

Electronic Supplementary Information

Photocatalytic chemo-, regio- & stereoselective olefinic β -C–H decarboxylative alkylation of enamides with diacyl peroxides

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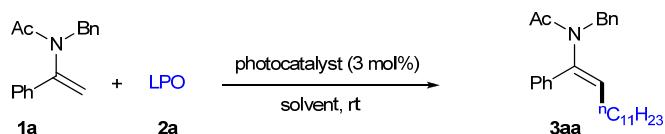
1. General Considerations

General Information: Unless otherwise noted, all chemicals were purchased and used without further purification. ^1H NMR and ^{13}C NMR spectra were recorded at ambient temperature on a 300 or 400 MHz NMR spectrometer (75 or 100 MHz for ^{13}C). NMR experiments are reported in δ units, parts per million (ppm), and were referenced to CDCl_3 (d 7.26 or 77.0) as the internal standard. The coupling constants J are given in Hz. Column chromatography was performed using EM Silica gel 60 (300-400 mesh).

2. General Synthetic Procedures

All Enamides (**1a-1q**) were prepared according to the previous reports¹. Besides lauroyl peroxide (LPO) was purchased, other diacyl peroxides were prepared according to the previous reports.²

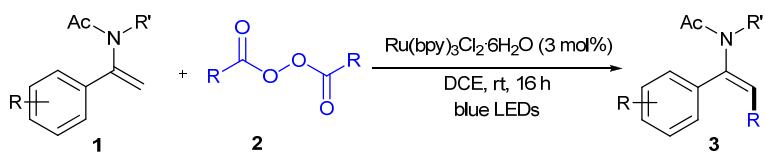
Table S1. Screening the optimal reaction conditions.^a



Entry	Photocatalyst	Solvent	Yield (%)
1	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	DCM	60
2	Rose bengal	DCM	23
3	Rhodamine 6G	DCM	58(64) ^b
4	Rhodamine B	DCM	10
5	Eosin B	DCM	0
6	Mes-AcrClO ₄	DCM	53
7	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	MeCN	70
8	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	acetone	68
9	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	DMSO	55
10	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	ethyl acetate	0
11	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	MeOH	42
12	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	DCE	75
13	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	THF	45
14	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	DCE	46 ^c (55) ^d (51) ^e
15	---	DCE	0
16	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	DCE	0 ^f

^a Reaction conditions: **1a** (0.2 mmol), **2a** (2 equiv), photocatalyst (3 mol%) and solvent (2 mL) under N₂ with 20 W 440-450 nm blue LEDs irradiation at rt for 16 h, isolated yield. ^b In DCE. ^c **2a** (1 equiv). ^d **2a** (3 equiv). ^e 6 W blue LEDs. ^f Without LEDs.

General Procedure for the Synthesis of Compounds (3aa-3wa, 3ab-3as):



Under N_2 , the mixture of enamides **1** (0.2 mmol), diacyl peroxide **2** (0.4 mmol), $\text{Ru(bpy)}_3\text{Cl}_2 \cdot 6\text{H}_2\text{O}$ (3 mol%, 3.7 mg) and DCE (2 mL) were added to a Schlenk tube and sealed. The mixture was stirred at room temperature under 440-450 nm blue LEDs for 16 hours. Then, the solvent was evaporated under reduced pressure, and the residue was purified by silica gel flash column chromatography to obtain the product **3**. The photoreactor is shown in Figure S1.



Figure S1. Photoreactor used in this work

The Light Source and the Material of the Irradiation Vessel:

The photochemical reaction was carried out under visible light irradiation by a 20W 440-450 nm blue LED at room temperature. This blue LED was purchased from taobao (link: <https://m.tb.cn/h.UGjMWKP?sm=3da13d&tk=meq7dlJPct4>). The blue LED's energy peak wavelength is 448 nm, the peak width at half-height is 18.0 nm, and irradiance@20 W is 35.07 mW/cm². The reaction vessel is a borosilicate glass tube. The distance between the tube and lamp is about 1cm, and no filter is applied.

