A Mild Method for Indium-Catalyzed 1, 4-Hydrosilylation of α, β-Enone Esters with Triethylsilane and Trifluoroacetic Acid

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1. General information

Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Analytical thin-layer chromatography (TLC) was performed on Silicycle silica gel plates with F-254 as an indicator and compounds were visualized by irradiation with UV light. Flash column chromatography was carried out using silica gel H (40 μm). 1H NMR and 13C NMR spectra were recorded on Varian Mercury-300 spectrometer (300 MHz 1H, 75 MHz 13C). The spectra were recorded in CDCl3 as solvent at room temperature, 1H and 13C NMR chemical shifts are reported in ppm relative to either the residual solvent peak (1H, 13C) or TMS (1H) as an internal standard. IR spectra were recorded using Pekin-Elmer 983 instrument and are reported in wave numbers (cm⁻¹). MS was performed on Agilent Technologies 1200 Series mass instrument (ESI). HRMS was performed on Bruker Daltonics Apex III mass instrument (ESI).

2. General procedure for the synthesis of the α, β-enone ester

A mixture of benzaldehyde S (0.1 mmol), and 2-carboxy-2-oxoethan-1-ylium (0.1 mol) in MeOH (30.0 mL) was treated with KOH (0.15 mol in 20.0 mL MeOH) at 0 °C dropwise. The solution was stirred at room temperature until no benzaldehyde was detected by TLC. The yellow suspension was filtered and then washed the solid with ice MeOH and ethyl ether giving M as yellow solid.

The M was added to the solution of AcCl (20 mmol) in EtOH (30 mL) at 0°C. The solution was stirred 2 hours at room temperature and 6 hours at reflux temperature, and then cooled to room temperature. The solution was quenched with H2O (10 mL) and EtOH was removed by vacuum distillation. The aqueous layer was extracted by DCM (10 mL×2). The combined organic layer was dried over anhydrous Na2SO4, and concentrated to dryness, the residue was purified by flash chromatography on silica gel to give the product 1.

3. Analytical date of the α, β-enone ester

(Å)-Ethyl 4-(2-fluorophenyl)-2-oxobut-3-enoate (1a)

Yellow solid (78%). 1H NMR (300 MHz, CDCl3): 8.04-7.11 (m, 6H), 4.41 (q, J = 7.2 Hz, 2H), 1.42 (t, J = 7.2 Hz, 3H). 13C NMR (75 MHz, CDCl3): 182.9, 162.0, 161.9 (d, J = 193 Hz), 140.7 (d, J = 3 Hz), 133.1 (d, J = 9 Hz), 129.4 (d, J = 2 Hz), 124.6 (d, J = 3 Hz), 122.6 (d, J = 7 Hz), 122.2 (d, J = 11 Hz), 116.4 (d, J = 22 Hz), 62.6, 14.0. MS (ESI) m/z 244.9 [M+Na]+. HRMS (ESI) calcd. for
(E)-Ethyl 4-(3-fluorophenyl)-2-oxobut-3-enoate (1b)

Yellow solid (83%). $^1$H NMR (300 MHz, CDCl$_3$): 7.83-7.12 (m, 6H), 4.39 (q, $J = 7.2$ Hz, 2H), 1.41 (t, $J = 7.2$ Hz, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): 182.5, 162.9 (d, $J = 246$ Hz), 161.9, 146.7 (d, $J = 2$ Hz), 136.2 (d, $J = 7$ Hz), 130.6 (d, $J = 8$ Hz), 125.0 (d, $J = 3$ Hz), 121.6, 118.4 (d, $J = 21$ Hz), 114.5 (d, $J = 19$ Hz), 62.6, 14.0. MS (ESI) $m/z$ 245.1 [M+Na]$^+$. HRMS (ESI) calcd. for C$_{12}$H$_{11}$FNaO$_3$ 245.0590 found 245.0585. IR (cm$^{-1}$): 3073, 2983, 2940, 2909, 1728, 1687, 1604, 1084, 771.

(E)-Ethyl 4-(4-fluorophenyl)-2-oxobut-3-enoate (1c)

Yellow solid (85%). $^1$H NMR (300 MHz, CDCl$_3$): 7.83 (d, $J = 16.1$ Hz, 1H), 7.64 (dd, $J = 8.8, 5.4$ Hz, 2H), 7.31 (d, $J = 16.1$ Hz, 1H), 7.12 (t, $J = 8.6$ Hz, 2H), 4.39 (q, $J = 7.1$ Hz, 2H), 1.41 (t, $J = 7.1$ Hz, 3H).

