
Palladium(II)-Catalyzed Cross-Coupling of Simple Alkenes with Acrylates: A Direct Approach to 1,3-Dienes through C–H Activation

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General Methods

Commercial grade solvents and reagents were used without further purification. Experiments involving moisture and/or air sensitive components were performed in oven-dried glassware under a positive pressure of nitrogen using freshly distilled solvents. Hexane, ethyl acetate were fractionally distilled. Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 pre-coated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible by staining with basic solution of potassium permanganate or acidic solution of ceric molybdate. Flash chromatography was performed using Merck silica gel 60 with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use. Melting points were uncorrected and were recorded on a Buchi B- 54 melting point apparatus. IR spectra were recorded as thin films on NaCl plates on a Bio-Rad FTS 165 FTIR spectrometer and are reported in frequency of

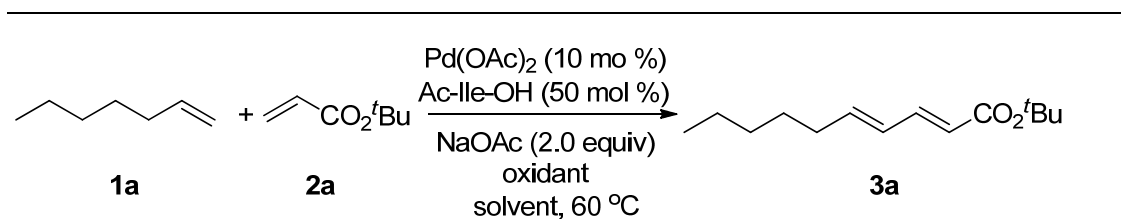
absorption (cm^{-1}). High resolution mass spectral analysis (HRMS) was performed on Waters Q-ToF Premier Mass Spectrometer. Proton nuclear magnetic resonance spectra (^1H NMR) were recorded on a Bruker Advance 300, 400 and 500 NMR spectrometer. Chemical shifts for ^1H NMR spectra are reported as δ in units of parts per million (ppm) downfield from SiMe_4 (δ 0.0) and relative to the signal of chloroform-*d* (δ 7.2600, singlet). Multiplicities were given as: s (singlet); d (doublet); t (triplet); q (quartet); dd (doublets of doublet); dt (doublets of triplet); or m (multiplets). The number of protons (*n*) for a given resonance is indicated by *n*H. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from SiMe_4 (δ 0.0) and relative to the signal of chloroform-*d* (δ 77.0, triplet).

General Procedure for the Synthesis of Mono-*N*-protected Amino Acid

Commercially available amino acids ligands were purchased from Sigma Aldrich and Alfa Aesar.

Ac-Val-OH, Bz-Val-OH, Piv-Val-OH, Tf-Ile-OH, Ac-Gly-OH, Ac-Leu-OH, Ac- t Leu-OH, Ac-Ile-OH were prepared according to the literature¹.

Screening of the Solvents and Oxidants

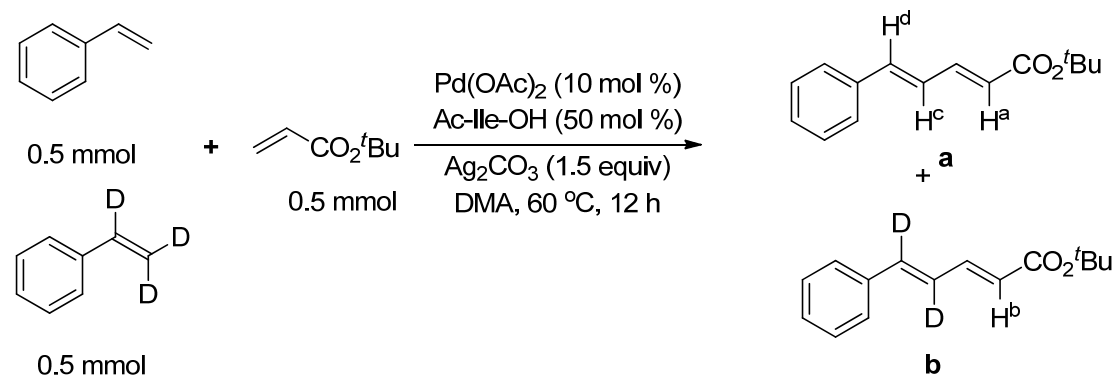


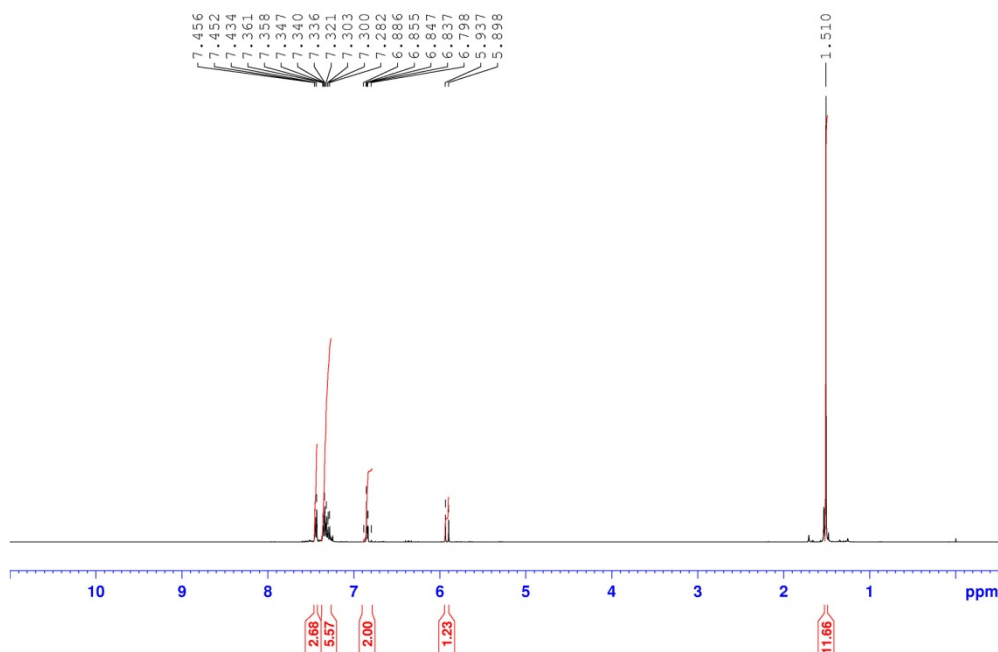
Entry	Oxidant (x equiv)	Solvent	Yield ^b (%)	<i>E/Z</i> ^c
1	Ag ₂ CO ₃ (1.5)	DMA	58	80:20
2	Ag ₂ O (1.5)	DMA	27	73:27
3	AgOAc (3.0)	DMA	18	81:19
4	Cu(OAc) ₂ (3.0)	DMA	11	75:25
5	BQ (1.5)	DMA	trace	-----
6	Ag ₂ CO ₃ (1.5)	DMF	44	79:21
7	Ag ₂ CO ₃ (1.5)	DMSO	39	67:33
8	Ag ₂ CO ₃ (1.5)	DCE	trace	-----
9	Ag ₂ CO ₃ (1.5)	NMP	42	77:23

^a Unless noted otherwise, the reactions were carried out on a 0.5 mmol scale of **2a** with 2 equiv of **1a** (1.0 mmol), 10 mol % of Pd(OAc)₂ (0.05 mmol), 50 mmol % ligand, NaOAc (1.0 mmol) and oxidant in DMA (1.0 mL) at 60 °C stirred for 24 h.

^b Isolated yield. ^c the ratio of *E/Z* was determined by crude ¹H NMR.

Competing Kinetic Isotope Effect (KIE) Experiment



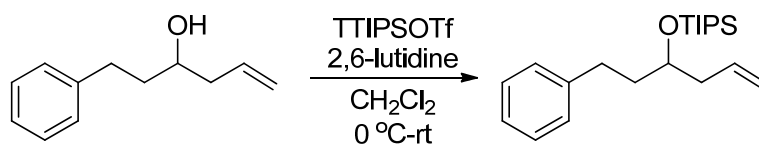


^1H NMR spectra of the mixture of compound **a** and **b**.

