

Highly regio- and stereoselective intermolecular tandem reaction to synthesize chloro-substituted 1, 3-butadienes

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Supplementary Information

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General methods

Commercial solvents and reagents were used without further purification.

Diphenylacetylene, phenylacetylene, methyl phenylpropiolate, methyl 2-nonynoate, ethyl 2-butynoate, N,N-dimethylacrylamide and 4-octyne were purchased from Alfa; benzoyl chloride and 2-chlorobenzoyl chloride were purchased from Aladian Corporation in China; diethyl acetylenedicarboxylate, methyl acrylate, ethyl acrylate and acrylonitrile were purchased from domestic corporation.

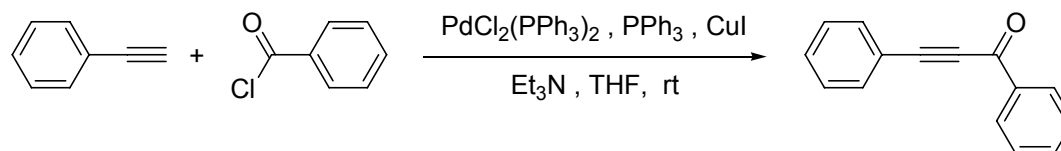
Analytical thin layer chromatography (TLC) plates and the silica gel for column chromatography were phased from Qingdao Haiyang Chemical and Special Silica Gel Co, Ltd.

Proton nuclear magnetic resonance (^1H NMR) and carbon nuclear magnetic resonance (^{13}C NMR) spectroscopy were performed on Bruker Advance 300, 500 and 600 NMR spectrometers. Chemical shifts of ^1H NMR spectra are reported as in units of parts per million (ppm) downfield from SiMe_4 (δ 0.0) and relative to the signal of chloroform-*d* ($J = 7.264$, singlet). Multiplicities were given as: s (singlet); br s (broad singlet); d (doublet); t (triplet); q (quartet); dd (doublet of doublets); m (multiplets), etc. The number of protons (n) for a given resonance is indicated by nH. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as in units of parts per million (ppm) downfield from SiMe_4 (δ 0.0) and relative to the signal of chloroform-*d* ($J = 77.03$, triplet).

HRMS data were collected on Q-star Elite, ELI-LC-MS/MS (product from ABI, America).

Experimental procedure

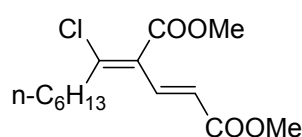
Synthesis of 1,3-diphenylprop-2-yn-1-one:



Benzoyl chloride (703 mg, 5.0 mmol), PdCl_2 (18 mg, 0.1 mmol), PPh_3 (52 mg, 0.2 mmol) and Et_3N (505 mg, 5.0 mmol) in anhydrous THF (15 mL) were stirred for 10 min under argon atmosphere at room temperature. CuI (38 mg, 0.2 mmol) was added and the reaction mixture was stirred for other 10 min before adding 1-ethynylbenzene (551 μl , 5.0 mmol). After 1 h at room temperature, the reaction mixture was worked-up with ethyl acetate and washed with 0.1 N HCl and a saturated NH_4Cl solution. The organic phase was separated, dried over anhydrous magnesium sulfate. The organic solvent was removed on a rotary evaporator under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 15:1) to give 855 mg (83% yield) of 1,3-diphenylprop-2-yn-1-one.

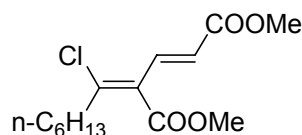
General procedure for the tandem reaction to afford 3a : A mixture of PdCl₂ (8.9 mg, 10 mmol%), CuCl₂ (6 eq, 3 mmol) and 2 mL methyl acrylate was stirred for 5 min in a capped tube (10 mL) at room temperature, then methyl 2-nonynoate (87 μ l, 0.5 mmol) was added. The mixture was stirred under N₂ at 60°C for 24 h. The reaction mixture was extracted with diethyl ether (2 \times 15 mL). The combined organic layer was washed with water (15 mL), brine (10 mL) and then dried over anhydrous magnesium sulfate. The organic solvent was removed on a rotary evaporator under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 10:1) and **3a** was obtained as a yellow oil (**3a**, 110 mg, 76%).