(E)-Ethyl 4-(2-chlorophenyl)-2-oxobut-3-enoate (1d)

Yellow oil (75%). $^1$H NMR (300 MHz, CDCl$_3$): 8.29 (d, $J = 16.2$ Hz, 1H), 7.75 (dd, $J = 7.6, 1.8$ Hz, 1H), 7.68 - 7.14 (m, 4H), 4.40 (q, $J = 7.1$ Hz, 2H), 1.42 (t, $J = 7.2$ Hz, 3H).

(E)-Ethyl 4-(3-chlorophenyl)-2-oxobut-3-enoate (1e)

Yellow solid (80%). $^1$H NMR (300 MHz, CDCl$_3$): 7.82-7.35 (m, 6 H), 4.40 (q, $J = 7.2$ Hz, 2 H), 1.42 (t, $J = 7.2$ Hz, 3 H).

(E)-Ethyl 4-(4-chlorophenyl)-2-oxobut-3-enoate (1f)

Yellow solid (84%). $^1$H NMR (300 MHz, CDCl$_3$): 7.82 (d, $J = 16.0$ Hz, 1H), 7.59-7.40 (m, 4H), 7.34 (d, $J = 16.0$ Hz, 1 H), 4.40 (q, $J = 6.9$ Hz, 2H), 1.42 (t, $J = 6.9$ Hz, 3H).

(E)-Ethyl 4-(2-bromophenyl)-2-oxobut-3-enoate (1g)

Yellow oil (78%). $^1$H NMR (300 MHz, CDCl$_3$): 8.25 (d, $J = 16.2$ Hz, 1 H), 7.74 (dd, $J = 7.8, 1.5$ Hz, 1 H), 7.65 (d, $J = 7.8$ Hz, 1 H), 7.40-7.29 (m, 3H), 4.41 (q, $J = 6.9$ Hz, 2 H), 1.43 (t, $J = 6.9$ Hz, 3 H).

(E)-Ethyl 4-(3-bromophenyl)-2-oxobut-3-enoate (1h)

Yellow oil (85%). $^1$H NMR (300 MHz, CDCl$_3$): 8.30 (d, $J = 16.2$ Hz, 1 H), 7.79 (dd, $J = 7.8, 1.5$ Hz, 1 H), 7.66 (d, $J = 7.8$ Hz, 1 H), 7.40-7.29 (m, 3H), 4.42 (q, $J = 6.9$ Hz, 2 H), 1.43 (t, $J = 6.9$ Hz, 3 H).
Yellow solid (86%). $^1$H NMR (300 MHz, CDCl$_3$): 7.80-7.31 (m, 6H), 4.40 (q, $J = 7.2$ Hz, 2H), 1.42 (t, $J = 7.2$ Hz, 3H).

**(E)-Ethyl 4-(4-bromophenyl)-2-oxobut-3-enoate (1i)**

Yellow solid (85%). $^1$H NMR (300 MHz, CDCl$_3$): 7.79 (d, $J = 16.0$ Hz, 1H), 7.58-7.49 (m, 4H), 7.37 (d, $J = 16.0$ Hz, 1H), 4.40 (q, $J = 7.2$ Hz, 2H), 1.42 (t, $J = 7.2$ Hz, 3H).

**(E)-Ethyl 4-(4-methoxyphenyl)-2-oxobut-3-enoate (1j)**

Yellow oil (82%). $^1$H NMR (300 MHz, CDCl$_3$): 7.84 (d, $J = 16.0$ Hz, 1H), 7.25 (d, $J = 16.0$ Hz, 1H), 7.61-6.93 (m, 4H), 4.39 (q, $J = 7.2$ Hz, 2H), 3.86 (s, 3H), 1.41 (t, $J = 7.2$ Hz, 3H).

**(E)-Ethyl 2-oxo-4-phenylbut-3-enoate (1k)**

Yellow oil (81%). $^1$H NMR (300 MHz, CDCl$_3$): 7.86 (d, $J = 16.1$ Hz, 1H), 7.63 (dd, $J = 7.4$, 1.8 Hz, 2H), 7.54-7.40 (m, 3H), 7.36 (d, $J = 16.1$ Hz, 1H), 4.54-4.30 (q, $J = 7.1$ Hz, 2H), 1.37 (t, $J = 7.1$ Hz, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): 182.8, 162.1, 148.4, 133.9, 131.6, 129.0, 129.0, 120.4, 62.5, 14.0. MS (ESI) $m/z$ 227.1 [M+Na]$^+$. HRMS (ESI) calcd. for C$_{12}$H$_{12}$NaO$_3$ 227.0679 found 227.0685. IR (cm$^{-1}$): 1729, 1694, 1666, 1608, 1576, 1259, 1144, 1081.