Note: The value of $k_{\text{H}}/k_{\text{D}}$ was calculated from the ^1H NMR spectra above which should be the mixture of compound **a** and **b** (the KIE scheme). The compound **a** was integrated as 1.00 (since the H^{c} and H^{d} in **a** with chemical shift 6.798-6.886 was integrated as 2.00). Therefore, the compound **b** was integrated as 0.23 (since the mixed H^{a} and H^{b} with chemical shift 5.918 (d, $J = 15.6$ Hz) was integrated as 1.23, so $1.23 - 1.00 = 0.23$). Therefore, $k_{\text{H}}/k_{\text{D}} = 1.00 / (1.23 - 1.00) = 4.3$.

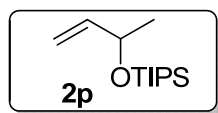
Synthesis of the Starting Materials (2n, 2p, 2t, 2q, 2o)

Triisopropyl((1-phenylhex-5-en-3-yl)oxy)silane 2n.



A solution of 1-phenylhex-5-en-3-ol (0.35 g, 2.0 mmol) in CH₂Cl₂ was added 2,6-lutidine (0.84 mL, 7.2 mmol) and TIPSOTf (0.81 mL, 3.0 mmol) at 0 °C. After stirring at 0 °C for 2 h, aq. NaHCO₃ was added followed by an extraction with ether. The combined organic extracts were washed with 1 M HCl and brine, dried and concentrated. The crude was purified by FC (silica gel, hexanes/EtOAc 20:1) to give the target product **2n** as colorless oil (0.59 g, 89%). ¹H NMR (400 MHz, CDCl₃) δ 7.29 – 7.25 (m, 2H), 7.18 – 7.15 (m, 3H), 5.91 – 5.80 (m, 1H), 5.10 – 5.05 (m, 2H), 3.97-3.92 (m, 1H), 2.67 (t, *J* = 8.4 Hz, 2H), 2.35 (t, *J* = 6.5 Hz, 2H), 1.90 – 1.72 (m, 2H), 1.11 – 1.06 (m, 21H); ¹³C NMR (100 MHz, CDCl₃) δ 142.70, 134.89, 128.33, 128.31, 125.63, 116.90, 71.48, 41.32, 38.29, 31.14, 18.19, 12.63; FTIR (NaCl, cm⁻¹): 3053.32, 2931.80, 2864.29, 2725.42, 1649.14, 1496.76, 1456.26, 1367.53, 1095.57, 1058.92, 972.12, 931.63, 742.59, 698.23; HRMS (ESI) *m/z* calculated for C₂₁H₃₇OSi [M+H]⁺: 333.2614, found 333.2618.

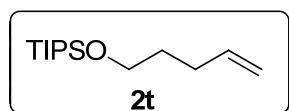
Triisopropyl(but-3-en-2-yloxy)silane (**2p**)



TIPS-protected allylic alcohol **2p** was prepared in 85% yield as colorless oil following the same procedure as **2n**. ¹H NMR (400 MHz, CDCl₃) δ 5.87 (ddd, *J* = 17.1, 10.4, 5.5 Hz, 1H), 5.17 (dt, *J* = 17.2, 1.6 Hz, 1H), 4.98 (dt, *J* = 10.4, 1.5 Hz, 1H), 4.42 – 4.35 (m, 1H), 1.25 (d, *J* = 6.3 Hz, 3H), 1.07 – 1.06 (m, 21H); ¹³C NMR (100 MHz, CDCl₃) δ 143.29, 112.27,

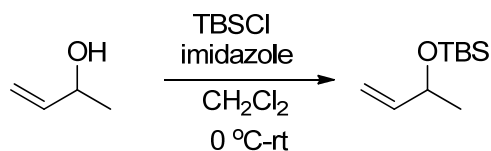
69.64, 24.68, 18.06, 12.33; FTIR (NaCl, cm^{-1}): 3053.32, 2960.73, 2866.22, 2725.42, 1641.42, 1463.97, 1456.26, 1367.53, 1153.43, 989.48, 920.05, 883.40; 680.87; HRMS (ESI) m/z calculated for $\text{C}_{13}\text{H}_{29}\text{OSi}$ $[\text{M}+\text{H}]^+$: 229.1988, found 229.1998.

Triisopropyl(pent-4-en-1-yloxy)silane (2t)



Alkenyl ether **2t** was prepared in 87% yield as colorless oil following the same procedure as **2n**. ^1H NMR (400 MHz, CDCl_3) δ 5.89 – 5.79 (m, 1H), 5.02 (ddd, $J = 17.1, 3.5, 1.7$ Hz, 1H), 4.95 (dd, $J = 10.2, 1.9$ Hz, 1H), 3.69 (t, $J = 6.5$ Hz, 2H), 2.14 – 2.12 (m, 2H), 1.66 – 1.62 (m, 2H), 1.10 – 1.02 (m, 21H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.68, 114.42, 62.75, 32.20, 30.07, 18.02, 12.01; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2943.37, 2864.29, 2725.42, 1641.42, 1463.97, 1382.96, 1367.53, 1105.21, 995.27, 908.47, 883.40, 736.81; HRMS (ESI) m/z calculated for $\text{C}_{14}\text{H}_{31}\text{OSi}$ $[\text{M}+\text{H}]^+$: 243.2144, found 243.2132.

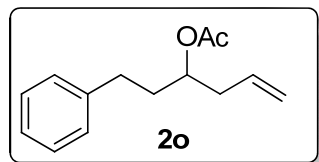
Synthesis of (but-3-en-2-yloxy)(tert-butyl)dimethylsilane 2q



A solution of the but-3-en-2-ol (0.26 mL, 3.0 mmol) in CH_2Cl_2 (10 mL) was added imidazole (0.41 g, 6.0 mmol) and TBSCl (0.54 g, 3.6 mmol) at 0 °C. After stirring at 0 °C for 2 h, aq. NaHCO_3 was added followed by an extraction with ether. The combined organic extracts were washed

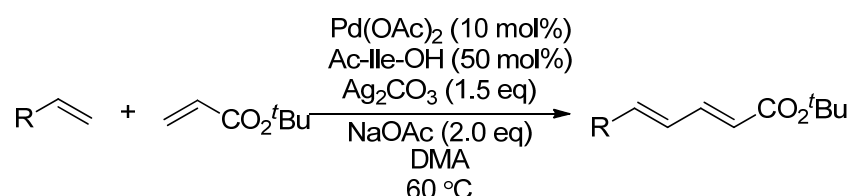
with brine, dried and concentrated. The crude was purified by FC (silica gel, hexanes/EtOAc 20:1) to give the target product **13** as colorless oil (0.45 g, 80%). ^1H NMR (400 MHz, CDCl_3) δ 5.85 (ddd, $J = 17.1, 10.4, 5.2$ Hz, 1H), 5.16 (dt, $J = 17.1, 1.7$ Hz, 1H), 4.98 (dt, $J = 10.4, 1.6$ Hz, 1H), 4.32 – 4.26 (m, 1H), 1.21 (d, $J = 6.4$ Hz, 3H), 0.90 (s, 9H), 0.06 (d, $J = 2.6$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.86, 112.39, 69.53, 25.88, 24.24, 18.30, -4.68, -4.83; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2931.80, 2864.29, 2725.42, 1421.54, 1265.30, 1051.20, 894.97, 742.59; HRMS (ESI) m/z calculated for $\text{C}_{10}\text{H}_{23}\text{OSi}$ $[\text{M}+\text{H}]^+$: 187.1518, found 187.1516.

1-phenylhex-5-en-3-yl acetate (**2o**)



This is a known compound and prepared according to the literature².

General Procedure for the Palladium-Catalyzed Cross-Coupling of Simple Alkenes with Tert-butyl acrylate.