Spectroscopic data of products



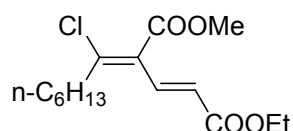
(2E,4Z)-dimethyl 4-(1-chloroheptylidene)pent-2-enedioate

3a yellow oil (**3a** and **4a**: 110 mg, 76%), δ_{H} (300 MHz; CDCl₃; Me₄Si) 0.86-0.94 (3 H, m, 3 \times CH₃), 1.26-1.35 (8 H, m, 8 \times CH₃(CH₂)₄CH₂), 2.49-2.58 (2 H, m, 2 \times CH₃(CH₂)₄CH₂), 3.77 (3 H, s, 3 \times OCH₃), 3.86 (3 H, s, 3 \times OCH₃), 5.94 (1 H, d, *J* 16.0 Hz, CH=CH), 7.74 (1 H, d, *J* 16.0 Hz, CH=CH); δ_{C} (75 MHz; CDCl₃; Me₄Si) 14.0, 22.4, 27.6, 28.4, 31.4, 38.3, 51.8, 52.5, 121.7, 128.7, 137.3, 148.8, 166.3, 166.8; HRMS (ESI): *m/z* (M + Na⁺) calcd for C₁₄H₂₁O₄NaCl, 311.1026, found: 311.1037.



(2E,4E)-dimethyl 4-(1-chloroheptylidene)pent-2-enedioate

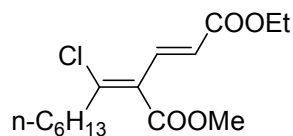
4a yellow oil (**3a** and **4a**: 110 mg, 76%), δ_{H} (300 MHz; CDCl₃; Me₄Si) 0.86-0.94 (3 H, m, 3 \times CH₃), 1.26-1.35 (8 H, m, 8 \times CH₃(CH₂)₄CH₂), 2.49-2.58 (2 H, m, 2 \times CH₃(CH₂)₄CH₂), 3.74 (3 H, s, 3 \times OCH₃), 3.84 (3 H, s, 3 \times OCH₃), 5.83 (1 H, d, *J* 15.9 Hz, CH=CH), 7.46 (1 H, d, *J* 15.9 Hz, CH=CH); δ_{C} (75 MHz; CDCl₃; Me₄Si) 13.9, 14.2, 22.4, 27.7, 28.4, 31.4, 38.3, 52.5, 60.6, 122.3, 128.8, 137.0, 148.5, 166.3, 166.4; HRMS (ESI): *m/z* (M + Na⁺) calcd for C₁₄H₂₁O₄NaCl, 311.1026, found: 311.1037.



(2E,4Z)-1-ethyl 5-methyl 4-(1-chloroheptylidene)pent-2-enedioate

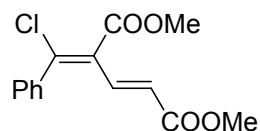
3b yellow oil (**3b** and **4b**: 118 mg, 78%), δ_{H} (300 MHz; CDCl₃; Me₄Si) 0.82 (3 H, t, *J* 7.1 Hz, 3 \times CH₃), 1.18-1.26 (11 H, m, 3 \times CH₃CH₂ and 8 \times CH₃(CH₂)₄CH₂), 2.42-2.48 (2 H, m, 2 \times CH₃(CH₂)₄CH₂), 3.78 (3 H, s, 3 \times OCH₃), 4.16 (2 H, q, *J* 7.1 Hz, 2 \times CH₂CH₃), 5.86 (1 H, d, *J*

16.0 Hz, $CH=CH$), 7.65 (1 H, d, J 16.0 Hz, $CH=CH$); δ_C (75 MHz; $CDCl_3$; Me_4Si); 13.9, 14.2, 22.4, 27.5, 28.2, 31.4, 37.6, 52.1, 52.3, 121.7, 129.6, 137.3, 148.8, 166.3, 166.8; HRMS (ESI) : m/z ($M + Na^+$) calcd for $C_{15}H_{23}O_4NaCl$, 325.1183, found: 325.1187.



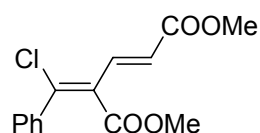
(2E,4E)-1-ethyl 5-methyl 4-(1-chloroheptylidene)pent-2-enedioate