**(E)-Ethyl 4-(4-nitrophenyl)-2-oxobut-3-enoate (1l)**

Yellow solid (80%). $^1$H NMR (300 MHz, CDCl$_3$): 8.28 (d, $J = 8.7$ Hz, 2H), 7.87 (d, $J = 16.2$ Hz, 1H), 7.79 (d, $J = 8.6$ Hz, 2H), 7.50 (d, $J = 16.2$ Hz, 1H), 4.41 (q, $J = 7.1$ Hz, 2H), 1.42 (t, $J = 7.1$ Hz, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): 182.0, 161.4, 149.7, 139.8, 139.4, 129.0, 124.2, 123.79, 62.83, 13.96. MS (ESI) $m/z$ 271.9 [M+Na]$^+$. HRMS (ESI) calcd. for C$_{12}$H$_{11}$NNaO$_5$ 272.0529 found 272.0533. IR (cm$^{-1}$): 1724, 1693, 1618, 1595, 1511, 1349, 1263, 1088.

**(E)-Ethyl 4-(furan-2-yl)-2-oxobut-3-enoate (1p)**

$^1$H NMR (300 MHz, DMSO-d$_6$): 7.99 (s, 1H), 7.62 (d, $J = 15.9$ Hz, 1H), 7.49 (d, $J = 15.9$ Hz, 1H), 7.01 (d, $J = 15.6$ Hz, 1H), 6.74-6.72 (m, 1H), 4.29 (q, $J = 7.2$ Hz, 2H), 1.30 (t, $J = 7.2$ Hz, 3H).

**(E)-Ethyl 2-oxo-4-(thiophen-2-yl)but-3-enoate (1q)**

$^1$H NMR (300 MHz, DMSO-d$_6$): 7.96 (s, 1H), 7.89 (d, $J = 4.5$ Hz, 1H), 7.73 (d, $J = 3.0$ Hz, 1H), 7.25-7.21 (m, 1H), 7.00 (d, $J = 15.9$ Hz, 1H), 4.30 (q, $J = 7.2$ Hz, 2H), 1.30 (t, $J = 7.2$ Hz, 3H).
4. General procedure for the synthesis of the enone

A mixture of benzaldehyde S (1 mmol) in acetone (10.0 mL) was treated with NaOH (10 mL, 1 M in water) at 0 °C dropwise. The solution was stirred at room temperature until no benzaldehyde was detected by TLC. The acetone was removed by vacuum distillation and then extracted by EtOAc (10 mL×2). The combined organic layer was dried over anhydrous Na₂SO₄, and concentrated to dryness, the residue was purified by flash chromatography on silica gel to give the product 1.

5. Analytical date of the enone

**(E)-4-Phenylbut-3-en-2-one (1m)**

Yellow solid (96%). ¹H NMR (300 MHz, CDCl₃): 7.62-7.11 (m, 5H), 6.33 (ddd, Ạ = 20.3, 15.8, 11.1 Hz, 2H), 2.26 (p, Ạ = 7.3 Hz, 2H), 1.12 (t, Ạ = 7.4 Hz, 3H).

**(E)-4-(p-Tolyl)but-3-en-2-one (1n)**

Yellow solid (100%). ¹H NMR (300 MHz, CDCl₃): 7.26 (d, Ạ = 8.1 Hz, 2H), 7.11 (d, Ạ = 8.1 Hz, 2H), 6.48-6.04 (m, 2H), 2.34 (s, 3H), 2.24 (p, Ạ = 7.3 Hz, 2H), 1.10 (t, Ạ = 7.4 Hz, 3H)

6. General procedure for the 1, 4-Hydrosilylation reaction

A mixture of α, β-enone ester (1.0 mmol), InCl₃ (0.10 mmol) and Et₃SiH (3.0 mmol, 0.48 mL) in toluene (2.0 mL) was treated with CF₃COOH (5.0 mmol, 0.38 mL) at room temperature dropwise. The solution was stirred at room temperature until no α, β-enone ester was detected by TLC. The solution was diluted with ethyl acetate (10 mL), NaHCO₃ (aq. 10 mL) and water (10 mL), the organic solution was separated and the aqueous layer was extracted by ethyl acetate (10 mL×2). The combined organic layer was dried over anhydrous Na₂SO₄, and concentrated to dryness, the residue was purified by flash chromatography on silica gel to give the product.