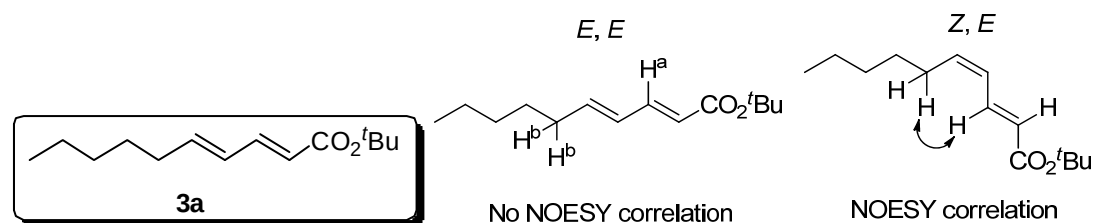


A 5 mL round bottomed flask equipped with a magnetic stirring bar was added with substitute alkene (1.0 mmol, 2.0 equiv), $\text{Pd}(\text{OAc})_2$ (10 mol%,

0.05 mmol), Ac-Ile-OH (50 mol%, 0.25 mmol), Ag₂CO₃ (0.75 mmol, 1.5 equiv), NaOAc (1.0 mmol, 2.0 equiv), *tert*-butyl acrylate (0.5 mmol, 1.0 equiv) in 1.0 mL DMA. The flask was stirred at 60 °C for 48 h. The reaction mixture was cooled to room temperature, followed by the addition of water and ethyl acetate. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3×10 mL). The combined organic layer was washed with brine and dried over anhydrous Na₂SO₄, filtrated and concentrated *in vacuo*, the residue was purified through column chromatography on silica gel to give the desired products.

Characterization Data for the Products

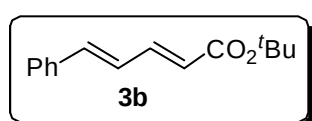
(2*E*,4*E*)-*tert*-butyl deca-2,4-dienoate (3a)



This compound was prepared by the general procedure described above and was obtained as a colorless oil of inseparable mixture (*E/Z* = 80:20) in 78% yield, the stereochemistry was deduced by NOESY correlation analysis. ¹H and ¹³C NMR are described for the *E* isomer: *R_f* = 0.68 (hexane : ethyl acetate = 10:1); ¹H NMR (400 MHz, CDCl₃) δ 7.14 (dd, *J* = 15.3, 10.0 Hz, 1H), 6.16 – 6.04 (m, 2H), 5.69 (d, *J* = 15.4 Hz, 1H), 2.13 (dd, *J* = 13.8, 7.0 Hz, 2H), 1.47 (s, 9H), 1.44 – 1.37 (m, 2H), 1.32-1.25

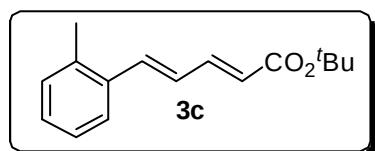
(m, 4H), 0.87 (t, $J = 6.7$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.64, 144.01, 143.94, 128.32, 121.07, 79.93, 32.88, 31.31, 28.40, 28.14, 22.42, 13.95; FTIR (NaCl, cm^{-1}): 3053.32, 2980.02, 2929.87, 2864.29, 1703.14, 1641.42, 1367.53, 1265.30, 1157.29, 1138.00, 734.88, 705.95; HRMS (ESI) m/z calculated for $\text{C}_{14}\text{H}_{25}\text{O}_2$ $[\text{M}+\text{H}]^+$:225.1855, found 225.1857.

(2E,4E)-tert-butyl 5-phenylpenta-2,4-dienoate (3b)



This compound was prepared by the general procedure described above and was obtained as a colorless oil in 80% yield with E/Z >99:1. ^1H NMR (400 MHz, CDCl_3) δ 7.44 (d, $J = 7.4$ Hz, 2H), 7.39 – 7.28 (m, 4H), 6.88-6.79 (m, 2H), 5.92 (d, $J = 15.3$ Hz, 1H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.31, 143.44, 139.64, 136.12, 128.80, 128.71, 127.04, 126.30, 123.32, 80.19, 28.14; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2929.87, 1701.22, 1625.99, 1392.61, 1367.53, 1346.31, 1265.30, 1157.29, 1132.21, 999.13, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{15}\text{H}_{19}\text{O}_2$ $[\text{M}+\text{H}]^+$:231.1385, found 231.1349.

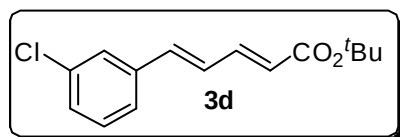
(2E,4E)-tert-butyl 5-(o-tolyl)penta-2,4-dienoate (3c)



This compound was prepared by the general procedure described above and was obtained as a pale yellow oil in 66% yield, ^1H NMR (400 MHz, CDCl_3) δ 7.54 – 7.49 (m, 1H), 7.37 (ddd, $J = 15.3, 11.1, 0.6$ Hz, 1H), 7.21 – 7.14 (m, 3H), 7.10 (d, $J = 15.5$ Hz, 1H), 6.80 – 6.72 (m,

1H), 5.92 (d, $J = 15.3$ Hz, 1H), 2.37 (s, 3H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.36, 143.75, 137.31, 136.36, 135.00, 130.59, 128.65, 127.38, 126.20, 125.50, 123.21, 80.20, 28.15, 19.69; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2929.87, 1701.22, 1618.28, 1458.18, 1392.61, 1265.30, 1163.08, 999.13, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{21}\text{O}_2$ $[\text{M}+\text{H}]^+$:245.1542, found 245.1540.

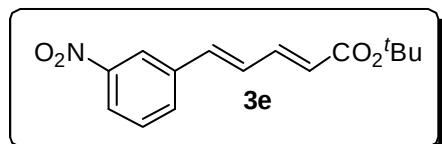
(2E,4E)-tert-butyl 5-(3-chlorophenyl)penta-2,4-dienoate (3d)



This compound was prepared by the general procedure described above and was

obtained as a pale yellow solid in 52% yield. mp 66-68 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.43 (s, 1H), 7.36 – 7.22 (m, 4H), 6.87 – 6.71 (m, 2H), 5.94 (d, $J = 15.3$ Hz, 1H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.13, 142.81, 138.00, 137.88, 134.75, 129.95, 128.65, 127.66, 126.78, 125.26, 124.38, 80.43, 28.15; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2929.87, 1701.22, 1627.92, 1475.54, 1367.53, 1265.30, 1161.15, 1134.14, 999.13, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{15}\text{H}_{18}\text{O}_2\text{Cl}$ $[\text{M}+\text{H}]^+$:265.0995, found 265.0991.

(2E,4E)-tert-butyl 5-(3-nitrophenyl)penta-2,4-dienoate (3e)

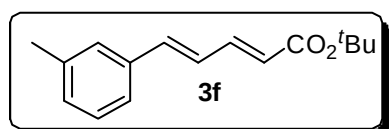


This compound was prepared by the general procedure described above and

was obtained as a pale yellow solid in 51% yield. mp 93-94 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.30 (t, $J = 1.9$ Hz, 1H), 8.13 (ddd, $J = 8.2, 2.2, 0.9$

Hz, 1H), 7.74 (d, $J = 7.8$ Hz, 1H), 7.53 (t, $J = 8.0$ Hz, 1H), 7.34 (dd, $J = 15.3, 10.2$ Hz, 1H), 6.96 – 6.87 (m, 2H), 6.02 (d, $J = 15.3$ Hz, 1H), 1.52 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.89, 148.66, 142.11, 137.91, 136.45, 132.64, 129.70, 129.17, 125.56, 123.05, 121.34, 80.63, 28.11; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 1701.22, 1629.85, 1529.55, 1352.10, 1249.87, 1161.15, 1134.14, 999.13, 823.60, 736.81; HRMS (ESI) m/z calculated for $\text{C}_{15}\text{H}_{18}\text{O}_4\text{N}$ $[\text{M}+\text{H}]^+$: 276.1236, found 276.1243.

