), 1.2-1.6 (m,

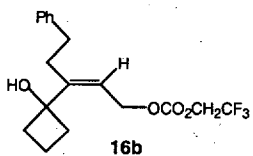
4H), 1.00 (dd, $J = 7.1, 5.4$ Hz, 2H), 0.92 (t, $J = 7.1$ Hz, 3H), 0.64 (dd, $J = 7.1, 5.4$ Hz, 2H). ^{13}C -NMR (CDCl_3 , 75 MHz): δ 154.90, 148.45, 122.86 (q, $^1J_{\text{C-F}} = 277$ Hz), 121.66, 66.66, 63.87 (q, $^2J_{\text{C-F}} = 38$ Hz), 54.82, 35.53, 30.55, 22.91, 14.39, 13.89. Calcd. for $\text{C}_{13}\text{H}_{19}\text{F}_3\text{O}_4$: C, 52.70; H, 6.46. Found: C, 52.52; H, 6.26.



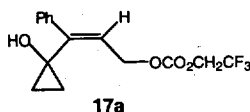
15b: R_f 0.6 (petroleum ether : $\text{Et}_2\text{O} = 1:1$). IR (neat) 3472, 2959, 2875, 1762, 1458, 1417, 1365, 1302, 1247, 1171, 962, 788 cm^{-1} . ^1H -NMR (CDCl_3 , 300 MHz): δ 5.29 (t, $J = 7.3$ Hz, 1H), 4.83 (d, $J = 7.3$ Hz, 2H), 4.50 (q, $J = 8.3$ Hz, 2H), 2.78 (s, 1H), 2.3-2.5 (m, 5H), 1.6-1.7 (m, 1H), 1.3-1.5 (m, 4H), 0.91 (t, $J = 7.1$ Hz, 3H). ^{13}C -NMR (CDCl_3 , 75 MHz): δ 154.34, 151.54, 122.44 (q, $^1J_{\text{C-F}} = 277$ Hz), 119.16, 77.55, 66.37, 63.39 (q, $^2J_{\text{C-F}} = 36$ Hz), 36.49, 33.59, 30.55, 22.46, 15.05, 13.92. Calcd. for $\text{C}_{14}\text{H}_{21}\text{F}_3\text{O}_4$: C, 54.19; H, 6.82. Found: C, 54.38; H, 6.91.



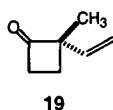
16a: R_f 0.6 (petroleum ether : $\text{Et}_2\text{O} = 1:1$). IR (neat) 3446, 3028, 2930, 1762, 1455, 1417, 1369, 1307, 1247, 1171, 962 cm^{-1} . ^1H -NMR (CDCl_3 , 300 MHz): δ 7.2-7.4 (m, 5H), 5.46 (t, $J = 7.1$ Hz, 1H), 5.05 (d, $J = 7.1$ Hz, 2H), 4.50 (q, $J = 8.3$ Hz, 2H), 3.22 (s, 1H), 2.86 (t, $J = 7.6$ Hz, 2H), 2.46 (t, $J = 7.6$ Hz, 2H), 1.02 (dd, $J = 7.1, 5.3$ Hz, 2H), 0.66 (dd, $J = 7.1, 5.3$ Hz, 2H). ^{13}C -NMR (CDCl_3 , 75 MHz): δ 154.35, 146.75, 141.51, 128.34, 128.27, 125.86, 124.24, 122.41 (q, $^1J_{\text{C-F}} = 277$ Hz), 122.06, 65.98, 63.39 (q, $^2J_{\text{C-F}} = 38$ Hz), 54.25, 37.05, 34.29, 13.45. HRMS: Calcd. for $\text{C}_{17}\text{H}_{19}\text{F}_3\text{O}_4$: 344.1235. Found: 344.1235.



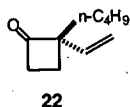
16b: R_f 0.4 (petroleum ether : $\text{Et}_2\text{O} = 1:1$). IR (neat) 3458, 2939, 1761, 1455, 1417, 1365, 1302, 1247, 1171, 1131, 962 cm^{-1} . ^1H -NMR (CDCl_3 , 300 MHz): δ 7.1-7.4 (m, 5H), 5.32 (t, $J = 7.3$ Hz, 1H), 4.82 (d, $J = 7.3$ Hz, 2H), 4.49 (q, $J = 8.3$ Hz, 2H), 2.82 (s, 1H), 2.75 (t, $J = 7.6$ Hz, 2H), 2.3-2.5 (m, 4H), 2.1-2.3 (m, 3H), 1.7-1.8 (m, 1H). ^{13}C -NMR (CDCl_3 , 75 MHz): δ 154.27, 150.39, 141.44, 128.35, 128.32, 125.91, 122.41 (q, $^1J_{\text{C-F}} = 278$ Hz), 119.86, 77.44, 66.33, 63.39 (q, $^2J_{\text{C-F}} = 37$ Hz), 36.49, 35.58, 34.68, 15.13. HRMS: Calcd. for $\text{C}_{18}\text{H}_{21}\text{F}_3\text{O}_4$: 358.1392. Found: 358.1399.