7. Analytical date of the silyl enol ether

**(Z)-Ethyl 4-(2-fluorophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2a).**
Yellow oil. $^1$H NMR (300 MHz, CDCl$_3$): 7.23-7.00 (m, 4H), 6.12 (t, $J = 7.5$ Hz, 1H), 4.21 (q, $J = 7.2$ Hz, 2H), 3.55 (d, $J = 7.5$ Hz, 2H), 1.30 (t, $J = 7.2$ Hz, 3H), 1.00 (t, $J = 8.1$ Hz, 9H), 0.75 (q, $J = 8.1$ Hz, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$): 164.7, 160.9 (d, $J = 183$ Hz), 141.6, 130.5 (d, $J = 3$ Hz), 127.9 (d, $J = 6$ Hz), 126.6 (d, $J = 12$ Hz), 124.1 (d, $J = 2$ Hz), 119.0, 115.2 (d, $J = 16.2$ Hz), 61.0, 25.0 (d, $J = 3$ Hz), 14.2, 6.7, 5.5. MS (ESI) $m/z$ 361.0 [M+Na]$^+$. HRMS (ESI) calcd. for C$_{18}$H$_{27}$FNaO$_3$Si 361.1611 found 361.1620. IR (cm$^{-1}$): 2957, 2912, 2877, 1723, 1646, 1586, 978, 754.

(Z)-Ethyl 4-(3-fluorophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2b)

White oil. $^1$H NMR (300 MHz, CDCl$_3$): 7.22-6.90 (m, 4H), 6.13 (t, $J = 7.5$ Hz, 1H), 4.21 (q, $J = 6.9$ Hz, 2H), 3.51 (d, $J = 7.5$ Hz, 2H), 1.30 (t, $J = 6.9$ Hz, 3H), 1.00 (t, $J = 7.8$ Hz, 9H), 0.75 (q, $J = 7.8$ Hz, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$): 164.7, 162.9 (d, $J = 167$ Hz), 142.3 (d, $J = 7$ Hz), 141.6, 129.9 (d, $J = 8$ Hz), 124.1 (d, $J = 3$ Hz), 119.5, 115.3 (d, $J = 21$ Hz), 113.6 (d, $J = 21$ Hz), 61.1, 31.4, 14.2, 6.8, 5.5. MS (ESI) $m/z$ 361.0 [M+Na]$^+$. HRMS (ESI) calcd. for C$_{18}$H$_{27}$FNaO$_3$Si 361.1611 found 361.1610. IR (cm$^{-1}$): 2957, 2912, 2877, 1723, 1645, 1077, 769, 742, 691.

(Z)-Ethyl 4-(4-fluorophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2c)

Yellow oil. $^1$H NMR (300 MHz, CDCl$_3$): 7.16 (dd, $J = 8.4$, 5.7 Hz, 2H), 6.97 (t, $J = 8.7$ Hz, 2H), 6.13 (t, $J = 7.5$ Hz, 1H), 4.21 (q, $J = 7.2$ Hz, 2H), 3.49 (d, $J = 6.3$ Hz, 1H), 1.30 (t, $J = 7.2$ Hz, 3H), 1.02 (t, $J = 7.8$ Hz, 9H), 0.75 (q, $J = 7.8$ Hz, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$): 164.8, 161.4 (d, $J = 243$ Hz), 141.2, 135.3 (d, $J = 3$ Hz), 128.9 (d, $J = 8$ Hz), 120.3, 115.2 (d, $J = 21$ Hz), 61.1, 31.0, 14.2, 6.8, 5.4. MS (ESI) $m/z$ 361.0 [M+Na]$^+$. HRMS (ESI) calcd. for C$_{18}$H$_{27}$FNaO$_3$Si 361.1611 found 361.1611. IR (cm$^{-1}$): 2957, 2912, 2877, 1723, 1645, 1077, 769, 742, 691.