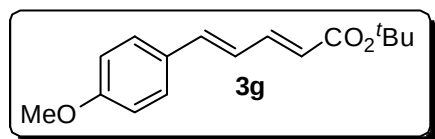
((2E,4E)-tert-butyl 5-(m-tolyl)penta-2,4-dienoate (3f))



This compound was prepared by the general procedure described above and was obtained

as a pale yellow oil in 59% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.37 – 7.30 (m, 1H), 7.26 – 7.22 (m, 3H), 7.11 – 7.09 (m, 1H), 6.86 – 6.79 (m, 2H), 5.91 (d, $J = 15.2$ Hz, 1H), 2.35 (s, 3H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.39, 143.59, 139.87, 138.31, 136.09, 129.67, 128.62, 127.74, 126.13, 124.27, 123.11, 80.19, 28.16, 21.31; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2929.87, 1697.36, 1625.99, 1456.26, 1392.61, 1367.53, 1336.67, 1265.30, 1159.22, 1132.21, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{21}\text{O}_2$ $[\text{M}+\text{H}]^+$: 245.1542, found 245.1540.

(2E,4E)-tert-butyl 5-(4-methoxyphenyl)penta-2,4-dienoate (3g)

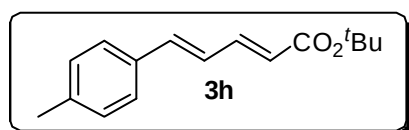


This compound was prepared by the general procedure described above and

was obtained as a pale yellow solid in 60% yield. mp 58-59 °C; ^1H NMR

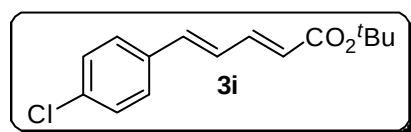
(400 MHz, CDCl_3) δ 7.41 – 7.36 (m, 2H), 7.33 (dd, $J = 15.2, 10.6$ Hz, 1H), 6.89 – 6.85 (m, 2H), 6.83 – 6.68 (m, 2H), 5.86 (d, $J = 15.2$ Hz, 1H), 3.81 (s, 3H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.54, 160.23, 143.90, 139.39, 128.95, 128.49, 124.23, 122.06, 114.19, 80.04, 55.27, 28.16; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 1697.36, 1625.99, 1600.92, 1510.26, 1348.24, 1265.30, 1163.08, 1130.29, 999.13, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{21}\text{O}_3$ $[\text{M}+\text{H}]^+$: 261.1491, found 261.1484.

(2E,4E)-tert-butyl 5-(p-tolyl)penta-2,4-dienoate (3h)



This compound was prepared by the general procedure described above and was obtained as a pale yellow oil in 86% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.36 – 7.30 (m, 3H), 7.14 (d, $J = 8.0$ Hz, 2H), 6.85 – 6.75 (m, 2H), 5.89 (d, $J = 15.2$ Hz, 1H), 2.34 (s, 3H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.41, 143.70, 139.71, 138.96, 133.39, 129.44, 127.00, 125.36, 122.70, 80.09, 28.14, 21.28; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2929.87, 1701.22, 1625.99, 1510.26, 1392.61, 1367.53, 1340.53, 1247.94, 1161.15, 999.13, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{21}\text{O}_2$ $[\text{M}+\text{H}]^+$: 245.1542, found 245.1536.

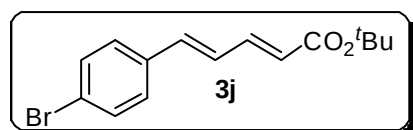
(2E,4E)-tert-butyl 5-(4-chlorophenyl)penta-2,4-dienoate (3i)



This compound was prepared by the general procedure described above and was

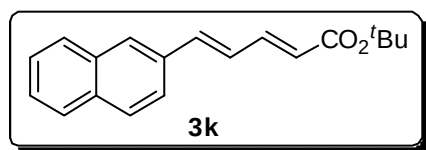
obtained as a pale yellow solid in 61% yield. mp 84-86 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.38 – 7.28 (m, 5H), 6.81 – 6.79 (m, 2H), 5.93 (d, J = 15.2 Hz, 1H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.20, 143.02, 138.11, 134.64, 134.49, 128.94, 128.18, 126.86, 123.87, 80.34, 28.14; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 1701.22, 1625.99, 1589.34, 1490.97, 1367.53, 1265.30, 1161.15, 1132.21, 1089.78, 999.13, 850.61, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{15}\text{H}_{18}\text{O}_2\text{Cl}$ $[\text{M}+\text{H}]^+$: 265.0995, found 265.0994.

(2E,4E)-tert-butyl 5-(4-bromophenyl)penta-2,4-dienoate (3j)



This compound was prepared by the general procedure described above and was obtained as a white solid in 45% yield. mp 89-90°C; ^1H NMR (400 MHz, CDCl_3) δ 7.47 (d, J = 8.5 Hz, 2H), 7.34 – 7.26 (m, 3H), 6.86 – 6.76 (m, 2H), 5.93 (d, J = 15.3 Hz, 1H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.23, 143.02, 138.19, 135.09, 131.92, 128.46, 126.99, 123.99, 122.77, 80.39, 28.16; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2929.87, 1701.22, 1625.99, 1490.97, 1392.61, 1367.53, 1340.53, 1265.30, 1247.94, 1163.80, 1130.29, 997.20, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{15}\text{H}_{18}\text{O}_2\text{Br}$ $[\text{M}+\text{H}]^+$: 309.0490, found 309.0490.

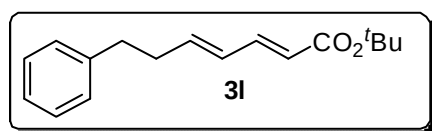
(2E,4E)-tert-butyl 5-(naphthalen-2-yl)penta-2,4-dienoate (3k)



This compound was prepared by the general procedure described above and was

obtained as a yellow solid in 53% yield. mp 60-61 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.84 – 7.80 (m, 4H), 7.64 (dd, J = 8.6, 1.5 Hz, 1H), 7.50 – 7.45 (m, 2H), 7.42 (dd, J = 15.2, 10.0 Hz, 1H), 7.05 – 6.93 (m, 2H), 5.98 (d, J = 15.2 Hz, 1H), 1.54 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.38, 143.51, 139.76, 133.64, 133.51, 133.44, 128.42, 128.19, 127.93, 127.68, 126.62, 126.53, 126.50, 123.35, 123.34, 80.25, 28.17; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2929.87, 1697.36, 1618.28, 1510.26, 1475.54, 1392.61, 1367.53, 1325.10, 1265.30, 1163.08, 1132.21, 999.13, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{19}\text{H}_{21}\text{O}_2$ $[\text{M}+\text{H}]^+$: 281.1542, found 281.1548.

(2E,4E)-tert-butyl 7-phenylhepta-2,4-dienoate (3I)

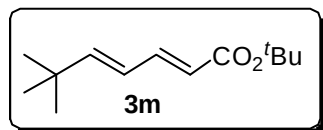


This compound was prepared by the general procedure described above and

was obtained as a colorless oil of inseparable mixture (E/Z = 77:23) in 47% yield, ^1H and ^{13}C NMR are described for the E isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.30-7.26 (m, 2H), 7.21 – 7.13 (m, 4H), 6.15 – 6.07 (m, 2H), 5.71 (d, J = 15.4 Hz, 1H), 2.76 – 2.72 (m, 2H), 2.48 (dd, J = 14.6, 6.6 Hz, 2H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.57, 143.70, 142.35, 141.13, 128.94, 128.41, 128.37, 126.01, 121.62, 80.06, 35.13, 34.64, 28.16; FTIR (NaCl, cm^{-1}): 3053.32, 2978.09, 2929.87, 1703.14, 1643.35, 1616.35, 1456.26, 1367.53, 1305.81, 1255.66, 1147.65, 1128.36,

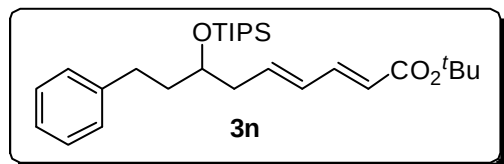
1001.06, 738.74, 700.16; HRMS (ESI) m/z calculated for $C_{17}H_{23}O_2$ $[M+H]^+$: 259.1698, found 259.1702.