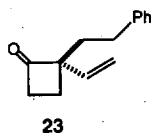
17a: R_f 0.4 (petroleum ether : Et₂O = 2:1). IR (neat) 3492, 1761, 1447, 1416, 1369, 1312, 1248, 1173, 964, 762, 699 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 7.5-7.6 (m, 2H), 7.3-7.4 (m, 3H), 5.98 (t, J = 7.3 Hz, 1H), 5.27 (d, J = 7.3 Hz, 2H), 4.51 (q, J = 8.1 Hz, 2H), 3.86 (s, 1H), 1.18 (dd, J = 7.3, 5.3 Hz, 2H), 0.62 (dd, J = 7.3, 5.3 Hz, 2H). ¹³C-NMR (CDCl₃, 75 MHz): δ 154.61, 146.94, 139.48, 128.21, 126.64, 123.89, 122.34 (q, $^1J_{C-F}$ = 277 Hz), 66.47, 63.56 (q, $^2J_{C-F}$ = 37 Hz), 54.07, 14.82. Calcd. for C₁₅H₁₅F₃O₄: C, 56.96; H, 4.78. Found: C, 57.10; H, 4.64.



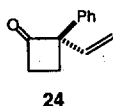
19: known compound¹
 $[\alpha]_D = -57.8$ (c 1.12, CHCl₃), [91% ee (*S*)]



22: R_f 0.7 (petroleum ether : Et₂O = 2:1). $[\alpha]_D = -49.3$ (c=1.35, CHCl₃), [90% ee (*S*)]. IR (neat) 2959, 2939, 2862, 1779, 1633, 1468, 1071, 995, 919 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.83 (dd, J = 17.3, 10.5 Hz, 1H), 5.16 (d, J = 17.3 Hz, 1H), 5.14 (d, J = 10.5 Hz, 1H), 2.9-3.1 (m, 2H), 2.1-2.2 (m, 1H), 1.9-2.0 (m, 1H), 1.64 (t, J = 7.3 Hz, 2H), 1.2-1.4 (m, 4H), 0.89 (t, J = 7.1 Hz, 3H). ¹³C-NMR (CDCl₃, 75 MHz): δ 212.29, 137.19, 114.46, 71.58, 42.25, 35.31, 26.46, 22.93, 21.46, 13.92. Calcd. for C₁₀H₁₆O: C, 78.90; H, 10.59. Found: C, 78.67; H, 10.57.

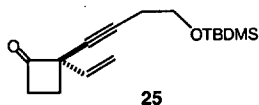


23: R_f 0.8 (petroleum ether : Et₂O = 2:1). $[\alpha]_D = -19.8$ (c=1.69, CHCl₃), [92% ee (*R*)]. IR (neat) 3027, 2926, 1776, 1632, 1604, 1497, 1454, 1072, 919, 745, 700 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 7.1-7.3 (m, 5H), 5.91 (dd, J = 17.6, 10.5 Hz, 1H), 5.24 (d, J = 17.6 Hz, 1H), 5.23 (d, J = 10.5 Hz, 1H), 2.9-3.1 (m, 2H), 2.5-2.8 (m, 2H), 2.1-2.2 (m, 1H), 1.9-2.0 (m, 3H). ¹³C-NMR (CDCl₃, 75 MHz): δ 211.43, 141.64, 136.70, 128.40, 128.26, 125.93, 115.15, 71.39, 42.39, 37.38, 30.71, 21.68. Anal. Calcd. for C₁₄H₁₆O: C, 83.96; H, 8.05. MW, 200.1201. Found: C, 83.96; H, 7.87, MW, 200.1201.

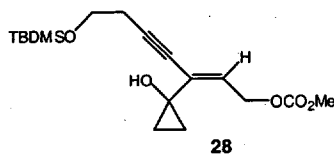


24: known compound²

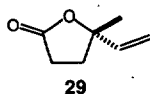
$[\alpha]_D = -8.52$ ($c=2.07$, CHCl_3), [70% ee (*S*)].