(Z)-Ethyl 4-(2-chlorophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2d)

White oil. $^1$H NMR (300 MHz, CDCl$_3$): 7.37-7.17 (m, 4H), 6.14 (t, $J = 7.2$ Hz, 1H), 4.21 (q, $J = 6.9$ Hz, 2H), 3.63 (d, $J = 7.2$ Hz, 2H), 1.30 (t, $J = 6.9$ Hz, 3H), 1.00 (t, $J = 7.5$ Hz, 9H), 0.77 (q, $J = 7.5$ Hz, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$): 164.7, 141.8, 137.4, 134.0, 130.2, 129.4, 127.7, 126.9, 118.7, 61.1, 29.6, 14.2, 6.8, 5.5. MS (ESI) $m/z$ 377.0 [M+Na]$^+$. HRMS (ESI) calcd. for C$_{18}$H$_{27}$ClNaO$_3$Si 377.1316 found 377.1326. IR (cm$^{-1}$): 3426, 3067, 2956, 2876, 1723, 1371, 1051, 905, 745.

(Z)-Ethyl 4-(3-chlorophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2e)

Yellow oil. $^1$H NMR (300 MHz, CDCl$_3$): 7.22-7.07 (m, 4H), 6.11 (t, $J = 7.2$ Hz, 1H), 4.21 (q, $J = 6.9$ Hz, 2H), 3.49 (d, $J = 7.2$ Hz, 2H), 1.31 (t, $J = 7.2$ Hz, 3H), 1.00 (t, $J = 7.2$ Hz, 9H), 0.75 (q, $J = 7.2$ Hz, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$): 164.6, 141.7, 141.6, 134.2, 129.7, 128.6, 126.6,
126.4, 119.4, 61.1, 31.4, 14.2, 6.8, 5.5. MS (ESI) m/z 376.9 [M+Na]⁺. HRMS (ESI) calcd. for C₁₈H₂₇ClNaO₃Si 377.1316 found 377.1314. IR (cm⁻¹): 2957, 2911, 2877, 1724, 1644, 1371, 979, 766, 742, 68.

(Z)-Ethyl 4-(4-chlorophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2f)

Yellow oil. ¹H NMR (300 MHz, CDCl₃): 7.18 (dd, J = 37.8, 8.4 Hz, 4 H), 6.11 (t, J = 7.8 Hz, 1 H), 4.21 (q, J = 7.2 Hz, 2 H), 3.49 (d, J = 7.5 Hz, 2 H), 1.3 (t, J = 7.2 Hz, 3 H), 1.00 (t, J = 7.8 Hz, 9 H), 0.77 (q, J = 7.8 Hz, 6 H). ¹³C NMR (75 MHz, CDCl₃): 164.7, 141.5, 138.2, 131.9, 129.8, 128.6, 119.8, 61.1, 31.1, 14.2, 6.8, 5.5. MS (ESI) m/z 377.0 [M+Na]⁺ HRMS (ESI) calcd. for C₁₈H₂₇ClNaO₃Si 377.1316 found 377.1318. IR (cm⁻¹): 2957, 2911, 2877, 1723, 1645, 1371, 784, 763, 702.

(Z)-Ethyl 4-(2-bromophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2g)

Yellow oil. ¹H NMR (300 MHz, CDCl₃): 7.56-7.05 (m, 4H), 6.14 (t, J = 7.2 Hz, 1H), 4.21 (q, J = 6.9 Hz, 2 H), 3.64 (d, J = 7.2 Hz, 2 H), 1.30 (t, J = 6.9 Hz, 3H), 1.00 (t, J = 7.8 Hz, 9H), 0.75 (q, J = 7.8 Hz, 6H). ¹³C NMR (75 MHz, CDCl₃): 164.6, 141.7, 139.1, 132.7, 130.2, 127.9, 127.5, 124.5, 118.8, 61.1, 32.3, 14.2, 6.8, 5.5. MS (ESI) m/z 423.0 [M+Na]⁺ HRMS (ESI) calcd. for C₁₈H₂₇BrNaO₃Si 421.0811 found 421.0801. IR (cm⁻¹): 2957, 2911, 2877, 1723, 1644, 1250, 978, 786, 745, 704.

(Z)-Ethyl 4-(3-bromophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2h)

White oil. ¹H NMR (300 MHz, CDCl₃): 7.36-7.14 (m, 4H), 6.11 (t, J = 7.8 Hz, 1 H), 4.21 (q, J = 7.2 Hz, 2 H), 3.47 (d, J = 7.2 Hz, 2 H), 1.31 (t, J = 7.2 Hz, 3 H), 1.00 (t, J = 7.8 Hz, 9 H), 0.75 (q, J = 7.8 Hz, 6 H). ¹³C NMR (75 MHz, CDCl₃): 164.6, 142.1, 141.6, 131.5, 130.0, 129.3, 127.1, 122.5, 119.3, 61.1, 31.4, 14.2, 6.7, 5.5. MS (ESI) m/z 421.0 [M+Na]⁺ HRMS (ESI) calcd. for C₁₈H₂₇BrNaO₃Si 421.0811 found 421.0802. IR (cm⁻¹): 2956, 2910, 2877, 1723, 1644, 1392, 1017, 765, 742, 669.