(2*E*,4*E*)-tert-butyl 6,6-dimethylhepta-2,4-dienoate (3m)



This compound was prepared by the general procedure described above and was obtained as a colorless oil in 76% yield. 1H NMR (400 MHz, $CDCl_3$) δ 7.14 (ddd, J = 15.4, 8.2, 1.6 Hz, 1H), 6.10 – 6.01 (m, 2H), 5.72 (d, J = 15.3 Hz, 1H), 1.46 (s, 9H), 1.04 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.53, 154.19, 144.45, 123.28, 121.39, 79.88, 33.73, 29.06, 28.14; FTIR (NaCl, cm^{-1}): 3053.32, 2962.66, 2868.15, 1697.36, 1637.56, 1625.99, 1475.54, 1392.61, 1367.53, 1332.81, 1265.30, 1249.87, 1155.36, 1130.29, 1002.98, 740.67, 705.95; HRMS (ESI) m/z calculated for $C_{13}H_{23}O_2$ $[M+H]^+$: 211.1698, found 211.1706.

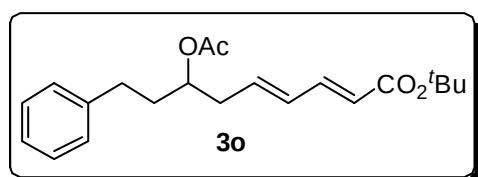
(2*E*,4*E*)-tert-butyl 9-phenyl-7-((triisopropylsilyl)oxy)nona-2,4-dienoate (3n)



This compound was prepared by the general procedure described above and was obtained as a colorless oil of inseparable mixture (E/Z = 80:20) in 69% yield, 1H and ^{13}C NMR are described for the *E* isomer: 1H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.25 (m, 2H), 7.20 – 7.13 (m, 4H), 6.22 – 6.10 (m, 2H), 5.73 (d, J = 15.4 Hz, 1H),

3.98 (dt, $J = 11.3, 5.7$ Hz, 1H), 2.67 – 2.63 (m, 2H), 2.45 – 2.43 (m, 2H), 1.83 – 1.76 (m, 2H), 1.49 (s, 9H), 1.09-1.06 (m, 21H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.58, 143.63, 142.27, 139.30, 130.58, 128.36, 128.29, 125.74, 121.69, 80.09, 71.31, 40.35, 38.58, 31.27, 28.16, 18.16, 12.58; FTIR (NaCl, cm^{-1}): 3053.32, 2943.37, 2866.22, 1701.22, 1697.36, 1643.35, 1641.42, 1616.35, 1456.26, 1367.53, 1149.57, 999.13, 908.47, 883.40, 732.95; HRMS (ESI) m/z calculated for $\text{C}_{28}\text{H}_{47}\text{O}_3\text{Si}$ $[\text{M}+\text{H}]^+$: 459.3295, found 459.3294.

(2E,4E)-tert-butyl 7-acetoxy-9-phenylnona-2,4-dienoate (3o)

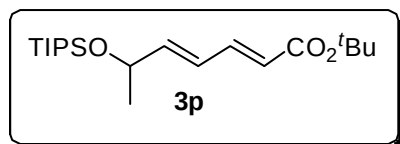


This compound was prepared by the general procedure described above and was obtained as a colorless oil of

inseparable mixture ($E/Z = 80:20$) in 41% yield, ^1H and ^{13}C NMR are described for the E isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.29-7.26 (m, 2H), 7.20 – 7.12 (m, 4H), 6.17 (dd, $J = 15.4, 11.0$ Hz, 1H), 6.02-5.95 (m, 1H), 5.73 (d, $J = 15.3$ Hz, 1H), 4.99 – 4.96 (m, 1H), 2.67 – 2.57 (m, 3H), 2.46 – 2.41 (m, 1H), 2.02 (s, 3H), 1.92 – 1.82 (m, 2H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.56, 166.31, 143.03, 141.17, 137.21, 131.21, 128.40, 128.24, 125.96, 122.39, 80.16, 72.46, 37.69, 35.34, 31.71, 28.09, 21.04; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2981.95, 1732.08, 1714.72, 1701.22, 1643.35, 1369.46, 1367.53, 1265.30, 1153.43, 1134.14,

1029.99, 1001.06, 734.88, 704.02; HRMS (ESI) m/z calculated for $C_{21}H_{29}O_4$ $[M+H]^+$: 345.2066, found 345.2054.

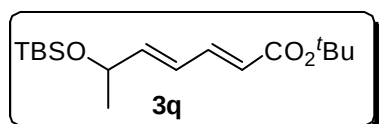
(2*E*,4*E*)-tert-butyl 6-((triisopropylsilyl)oxy)hepta-2,4-dienoate (3p)



This compound was prepared by the general procedure described above and was obtained

as a colorless oil of inseparable mixture ($E/Z = 87:13$) in 47% yield, 1H and ^{13}C NMR are described for the *E* isomer: 1H NMR (400 MHz, $CDCl_3$) δ 7.17 (dd, $J = 15.3, 11.1$ Hz, 1H), 6.36 – 6.22 (m, 1H), 6.09 (dd, $J = 15.2, 5.3$ Hz, 1H), 5.78 (d, $J = 15.3$ Hz, 1H), 4.49 (t, $J = 6.3$ Hz, 1H), 1.48 (s, 9H), 1.26 (d, $J = 6.4$ Hz, 3H), 1.07 – 1.03 (m, 21H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.43, 146.72, 143.22, 125.75, 122.76, 80.13, 28.15, 24.46, 18.01, 12.27; FTIR (NaCl, cm^{-1}): 3053.32, 2931.80, 2864.29, 1714.72, 1699.29, 1649.14, 1618.28, 1456.26, 1367.53, 1265.30, 1134.14, 1089.78, 999.13, 894.97, 742.59, 705.95; HRMS (ESI) m/z calculated for $C_{20}H_{39}O_3Si$ $[M+H]^+$: 355.2668, found 355.2671.

(2*E*,4*E*)-tert-butyl 6-((tert-butyldimethylsilyl)oxy)hepta-2,4-dienoate (3q)

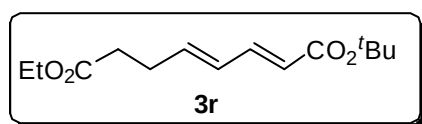


This compound was prepared by the general procedure described above and was obtained

as a colorless oil of inseparable mixture ($E/Z = 85:15$) in 54% yield, 1H and ^{13}C NMR are described for the *E* isomer: 1H NMR (400 MHz, $CDCl_3$)

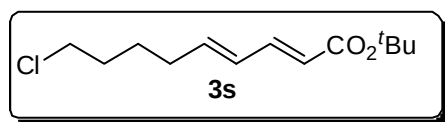
δ 7.16 (dd, $J = 15.3, 11.1$ Hz, 1H), 6.31 – 6.25 (m, 1H), 6.06 (dd, $J = 15.2, 5.0$ Hz, 1H), 5.79 (d, $J = 15.3$ Hz, 1H), 4.42 – 4.36 (m, 1H), 1.48 (s, 9H), 1.23 (d, $J = 6.5$ Hz, 3H), 0.89 (s, 9H), 0.05 (d, $J = 4.5$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.41, 146.28, 143.16, 125.80, 122.82, 80.13, 28.15, 25.81, 24.05, -4.78, -4.83; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 1978.09, 2929.87, 2856.58, 1714.72, 1699.29, 1697.36, 1645.28, 1618.28, 1367.53, 1265.30, 1134.14, 1001.60, 835.18, 740.67; HRMS (ESI) m/z calculated for $\text{C}_{17}\text{H}_{33}\text{O}_3\text{Si}$ $[\text{M}+\text{H}]^+$: 313.2199, found 313.2190.