25: R_f 0.7 (petroleum ether : Et_2O = 4:1). $[\alpha]_D = +21.8$ ($c=1.88$, CHCl_3), [89% ee (*R*)]. IR (neat) 2956, 2930, 2857, 1797, 1632, 1472, 1391, 1256, 1109, 1056, 919, 837 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 300 MHz): δ 5.86 (dd, $J = 16.8, 10.0$ Hz, 1H), 5.43 (d, $J = 16.8$ Hz, 1H), 5.22 (d, $J = 10.0$ Hz, 1H), 3.72 (t, $J = 7.1$ Hz, 2H), 3.1-3.3 (m, 2H), 2.47 (t, $J = 7.1$ Hz, 2H), 2.2-2.4 (m, 2H), 0.89 (s, 9H), 0.07 (s, 6H). $^{13}\text{C-NMR}$ (CDCl_3 , 75 MHz): δ 203.13, 134.11, 116.36, 84.75, 77.32, 64.79, 61.86, 44.59, 25.86, 25.76, 23.32, 18.30, -5.30. HRMS: Calcd. for $\text{C}_{16}\text{H}_{27}\text{O}_2\text{Si}$ ($\text{M}^+ + \text{H}$): 279.1780. Found: 279.1768. Anal. Calcd. for $\text{C}_{16}\text{H}_{26}\text{O}_2\text{Si}$: C, 69.01; H, 9.41. Found: C, 69.18; H, 9.56.

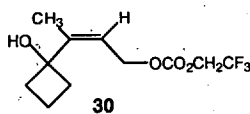


28: R_f 0.7 (petroleum ether : Et_2O = 1:2). IR (neat) 3426, 2956, 2850, 1751, 1444, 1271, 1105, 940, 837, 777 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 300 MHz): δ 5.93 (t, $J = 7.6$ Hz, 1H), 5.07 (d, $J = 7.6$ Hz, 2H), 3.79 (s, 3H), 3.66 (s, 1H), 2.51 (t, $J = 7.1$ Hz, 2H), 1.0-1.1 (m, 4H), 0.89 (s, 9H), 0.07 (s, 6H). $^{13}\text{C-NMR}$ (CDCl_3 , 75 MHz): δ 156.13, 131.12, 130.70, 88.02, 80.28, 64.25, 61.65, 55.40, 54.95, 25.79, 23.61, 18.24, 15.54, -5.37. HRMS: Calcd for $\text{C}_{14}\text{H}_{21}\text{O}_5\text{Si}$ ($\text{M}^+ - \text{C}_4\text{H}_9$): 297.1158. Found: 297.1154. Anal. Calcd. for $\text{C}_{18}\text{H}_{30}\text{O}_5\text{Si}$: C, 60.96; H, 8.53. Found: C, 60.73; H, 8.39.

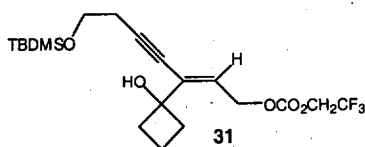


29: known compound³

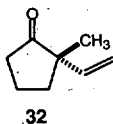
$[\alpha]_D -15.0$ (c 0.90, CHCl_3), [91% ee (*S*)].



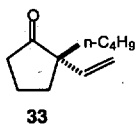
30: R_f 0.5 (petroleum ether : Et₂O = 1:1). IR (neat) 3428, 2980, 1761, 14.17, 1303, 1248, 1172, 1140, 962, 787 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.32 (tq, J = 7.6, 1.2 Hz, 1H), 4.80 (dq, J = 7.6, 0.7 Hz, 2H), 4.50 (q, J = 8.3 Hz, 2H), 2.78 (s, 1H), 2.3-2.5 (m, 2H), 2.1-2.3 (m, 3H), 1.80 (d, J = 1.2 Hz, 3H), 1.6-1.8 (m, 1H). ¹³C-NMR (CDCl₃, 75 MHz): δ 154.30, 147.44, 122.41 (q, $^1J_{C-F}$ = 278 Hz), 119.62, 77.26, 66.13, 63.33 (q, $^2J_{C-F}$ = 37 Hz), 36.10, 20.42, 15.08. Calcd. for C₁₁H₁₅F₃O₄: C, 49.26; H, 5.64. Found: C, 49.42; H, 5.84.