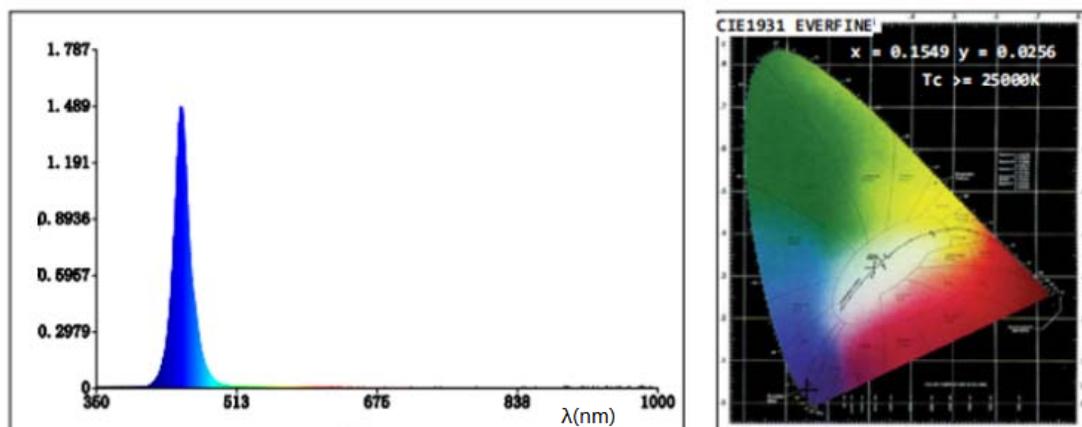
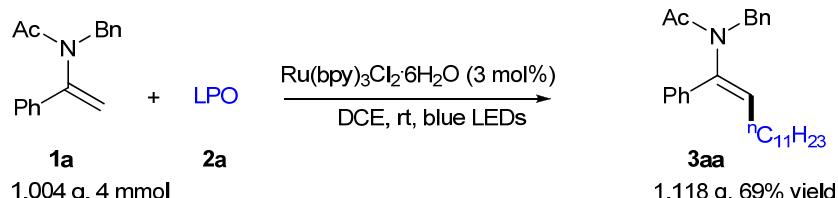


Figure S2. The spectral distribution of 20 W 440-450 nm purple LED (From the above link)

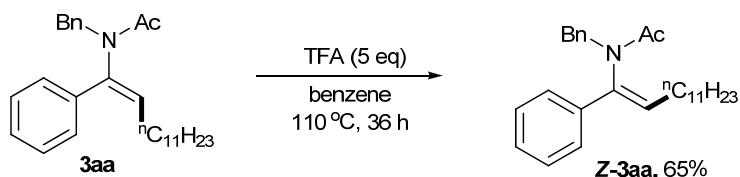
Synthetic applications:

(1) Gram-Scale Synthesis of β -alklated Enamide 3aa



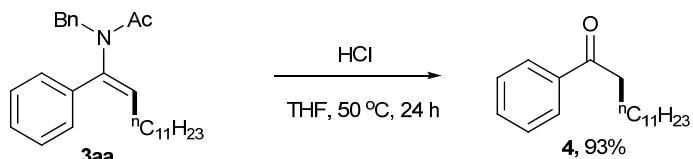
Under N₂, the mixture of enamides **1a** (1.004 g, 4 mmol), LPO **2a** (8 mmol, 2 equiv.), Ru(bpy)₃Cl₂·6H₂O (0.12 mmol, 3 mol%) and DCE (10 mL) were added to a Schlenk flask and sealed. The mixture was stirred at room temperature under 440-450 nm blue LEDs for 16 hours. Then, the solvent was evaporated under reduced pressure, and the residue was purified by silica gel flash column chromatography to obtain the product **3aa** in 69% yield (1.118 g).