4b yellow oil (**3b** and **4b**: 118 mg, 78%), δ_H (300 MHz; $CDCl_3$; Me_4Si) 0.89 (3 H, t, J 6.1 Hz, $3 \times CH_3$), 1.18-1.26 (11 H, m, $3 \times CH_3CH_2$ and $8 \times CH_3(CH_2)_4CH_2$), 2.54-2.58 (2 H, m, $2 \times CH_3(CH_2)_4CH_2$), 3.76 (3 H, s, $3 \times OCH_3$), 4.12 (2 H, q, J 7.1 Hz, $2 \times CH_2CH_3$), 5.76 (1 H, d, J 15.7 Hz, $CH=CH$), 7.37 (1 H, d, J 15.7 Hz, $CH=CH$); δ_C (75 MHz; $CDCl_3$; Me_4Si) 14.0, 14.1, 22.4, 27.6, 28.4, 31.4, 38.3, 51.8, 52.5, 121.7, 128.7, 137.3, 148.8, 166.3, 166.8; HRMS (ESI) : m/z ($M + Na^+$) calcd for $C_{15}H_{23}O_4NaCl$, 325.1183, found: 325.1187.



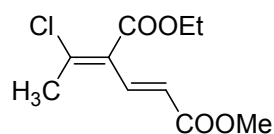
(2E,4Z)-dimethyl 4-(chloro(phenyl)methylene)pent-2-enedioate

3c yellow oil (**3c** and **4c**: 102 mg, 73%), δ_H (600 MHz; $CDCl_3$; Me_4Si) 3.55 (3 H, s, $3 \times OCH_3$), 3.80 (3 H, s, $3 \times OCH_3$), 6.12 (1 H, d, J 16.0 Hz, $CH=CH$), 7.36-7.46 (5 H, m, $5 \times ph$), 7.88 (1 H, d, J 16.0 Hz, $CH=CH$); δ_C (150 MHz; $CDCl_3$; Me_4Si) 50.9, 51.5, 122.1, 127.0, 127.4, 129.0, 129.3, 136.4, 136.6, 142.3, 165.6, 165.7; HRMS (ESI): m/z ($M + Na^+$) calcd for $C_{14}H_{13}O_4NaCl$, 303.0400, found: 303.0405.



(2E,4E)-dimethyl 4-(chloro(phenyl)methylene)pent-2-enedioate

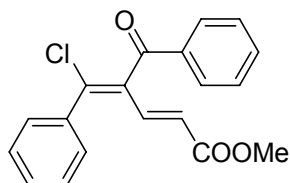
4c yellow oil (**3c** and **4c**: 102 mg, 73%), δ_H (600 MHz; $CDCl_3$; Me_4Si) 3.71 (3 H, s, $3 \times OCH_3$), 3.94 (3 H, s, $3 \times OCH_3$), 5.90 (1 H, d, J 15.9 Hz, $CH=CH$), 7.36-7.46 (5 H, m, $5 \times ph$), 7.27 (1 H, d, J 15.9 Hz, $CH=CH$); δ_C (150 MHz; $CDCl_3$; Me_4Si) 50.8, 51.8, 120.5, 127.0, 127.6, 128.4, 129.5, 136.0, 136.6, 142.3, 165.3, 165.5; HRMS (ESI): m/z ($M + Na^+$) calcd for $C_{14}H_{13}O_4NaCl$, 303.0400, found: 303.0405.



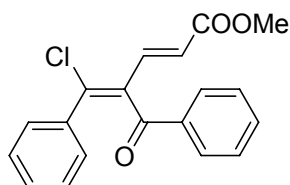
(2E,4Z)-5-ethyl 1-methyl 4-(1-chloroethylidene)pent-2-enedioate

3d colourless oil (**3d** and **4d**: 62 mg, 53%), δ_H (600 MHz; $CDCl_3$; Me_4Si) 1.29 (3H, t, J 7.1 Hz, 3

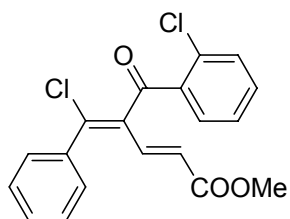
$\times \text{CH}_2\text{CH}_3$), 2.29 (3 H, s, $3 \times \text{CH}_3$), 3.70 (3 H, s, $3 \times \text{OCH}_3$), 4.26 (2 H, q, J 7.1 Hz, $2 \times \text{CH}_2\text{CH}_3$), 5.92 (1 H, d, J 16.0 Hz, $\text{CH}=\text{CH}$), 7.65 (1 H, d, J 16.0 Hz, $\text{CH}=\text{CH}$); δ_{C} (150 MHz; CDCl_3 ; Me_4Si) 13.1, 24.5, 50.8, 60.9, 120.7, 128.1, 138.2, 142.9, 164.7, 165.8.