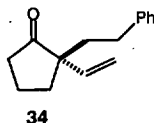
31: R_f 0.6 (petroleum ether : Et₂O = 1:1). IR (neat) 3442, 2955, 2933, 2859, 2361, 1765, 1302, 1251, 1172, 1107, 838, 779 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.88 (t, J = 7.1 Hz, 1H), 4.95 (d, J = 7.1 Hz, 2H), 4.50 (q, J = 8.3 Hz, 2H), 3.75 (t, J = 7.1 Hz, 2H), 2.5-2.6 (m, 3H), 2.56 (t, J = 7.1 Hz, 2H), 2.1-2.3 (m, 2H), 1.9-2.0 (m, 1H), 1.7-1.8 (m, 1H). ¹³C-NMR (CDCl₃, 75 MHz): δ 154.09, 134.57, 129.99, 122.41 (q, $^1J_{C-F}$ = 277 Hz), 90.17, 79.93, 75.84, 65.71, 63.43 (q, $^2J_{C-F}$ = 38 Hz), 61.71, 37.01, 25.79, 23.74, 18.24, 13.71, -5.38. Calcd. for C₂₀H₃₁F₃O₅Si: C, 55.03; H, 7.16. Found: C, 55.26; H, 7.01.



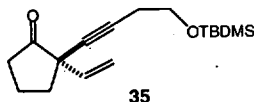
32: known compound⁴
 $[\alpha]_D = -59.6$ (c=1.02, CHCl₃), [87% ee (*S*)].



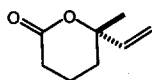
33: R_f 0.7 (petroleum ether : Et₂O = 2:1). $[\alpha]_D = -23.4$ (c=2.30, CHCl₃), [82% ee (*S*)]. IR (neat) 2959, 2933, 2861, 1738, 1633, 1468, 1407, 1153, 1002, 917 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.70 (dd, J = 17.3, 10.5 Hz, 1H), 5.13 (d, J = 10.5 Hz, 1H), 5.06 (d, J = 17.3 Hz, 1H), 2.0-2.4 (m, 3H), 1.8-2.0 (m, 3H), 1.0-1.6 (m, 6H), 0.88 (t, J = 6.6 Hz, 3H). ¹³C-NMR (CDCl₃, 75 MHz): δ 220.15, 139.24, 114.89, 56.00, 37.54, 35.84, 32.46, 26.41, 23.06, 18.74, 13.90. Calcd. for C₁₁H₁₈O: C, 79.46; H, 10.91. Found: C, 79.60; H, 10.75.



34: R_f 0.7 (petroleum ether : Et₂O = 10:1). $[\alpha]_D = -20.1$ ($c=2.00$, CHCl₃), [69% ee (*S*)]. IR (neat) 2946, 2357, 1736, 1631, 1496, 1454, 1403, 1153, 921 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 7.1-7.3 (m, 5H), 5.77 (dd, $J = 17.6, 10.7$ Hz, 1H), 5.22 (d, $J = 10.7$ Hz, 1H), 5.14 (d, $J = 17.6$ Hz, 1H), 2.2-2.7 (m, 5H), 1.6-2.1 (m, 5H). ¹³C-NMR (CDCl₃, 75 MHz): δ 219.57, 142.01, 138.71, 128.34, 128.29, 125.83, 115.70, 56.06, 38.04, 37.51, 32.75, 30.73, 18.80. Anal. Calcd. for C₁₅H₂₈O: C, 84.07; H, 8.47. Found: C, 83.89; H, 8.63.



35: R_f 0.6 (petroleum ether : Et₂O = 4:1). $[\alpha]_D = +91.3$ ($c=1.87$, CHCl₃), [93% ee (*R*)]. IR (neat) 2956, 2858, 1753, 1639, 1472, 1406, 1256, 1111, 1006, 919, 838, 777 cm⁻¹. ¹H-NMR (CDCl₃, 300 MHz): δ 5.72 (dd, $J = 17.1, 10.0$ Hz, 1H), 5.48 (d, $J = 17.1$ Hz, 1H), 5.28 (d, $J = 10.0$ Hz, 1H), 3.70 (t, $J = 7.1$ Hz, 2H), 2.5-2.6 (m, 1H), 2.44 (t, $J = 7.1$ Hz, 2H), 1.9-2.3 (m, 5H), 0.89 (s, 9H), 0.06 (s, 6H). ¹³C-NMR (CDCl₃, 75 MHz): δ 213.36, 135.70, 117.14, 83.18, 78.39, 61.86, 52.57, 37.70, 36.33, 25.83, 23.24, 19.31, 18.25, -5.35. Anal. Calcd. for C₁₇H₂₈O₂Si: C, 69.81; H, 9.65; MW, 292.1859. Found: C, 69.68; H, 9.88; MW, 292.1857.



known compound⁵

$[\alpha]_D +5.39$ (c 1.49, CHCl₃), [87% ee (*S*)].

References:

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