(2) Conversion of the Configuration of 3aa



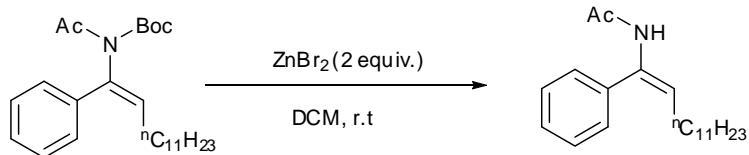
A mixture of **3aa** (40.6 mg, 0.1 mmol), trifluoroacetic acid (57 mg, 0.5 mmol) in dry benzene (2.0 mL) were stirred at 110 °C for 36 h. Upon completion, the solution was concentrated in vacuum and the product was isolated through flash column chromatography to furnish *Z*-**3aa** as colourless oil (26.4 mg, 65% yield).

(3) Hydrolysis of 3aa



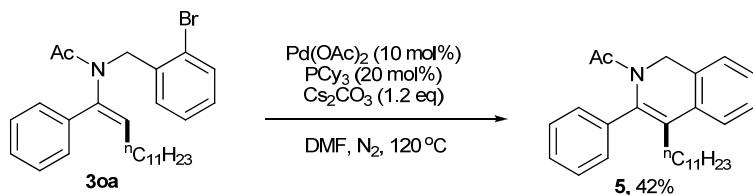
3aa (40.6 mg, 0.1 mmol) was dissolved in THF (2.0 mL) and concentrated hydrochloric acid (0.4 mL) were added sequentially. The mixture was stirred at 50 °C for 24 h. Upon completion, the solution was concentrated in vacuum and the product was isolated through flash column chromatography to give **4** as colourless oil (25.4 mg, 93% yield).

(4) Cleavage of N-Boc Protecting Group



The mixture of **3ua** (41.5 mg, 0.1 mmol) and ZnBr_2 (44.4 mg, 0.2 mmol) in DCM (1.0 mL) was stirred at room temperature for 6 hours. The solvent was then removed under vacuum. The residue was purified by flash column chromatography, giving the desired product **5** (16.1 mg, 51% yield).

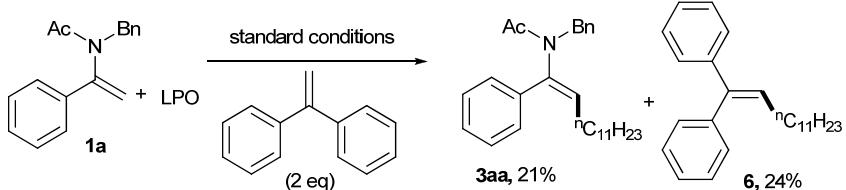
(5) The palladium-catalyzed Heck-coupling reaction



A mixture of **3oa** (48.5 mg, 0.1 mmol), $\text{Pd}(\text{OAc})_2$ (2.3 mg, 10 mol%), tricyclohexylphosphane (5.6 mg, 20 mol%), and Cs_2CO_3 (39.1 mg, 0.12 mmol) in DMF (2.0 mL) were stirred at 120 °C for 24 h. Upon completion, the solution was concentrated in vacuum and the product was isolated through flash column chromatography to give **5** as colourless oil (16.8 mg, 42% yield).