(2*E*,4*E*)-1-tert-butyl 8-ethyl octa-2,4-dienedioate (3r)



This compound was prepared by the general procedure described above and was obtained as a colorless oil of inseparable mixture ($E/Z = 75:25$) in 54% yield, ^1H and ^{13}C NMR are described for the *E* isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.11 (dd, $J = 15.4, 10.7$ Hz, 1H), 6.20 – 6.01 (m, 2H), 5.72 (d, $J = 15.4$ Hz, 1H), 4.14 – 4.09 (m, 2H), 2.45 – 2.39 (m, 4H), 1.46 (s, 9H), 1.23 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 172.54, 166.43, 143.33, 140.71, 129.33, 122.16, 80.13, 60.48, 33.34, 28.15, 28.08, 14.22; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2981.95, 2933.73, 1730.15, 1701.22, 1643.35, 1618.28, 1456.26, 1392.61, 1367.53, 1265.30, 1151.50, 1001.06, 738.74; HRMS (ESI) m/z calculated for $\text{C}_{14}\text{H}_{23}\text{O}_4$ $[\text{M}+\text{H}]^+$: 255.1596, found 255.1598.

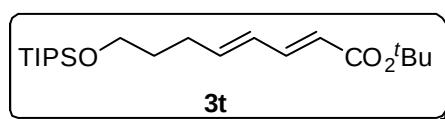
(2*E*,4*E*)-tert-butyl 9-chloronona-2,4-dienoate (3s)



This compound was prepared by the general procedure described above and

was obtained as a colorless oil inseparable mixture ($E/Z = 81:19$) in 37% yield, ^1H and ^{13}C NMR are described for the E isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.14 (dd, $J = 15.3, 10.6$ Hz, 1H), 6.19 – 6.01 (m, 2H), 5.72 (d, $J = 15.4$ Hz, 1H), 3.53 (td, $J = 6.6, 1.7$ Hz, 2H), 2.19 (q, $J = 7.1$ Hz, 2H), 1.81 – 1.74 (m, 2H), 1.62 – 1.55 (m, 2H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.53, 143.61, 142.51, 128.94, 121.64, 80.08, 44.69, 32.07, 31.93, 28.15, 25.94; FTIR (NaCl, cm^{-1}): 3053.32, 2983.88, 2929.87, 2852.72, 1699.29, 1643.35, 1616.35, 1456.26, 1367.53, 1265.30, 1141.86, 1001.06, 738.74, 704.02; HRMS (ESI) m/z calculated for $\text{C}_{13}\text{H}_{22}\text{O}_2\text{Cl}$ $[\text{M}+\text{H}]^+$: 245.1308, found 245.1303.

(2E,4E)-tert-butyl 8-((triisopropylsilyl)oxy)octa-2,4-dienoate (3t)

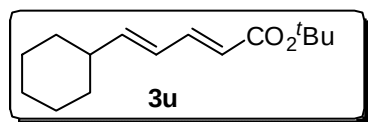


This compound was prepared by the general procedure described above and

was obtained as a colorless oil inseparable mixture ($E/Z = 80:20$) in 67% yield, ^1H and ^{13}C NMR are described for the E isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.15 (dd, $J = 15.4, 9.9$ Hz, 1H), 6.19 – 6.06 (m, 2H), 5.70 (d, $J = 15.4$ Hz, 1H), 3.71 – 3.67 (m, 2H), 2.31 – 2.22 (m, 2H), 1.69 – 1.63 (m, 2H), 1.48 (s, 9H), 1.07 – 1.03 (m, 21H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.64, 143.94, 143.39, 128.59, 121.18, 79.97, 62.47, 31.99, 29.34, 28.16, 17.99, 11.96; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2943.37,

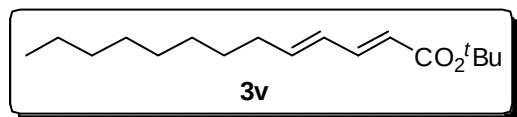
2866.22, 1714.72, 1699.29, 1643.35, 1616.35, 1367.53, 1253.73, 1157.29, 1109.07, 999.13, 738.74, 680.87; HRMS (ESI) m/z calculated for $C_{21}H_{41}O_3Si$ $[M+H]^+$: 369.2825, found 369.2831.

(2*E*,4*E*)-tert-butyl 5-cyclohexylpenta-2,4-dienoate (3u)



This compound was prepared by the general procedure described above and was obtained as a colorless oil inseparable mixture ($E/Z = 82:18$) in 72% yield, 1H and ^{13}C NMR are described for the *E* isomer: 1H NMR (400 MHz, $CDCl_3$) δ 7.14 (dd, $J = 15.3, 10.3$ Hz, 1H), 6.13 – 5.98 (m, 2H), 5.71 (d, $J = 15.3$ Hz, 1H), 2.10 – 2.01 (m, 1H), 1.74 – 1.63 (m, 5H), 1.47 (s, 9H), 1.32 – 1.05 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.63, 149.29, 144.38, 125.83, 121.22, 79.93, 41.03, 32.30, 28.16, 25.99, 25.79; FTIR (NaCl, cm^{-1}): 3053.32, 2983.88, 2929.87, 2852.72, 1699.29, 1639.49, 1616.53, 1456.26, 1367.53, 1265.30, 1161.15, 1139.93, 1001.06, 908.47, 738.74; HRMS (ESI) m/z calculated for $C_{15}H_{25}O_2$ $[M+H]^+$: 237.1855, found 237.1848.

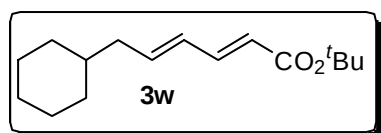
(2*E*,4*E*)-tert-butyl trideca-2,4-dienoate (3v)



This compound was prepared by the general procedure described above and was obtained as a colorless oil inseparable mixture ($E/Z = 80:20$) in 75% yield, 1H and ^{13}C NMR are described for the *E* isomer: 1H NMR (400 MHz, $CDCl_3$) δ 7.15 (dd, $J = 15.3, 10.0$ Hz, 1H), 6.16 – 6.03 (m, 2H), 5.70 (d, $J = 15.4$ Hz, 1H), 2.14 (dd, $J = 13.7, 7.0$ Hz, 2H), 1.48 (s,

9H), 1.29-1.26 (m, 12H), 0.87 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.67, 144.05, 143.99, 128.33, 121.08, 79.96, 32.95, 31.83, 29.38, 29.21, 29.15, 28.76, 28.17, 22.64, 14.07; FTIR (NaCl, cm^{-1}): 3053.32, 2956.87, 2927.94, 2854.65, 1701.22, 1641.42, 1616.35, 1456.26, 1367.53, 1265.30, 1136.07, 1001.06, 740.67; HRMS (ESI) m/z calculated for $\text{C}_{17}\text{H}_{31}\text{O}_2$ $[\text{M}+\text{H}]^+$: 267.2324, found 267.2321.

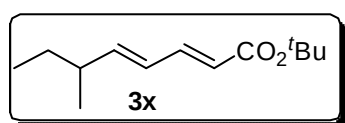
(2*E*,4*E*)-tert-butyl 6-cyclohexylhexa-2,4-dienoate (3w)



This compound was prepared by the general procedure described above and was obtained

as a colorless oil inseparable mixture ($E/Z = 85:15$) in 71% yield, ^1H and ^{13}C NMR are described for the *E* isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.15 (dd, $J = 15.3, 9.9$ Hz, 1H), 6.15 – 6.02 (m, 2H), 5.70 (d, $J = 15.4$ Hz, 1H), 2.04 (t, $J = 6.5$ Hz, 2H), 1.69 – 1.62 (m, 5H), 1.47 (s, 9H), 1.37 – 1.32 (m, 1H), 1.25 – 1.10 (m, 3H), 0.94 – 0.85 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.67, 143.94, 142.65, 129.34, 121.06, 79.96, 40.92, 37.83, 33.10, 28.16, 26.41, 26.23; FTIR (NaCl, cm^{-1}): 3053.32, 2983.88, 2929.87, 2852.72, 1697.36, 1639.49, 1616.35, 1456.26, 1367.53, 1317.38, 1265.30, 1139.93, 1001.06, 908.47, 734.88; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{27}\text{O}_2$ $[\text{M}+\text{H}]^+$: 251.2011, found 251.2014.