(Z)-Ethyl 4-(4-bromophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2i)

Yellow oil. ¹H NMR (300 MHz, CDCl₃): 7.40 (d, J = 8.7 Hz, 2 H), 7.08 (d, J = 8.7 Hz, 2 H), 6.11 (t, J = 7.2 Hz, 3 H), 4.21 (q, J = 7.1 Hz, 2 H), 3.47 (d, J = 7.2 Hz, 2 H), 1.3 (t, J = 7.1 Hz, 3 H), 1.00 (t, J = 7.8 Hz, 9 H), 0.75 (q, J = 7.8 Hz, 6 H). ¹³C NMR (75 MHz, CDCl₃): 164.6, 141.5, 138.7, 131.5, 130.2, 119.9, 119.6, 61.1, 31.2, 14.2, 6.7, 5.5. MS (ESI) m/z 420.9 [M+Na]⁺ HRMS (ESI) calcd. for C₁₈H₂₇BrNaO₃Si 421.0811 found 421.0815. IR (cm⁻¹): 2956, 2911, 2876, 1723, 1645, 1371, 1250, 782, 742, 700.
(Z)-Ethyl 4-(4-methoxyphenyl)-2-((triethylsilyl)oxy)but-2-enoate (2j)

\[ \text{C}_{19}	ext{H}_{30}	ext{NaO}_4\text{Si} \]

\[ m/z 373.0 \ [\text{M+Na}]^+ \]

HRMS (ESI) calcd. for C\(_{19}\)H\(_{30}\)NaO\(_4\)Si 373.1811 found 373.1810.

IR (cm\(^{-1}\)): 2956, 2876, 2835, 1722, 1644, 1512, 1318, 1106, 978, 807.

(Z)-Ethyl 4-phenyl-2-((triethylsilyl)oxy)but-2-enoate (2k)

\[ \text{C}_{18}\text{H}_{28}\text{NaO}_3\text{Si} \]

\[ m/z 343.2 \ [\text{M+Na}]^+ \]

HRMS (ESI) calcd. for C\(_{18}\)H\(_{28}\)NaO\(_3\)Si 343.1700 found 343.1710.

IR (cm\(^{-1}\)): 2956, 2877, 1723, 1645, 1372, 1253, 1138, 742.

(Z)-Ethyl 4-(4-nitrophenyl)-2-((triethylsilyl)oxy)but-2-enoate (2l)

\[ \text{C}_{18}\text{H}_{27}\text{NNaO}_5\text{Si} \]

\[ m/z 388.0 \ [\text{M+Na}]^+ \]

HRMS (ESI) calcd. for C\(_{18}\)H\(_{27}\)NNaO\(_5\)Si 388.1551 found 388.1554.

IR (cm\(^{-1}\)): 2961, 1727, 1519, 1347, 1260, 1074, 1018.

(E)-But-1-en-1-ylbenzene (2m)

\[ \text{C}_{10}\text{H}_{10}\text{NaO}_2\text{Si} \]

\[ m/z 343.2 \ [\text{M+Na}]^+ \]

HRMS (ESI) calcd. for C\(_{10}\)H\(_{10}\)NaO\(_2\)Si 343.1700 found 343.1710.

IR (cm\(^{-1}\)): 2956, 2877, 1723, 1645, 1372, 1253, 1138, 742.

(E)-1-(But-1-en-1-yl)-4-methylbenzene (2n)

\[ \text{C}_{10}\text{H}_{12}\text{NaO}_2\text{Si} \]

\[ m/z 343.2 \ [\text{M+Na}]^+ \]

HRMS (ESI) calcd. for C\(_{10}\)H\(_{12}\)NaO\(_2\)Si 343.1700 found 343.1710.

IR (cm\(^{-1}\)): 2956, 2877, 1723, 1645, 1372, 1253, 1138, 742.

8. NMR spectra
HMBC for Compound 2i
HMBC for Compound 2i
HMBC for Compound 2i
ZWI-899-104-1C

\[
\text{O}_2\text{N} \quad \text{C} \quad \text{O}_2\text{Et}
\]