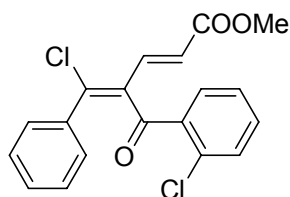
3e yellow oil (**3e** and **4e**: 67 mg, 41%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 3.68 (3 H, s, $3 \times \text{OCH}_3$), 5.79 (1 H, d, J 16.1 Hz, $\text{CH}=\text{CH}$), 7.08-7.67 (10 H, m, $10 \times \text{ph}$), 8.01 (1 H, d, J 16.1 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 51.8, 123.6, 128.2, 128.6, 128.9, 129.4, 130.0, 133.9, 135.3, 135.9, 137.0, 138.9, 140.4, 166.6, 194.6; HRMS (ESI): m/z ($\text{M} + \text{Na}^+$) calcd for $\text{C}_{19}\text{H}_{15}\text{O}_3\text{NaCl}$, 349.0607, found: 349.0610.



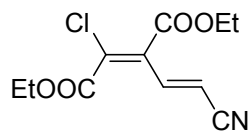
4e yellow oil (**3e** and **4e**: 67 mg, 41%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 3.58 (3 H, s, $3 \times \text{OCH}_3$), 5.65 (1 H, d, J 16.1 Hz, $\text{CH}=\text{CH}$), 7.08-7.67 (10 H, m, $10 \times \text{ph}$), 8.17 (1 H, d, J 16.1 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 51.8, 123.6, 128.2, 128.6, 128.9, 129.4, 130.0, 133.9, 135.3, 135.9, 137.0, 138.9, 140.4, 166.6, 194.6; HRMS (ESI): m/z ($\text{M} + \text{Na}^+$) calcd for $\text{C}_{19}\text{H}_{15}\text{O}_3\text{NaCl}$, 349.0607, found: 349.0610.



3f yellow oil (**3f** and **4f**: 114 mg, 63%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 3.72 (3 H, s, $3 \times \text{OCH}_3$), 6.06 (1 H, d, J 16.1 Hz, $\text{CH}=\text{CH}$), 6.97-7.29 (9 H, m, $9 \times \text{ph}$), 7.96 (1 H, d, J 16.1 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 51.8, 123.7, 126.4, 128.3, 128.7, 130.2, 130.9, 131.5, 132.9, 133.1, 136.2, 136.4, 137.2, 138.6, 143.3, 166.9, 193.0; HRMS (ESI): m/z ($\text{M} + \text{Na}^+$) calcd for $\text{C}_{19}\text{H}_{14}\text{O}_3\text{NaCl}_2$, 383.0218, found: 383.0218.

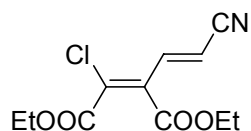


4f yellow oil (**3f** and **4f**: 114 mg, 63%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 3.78 (3 H, s, $3 \times \text{OCH}_3$), 5.89 (1 H, d, J 16.1 Hz, $\text{CH}=\text{CH}$), 7.02-7.45 (9 H, m, $9 \times \text{ph}$), 8.12 (1 H, d, J 16.1 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 51.8, 123.8, 126.5, 128.4, 128.8, 130.2, 130.9, 131.5, 132.9, 133.1, 136.1, 136.3, 137.2, 138.6, 143.4, 166.7, 193.0; HRMS (ESI): m/z ($\text{M} + \text{Na}^+$) calcd for $\text{C}_{19}\text{H}_{14}\text{O}_3\text{NaCl}_2$, 383.0218, found: 383.0218.



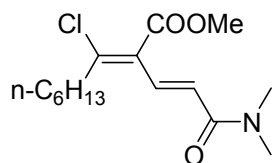
diethyl 2-chloro-3-((*E*)-2-cyanovinyl)fumarate

3g yellow oil (**3g** and **4g**: 95 mg, 74%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 1.23-1.30 (6 H, m, $6 \times \text{CH}_2\text{CH}_3$), 4.20-4.28 (4 H, m, $4 \times \text{CH}_2\text{CH}_3$), 5.76 (1 H, d, J 16.6 Hz, $\text{CH}=\text{CH}$), 7.40 (1 H, d, J 16.6 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 13.6, 13.7, 62.7, 63.3, 106.2, 116.4, 131.5, 135.7, 141.2, 161.1, 163.5; HRMS (ESI): m/z ($\text{M} + \text{Na}^+$) calcd for $\text{C}_{11}\text{H}_{12}\text{NO}_4\text{NaCl}$, 280.0353, found: 280.0355.