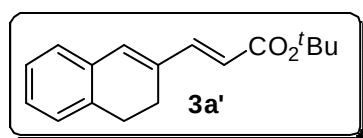
(2*E*,4*E*)-tert-butyl 6-methylocta-2,4-dienoate (3x)



This compound was prepared by the general procedure described above and was obtained as

a colorless oil inseparable mixture (*E/Z* = 86:14) in 81% yield, ^1H and ^{13}C NMR are described for the *E* isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.14 (dd, J = 15.3, 10.8 Hz, 1H), 6.09 (dd, J = 15.2, 10.8 Hz, 1H), 5.94 (dd, J = 15.2, 7.8 Hz, 1H), 5.71 (d, J = 15.4 Hz, 1H), 2.13 (dt, J = 13.8, 6.9 Hz, 1H), 1.47 (s, 9H), 1.38 - 1.30 (m, 2H), 1.00 (d, J = 6.7 Hz, 3H), 0.84 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.60, 149.32, 144.14, 126.73, 121.21, 79.91, 38.74, 29.31, 28.14, 19.54, 11.62; FTIR (NaCl, cm^{-1}): 3053.32, 2966.52, 2929.87, 1699.29, 1639.49, 1616.35, 1456.26, 1367.53, 1309.67, 1265.30, 1170.79, 1139.93, 1001.06, 908.47, 738.74, 705.95; HRMS (ESI) m/z calculated for $\text{C}_{13}\text{H}_{23}\text{O}_2$ $[\text{M}+\text{H}]^+$: 211.1698, found 211.1703.

(*E*)-tert-butyl 3-(3,4-dihydronaphthalen-2-yl)acrylate (3a')

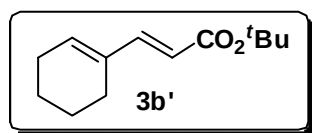


This compound was prepared by the general procedure described above and was obtained as

a colorless oil in 66% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.39 (d, J = 15.7 Hz, 1H), 7.18 – 7.10 (m, 4H), 6.72 (s, 1H), 5.92 (d, J = 15.7 Hz, 1H), 2.87 (t, J = 8.2 Hz, 2H), 2.47 (t, J = 8.2 Hz, 2H), 1.51 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.70, 144.98, 136.21, 135.49, 134.54, 133.72, 128.27, 127.43, 127.40, 126.67, 118.82, 80.16, 28.18, 27.45, 22.76; $^1\text{FTIR}$ (NaCl, cm^{-1}): 3053.32, 2981.95, 1697.36, 1616.35, 1392.61, 1367.53, 1330.88, 1309.67, 1288.45, 1265.30, 1147.65, 848.68, 738.74;

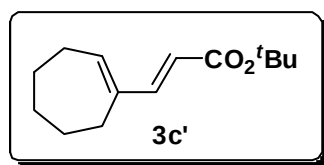
HRMS (ESI) m/z calculated for $C_{17}H_{21}O_2$ $[M+H]^+$: 257.1542, found 257.1548.

(*E*)-tert-butyl 3-(cyclohex-1-en-1-yl)acrylate (3b')



This compound was prepared by the general procedure described above and was obtained as a colorless oil in 73% yield. 1H NMR (400 MHz, $CDCl_3$) δ 7.19 (d, J = 15.8 Hz, 1H), 6.12 (s, 1H), 5.69 (d, J = 15.7 Hz, 1H), 2.20 – 2.11 (m, 4H), 1.71 – 1.59 (m, 4H), 1.49 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.00, 147.00, 137.88, 134.85, 116.41, 79.82, 28.15, 26.32, 24.14, 22.05, 22.02; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2981.95, 2931.80, 2864.29, 1699.29, 1627.92, 1456.26, 1392.61, 1367.53, 1317.38, 1265.30, 1153.43, 972.12, 756.45; HRMS (ESI) m/z calculated for $C_{13}H_{21}O_2$ $[M+H]^+$: 209.1542, found 209.1541.

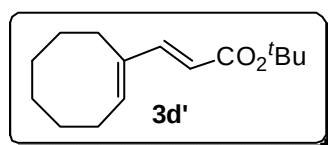
(*E*)-tert-butyl 3-(cyclohept-1-en-1-yl)acrylate (3c')



This compound was prepared by the general procedure described above and was obtained as a colorless oil in 46% yield. 1H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, J = 15.7 Hz, 1H), 6.27 (t, J = 6.8 Hz, 1H), 5.73 (d, J = 15.7 Hz, 1H), 2.33 – 2.26 (m, 4H), 1.80 – 1.74 (m, 2H), 1.54 – 1.51 (m, 4H), 1.49 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.17, 148.03, 142.94, 141.97, 116.48, 79.89, 31.94, 29.03, 28.21, 27.24, 26.29, 25.95; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2931.80, 2864.29, 1699.29, 1618.28,

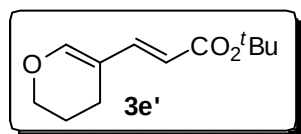
1456.26, 1367.53, 1313.52, 1265.30, 1153.43, 894.97, 850.61, 738.74;
HRMS (ESI) m/z calculated for $C_{14}H_{23}O_2$ $[M+H]^+$: 223.1698, found 223.1695.

(E)-tert-butyl 3-((E)-cyclooct-1-en-1-yl)acrylate (3d')



This compound was prepared by the general procedure described above and was obtained as a colorless oil in 43% yield. 1H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, J = 15.7 Hz, 1H), 6.10 (t, J = 8.4 Hz, 1H), 5.75 (d, J = 15.7 Hz, 1H), 2.41 – 2.38 (m, 2H), 2.29 – 2.23 (m, 2H), 1.58 – 1.53 (m, 4H), 1.49 (s, 9H), 1.47 – 1.42 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.18, 146.97, 141.12, 138.42, 116.98, 79.94, 29.92, 28.35, 28.22, 27.59, 26.74, 25.96, 24.41; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2931.80, 2864.29, 1669.27, 1421.54, 1367.53, 1313.52, 1265.30, 1153.50, 894.97, 738.74; HRMS (ESI) m/z calculated for $C_{15}H_{25}O_2$ $[M+H]^+$: 237.1855, found 237.1859.

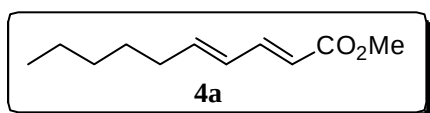
(E)-tert-butyl 3-(3,4-dihydro-2H-pyran-5-yl)acrylate (3e')



This compound was prepared by the general procedure described above and was obtained as a colorless oil in 45% yield. 1H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, J = 15.5 Hz, 1H), 6.83 (s, 1H), 5.56 (d, J = 15.2 Hz, 1H), 4.06 – 4.03 (m, 2H), 2.15 (t, J = 6.4 Hz, 2H), 1.95 – 1.89 (m, 2H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.14, 152.04, 143.90, 113.39, 112.59, 79.62, 66.73, 28.24, 21.30, 19.46; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81,

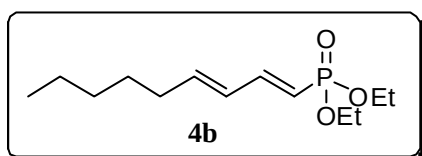
2931.80, 1720.50, 1421.54, 1367.53, 1313.52, 1265.30, 1155.36, 738.74;
HRMS (ESI) m/z calculated for $C_{12}H_{19}O_3$ $[M+H]^+$: 211.1334, found 211.1333.