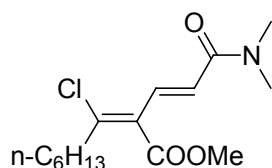
diethyl 2-chloro-3-((*E*)-2-cyanovinyl)maleate

4g yellow oil (**3g** and **4g**: 95 mg, 74%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 1.23-1.30 (6 H, m, $6 \times \text{CH}_2\text{CH}_3$), 4.18-4.23 (4 H, m, $4 \times \text{CH}_2\text{CH}_3$), 5.93 (1 H, d, J 16.6 Hz, $\text{CH}=\text{CH}$), 7.56 (1 H, d, J 16.6 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 13.5, 13.8, 62.5, 62.9, 106.2, 115.9, 128.2, 135.1, 141.2, 161.1, 162.4; HRMS (ESI): m/z ($\text{M} + \text{Na}^+$) calcd for $\text{C}_{11}\text{H}_{12}\text{NO}_4\text{NaCl}$, 280.0353, found: 280.0355.



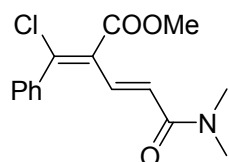
(*ZZ*)-methyl 2-((*E*)-2-(dimethylcarbamoyl)vinyl)-3-chloronon-2-enoate

3h yellow oil (**3h** and **4h**: 124 mg, 82%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 0.87-0.90 (3 H, m, $3 \times \text{CH}_3$), 1.24-1.34 (8 H, m, $8 \times \text{CH}_3(\text{CH}_2)_4\text{CH}_2$), 2.50 (2 H, t, J 7.6 Hz, $2 \times \text{CH}_3(\text{CH}_2)_4\text{CH}_2$), 3.02 (3 H, s, $3 \times \text{N}(\text{CH}_3)_2$), 3.07 (3 H, s, $3 \times \text{N}(\text{CH}_3)_2$), 3.85 (3 H, s, $3 \times \text{OCH}_3$), 6.41 (1 H, d, J 15.5 Hz, $\text{CH}=\text{CH}$), 7.67 (1 H, d, J 15.5 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 14.0, 22.4, 27.7, 28.4, 31.4, 35.8, 37.3, 38.3, 52.3, 121.6, 128.9, 134.8, 147.2, 166.2, 166.9; HRMS (ESI): m/z ($\text{M} + \text{H}^+$) calcd for $\text{C}_{15}\text{H}_{25}\text{NO}_3\text{Cl}$, 302.1523, found: 302.1520.



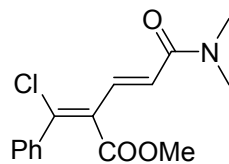
(2E)-methyl 2-((E)-2-(dimethylcarbamoyl)vinyl)-3-chloronon-2-enoate

4h yellow oil (**3h** and **4h**: 124 mg, 82%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 0.96-1.01 (3 H, m, $3 \times \text{CH}_3$), 1.24-1.34 (8 H, m, $8 \times \text{CH}_3(\text{CH}_2)_4\text{CH}_2$), 2.64 (2 H, t, J 7.6 Hz, $2 \times \text{CH}_3(\text{CH}_2)_4\text{CH}_2$), 3.00 (3 H, s, $3 \times \text{N}(\text{CH}_3)_2$), 3.13 (3 H, s, $3 \times \text{N}(\text{CH}_3)_2$), 3.87 (3 H, s, $3 \times \text{OCH}_3$), 6.28 (1 H, d, J 15.2 Hz, $\text{CH}=\text{CH}$), 7.46 (1 H, d, J 15.2 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 14.0, 22.4, 27.7, 28.4, 31.5, 35.2, 37.3, 38.3, 52.3, 120.2, 131.1, 133.0, 147.2, 165.8, 167.1; HRMS (ESI): m/z ($\text{M} + \text{H}^+$) calcd for $\text{C}_{15}\text{H}_{25}\text{NO}_3\text{Cl}$, 302.1523, found: 302.1520.



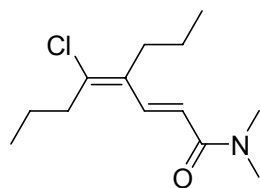
(2Z,3E)-methyl 4-(dimethylcarbamoyl)-2-(chloro(phenyl)methylene)but-3-enoate

3i yellow solid (**3i** and **4i**: 91 mg, 62%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 2.94 (3 H, s, $\text{N}(\text{CH}_3)_2$), 2.98 (3 H, s, $\text{N}(\text{CH}_3)_2$), 3.41 (3 H, s, OCH_3), 6.53 (1 H, d, J 15.4, $\text{CH}=\text{CH}$), 7.26-7.34 (5 H, m, *Ph*), 7.74 (1 H, d, J 15.4, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 35.7, 37.2, 52.2, 123.1, 127.9, 128.3, 130.0, 130.3, 134.8, 137.9, 141.8, 165.8, 167.0; HRMS (ESI): m/z ($\text{M} + \text{H}^+$) calcd for $\text{C}_{15}\text{H}_{17}\text{NO}_3\text{Cl}$, 294.0897, found: 294.0897.



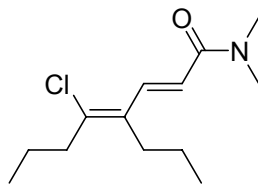
(2E,3E)-methyl 4-(dimethylcarbamoyl)-2-(chloro(phenyl)methylene)but-3-enoate

4i yellow solid (**3i** and **4i**: 91 mg, 62%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 2.85 (3 H, s, $\text{N}(\text{CH}_3)_2$), 2.90 (3 H, s, $\text{N}(\text{CH}_3)_2$), 3.84 (3 H, s, OCH_3), 6.26 (1 H, d, J 15.4, $\text{CH}=\text{CH}$), 7.26-7.34 (5 H, m, *Ph*), 7.17 (1 H, d, J 15.4, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 36.1, 37.0, 53.5, 121.2, 128.5, 129.3, 130.2, 131.7, 134.5, 138.6, 141.8, 165.5, 169.5; HRMS (ESI): m/z ($\text{M} + \text{H}^+$) calcd for $\text{C}_{15}\text{H}_{17}\text{NO}_3\text{Cl}$, 294.0897, found: 294.0897.



(2E,4E)-5-chloro-N,N-dimethyl-4-propylocta-2,4-dienamide

3j colourless oil (**3g** and **4g**: 95 mg, 78%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 0.86-0.94 (6 H, m, $6 \times \text{CH}_3$), 1.38-1.49 (4 H, m, $4 \times \text{CH}_2\text{CH}_2\text{CH}_3$), 2.27-2.31 (2 H, m, $2 \times \text{CH}_2\text{CH}_2\text{CH}_3$), 2.40-2.47 (2 H, m, $2 \times \text{CH}_2\text{CH}_2\text{CH}_3$), 3.04 (3 H, s, $3 \times \text{N}(\text{CH}_3)_2$), 3.10 (3 H, s, $3 \times \text{N}(\text{CH}_3)_2$), 6.34 (1 H, d, J 15.1 Hz, $\text{CH}=\text{CH}$), 7.56 (1 H, d, J 15.1 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 13.1, 14.1, 21.2, 21.6, 31.0, 35.8, 37.3, 37.4, 117.4, 131.7, 137.7, 140.1, 167.2; HRMS (ESI): m/z ($\text{M} + \text{H}^+$) calcd for $\text{C}_{13}\text{H}_{23}\text{NOCl}$, 244.1468, found: 244.1469.



(2E,4Z)-5-chloro-N,N-dimethyl-4-propylocta-2,4-dienamide

4j colourless oil (**3g** and **4g**: 95 mg, 78%), δ_{H} (500 MHz; CDCl_3 ; Me_4Si) 0.86-0.94 (6 H, m, 6 \times CH_3), 1.55-1.65 (4 H, m, 4 \times $\text{CH}_2\text{CH}_2\text{CH}_3$), 2.40-2.47 (2 H, m, 2 \times $\text{CH}_2\text{CH}_2\text{CH}_3$), 2.58-2.62 (2 H, m, 2 \times $\text{CH}_2\text{CH}_2\text{CH}_3$), 3.04 (3 H, s, 3 \times N (CH_3)₂), 3.11 (3 H, s, 3 \times N (CH_3)₂), 6.31 (1 H, d, J 15.5 Hz, $\text{CH}=\text{CH}$), 7.80 (1 H, d, J 15.5 Hz, $\text{CH}=\text{CH}$); δ_{C} (125 MHz; CDCl_3 ; Me_4Si) 13.4, 14.1, 21.2, 22.3, 32.0, 35.7, 35.8, 38.2, 118.7, 133.4, 139.4, 142.2, 167.0; HRMS (ESI): m/z ($\text{M} + \text{H}^+$) calcd for $\text{C}_{13}\text{H}_{23}\text{NOCl}$, 244.1468, found: 244.1469.

Copies of NMR spectra of products