(2E,4E)-methyl deca-2,4-dienoate (4a)



This compound was prepared by the general procedure described above and was obtained as a colorless oil inseparable mixture ($E/Z = 80:20$) in 63% yield, 1H and ^{13}C NMR are described for the E isomer: 1H NMR (400 MHz, $CDCl_3$) δ 7.27 (dd, $J = 15.4, 10.1$ Hz, 1H), 6.21 – 6.09 (m, 2H), 5.79 (d, $J = 15.4$ Hz, 1H), 3.74 (s, 3H), 2.16 (dd, $J = 13.3, 7.2$ Hz, 2H), 1.47 – 1.39 (m, 2H), 1.34 – 1.26 (m, 4H), 0.89 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.75, 145.40, 144.99, 128.28, 118.64, 51.41, 32.95, 31.36, 28.36, 22.45, 13.97; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 1956.87, 2829.87, 1712.79, 1643.35, 1435.04, 1421.54, 1265.30, 1143.79, 1002.98, 894.97, 738.74, 704.02; HRMS (ESI) m/z calculated for $C_{11}H_{19}O_2$ $[M+H]^+$: 183.1387, found 183.1385.

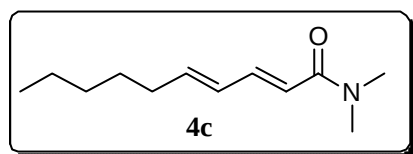
diethyl (1E,3E)-nona-1,3-dien-1-ylphosphonate (4b)



This compound was prepared by the general procedure described above and was obtained as a colorless oil inseparable mixture ($E/Z = 90:10$) in 35% yield, 1H and ^{13}C NMR are described for

the *E* isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.05 (ddd, $J = 21.0, 16.8, 9.7$ Hz, 1H), 6.15– 6.01 (m, 2H), 5.54 (dd, $J = 19.5, 16.9$ Hz, 1H), 4.09 – 4.02 (m, 4H), 2.13 (dd, $J = 14.0, 7.0$ Hz, 2H), 1.44 – 1.37 (m, 2H), 1.34 – 1.24 (m, 10H), 0.87 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.34 (d, $J = 6$ Hz), 144.02, 129.29 (d, $J = 26$ Hz), 114.29 (d, $J = 190$ Hz), 61.57 (d, $J = 5$ Hz), 32.71, 31.31, 28.30, 22.41, 16.33 (d, $J = 6$ Hz), 13.94; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2958.80, 2929.87, 2854.65, 1643.35, 1616.35, 1392.61, 1265.30, 1238.30, 1163.08, 1097.50, 1026.13, 966.34, 846.75, 736.81; HRMS (ESI) m/z calculated for $\text{C}_{13}\text{H}_{26}\text{O}_3\text{P}$ $[\text{M}+\text{H}]^+$: 261.1620, found 261.1624.

(2*E*,4*E*)-*N,N*-dimethyldeca-2,4-dienamide (4c)

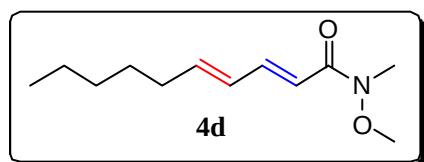


This compound was prepared by the general procedure described above and was obtained as a colorless oil inseparable

mixture (*E/Z* = 80:20) in 51% yield, ^1H and ^{13}C NMR are described for the *E* isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.29 – 7.22 (m, 1H), 6.24 (d, $J = 14.9$ Hz, 1H), 6.19 – 6.04 (m, 2H), 3.08 (d, $J = 4.6$ Hz, 4H), 3.02 (d, $J = 4.3$ Hz, 4H), 2.15 (q, $J = 7.1$ Hz, 2H), 1.46 – 1.39 (m, 2H), 1.34 – 1.26 (m, 4H), 0.92 – 0.87 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.05, 142.96, 142.84, 128.66, 118.23, 37.21, 35.68, 32.83, 31.29, 28.40, 22.39, 13.92; FTIR (NaCl, cm^{-1}): 3053.32, 2981.95, 2956.87, 2929.87, 2868.15,

1651.07, 1625.99, 1598.99, 1492.90, 1396.46, 1265.30, 1132.21, 999.13, 738.74, 704.02; HRMS (ESI) m/z calculated for $C_{12}H_{22}ON$ $[M+H]^+$: 196.1701, found 196.1703.

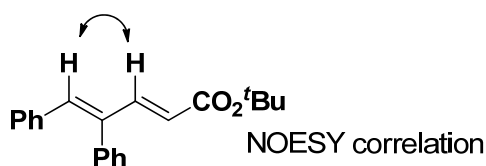
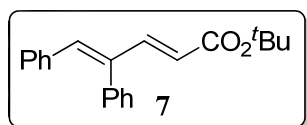
(2E,4E)-N-methoxy-N-methyldeca-2,4-dienamide (4d)



This compound was prepared by the general procedure described above and was obtained as a colorless oil inseparable

mixture ($E/Z = 80:20$) in 45% yield, 1H and ^{13}C NMR are described for the E isomer: 1H NMR (400 MHz, $CDCl_3$) δ 7.31 (dd, $J = 15.2, 10.7$ Hz, 1H), 6.38 (d, $J = 15.2$ Hz, 1H), 6.26 – 6.09 (m, 2H), 3.70 (s, 3H), 3.25 (s, 3H), 2.16 (q, $J = 7.1$ Hz, 2H), 1.45 – 1.41 (m, 2H), 1.32 – 1.28 (m, 4H), 0.89 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.53, 144.02, 143.94, 128.77, 116.79, 61.67, 32.92, 32.42, 31.35, 28.40, 22.44, 13.96; FTIR (NaCl, cm^{-1}): 3053.32, 2985.81, 2931.80, 1658.78, 1649.14, 1631.78, 1456.26, 1421.54, 1265.30, 1002.98, 736.81; HRMS (ESI) m/z calculated for $C_{12}H_{22}O_2N$ $[M+H]^+$: 212.1651, found 212.1652.

(2E,4Z)-tert-butyl 4,5-diphenylpenta-2,4-dienoate (7)



This compound was prepared by the general procedure described above

and was obtained as a white solid in 65% yield. mp 111-112°C; ^1H NMR (400 MHz, CDCl_3) δ 7.59 (dd, $J = 15.4, 0.4$ Hz, 1H), 7.44 – 7.38 (m, 3H), 7.17 – 7.10 (m, 5H), 6.94 (dd, $J = 7.5, 1.9$ Hz, 2H), 6.89 (s, 1H), 5.43 (d, $J = 15.4$ Hz, 1H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.63, 148.55, 139.68, 137.99, 137.11, 135.80, 129.94, 129.23, 129.11, 128.11, 128.04, 127.75, 122.22, 80.23, 28.15; FTIR (NaCl, cm^{-1}): 3053.32, 2983.88, 1697.36, 1616.35, 1444.68, 1421.54, 1367.53, 1311.59, 1265.30, 1151.50, 1099.43, 738.74; HRMS (ESI) m/z calculated for $\text{C}_{21}\text{H}_{23}\text{O}_2$ $[\text{M}+\text{H}]^+$: 307.1698, found 307.1696.

Reference:

1. (a) Engle, K. M.; Wang, D. H.; Yu, J. Q. *J. Am. Soc. Chem.* **2010**, 132, 14137. (b) Shi, B. F.; Maugel, N.; Zhang, Y. H.; Yu, J. Q. *Angew. Chem., Int. Ed.* **2008**, 47, 4882. (c) Marcovici-Mizrahi, D.; Gottlieb, H. E.; Marks, V.; Nudelman, A. *J. Org. Chem.* **1996**, 61, 8402.
2. Dalgard, J. E.; Rychnovsky, S. D. *J. Am. Soc. Chem.* **2004**, 126, 15662.

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