3. Mechanism Studies

3.1 Radical inhibiting and trapping experiment



Under N_2 , the mixture of enamides **1a** (0.1 mmol), 1,1-diphenylethylene (0.2 mmol), LPO (2 equiv.), $\text{Ru}(\text{bpy})_3\text{Cl}_2 \cdot 6\text{H}_2\text{O}$ (3 mol%) and DCE (1 mL) were added to a Schlenk tube and sealed. The mixture was stirred at room temperature under 440-450 nm blue LEDs for 16 hours. Then, the solvent was evaporated under reduced pressure, and the residue was purified by silica gel flash column chromatography to obtain **3aa** (8.4 mg, 21% yield) and the adduct **6** as colourless oil (16 mg, 24% yield based on **2a**). The NMR data of compound **6** was as following. ^1H NMR (400 MHz, CDCl_3) δ 7.38-7.16 (m, 10H), 6.08 (t, $J = 7.5$ Hz, 1H), 2.10 (dd, $J = 14.7, 7.4$ Hz, 2H), 1.42 (dd, $J = 14.6, 7.1$ Hz, 2H), 1.29-1.23 (m, 16H), 0.88 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 143.0, 141.4, 140.4, 130.4, 130.0, 128.1, 128.1, 127.2, 126.8, 126.8, 32.0, 30.0, 29.8, 29.7, 29.7, 29.6, 29.6, 29.4, 29.3, 22.8, 14.2.

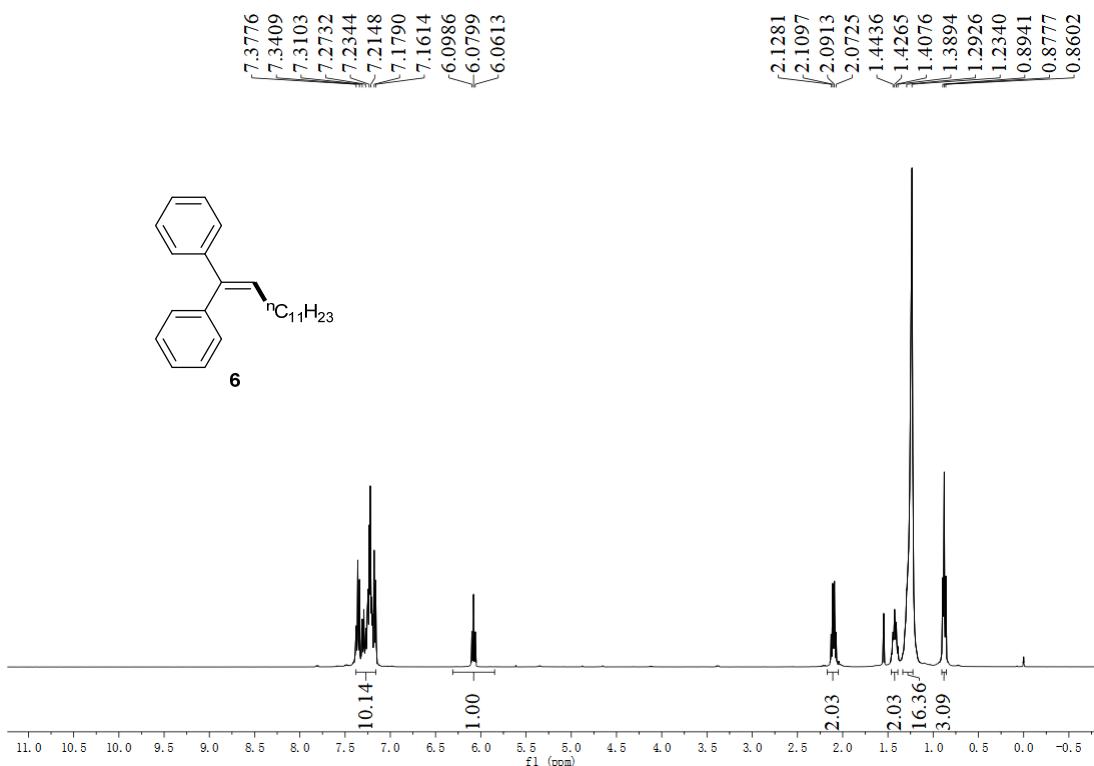


Figure S3. ^1H NMR of the adduct formed by $\cdot\text{C}_{11}\text{H}_{23}$ and 1,1-diphenylethylene.

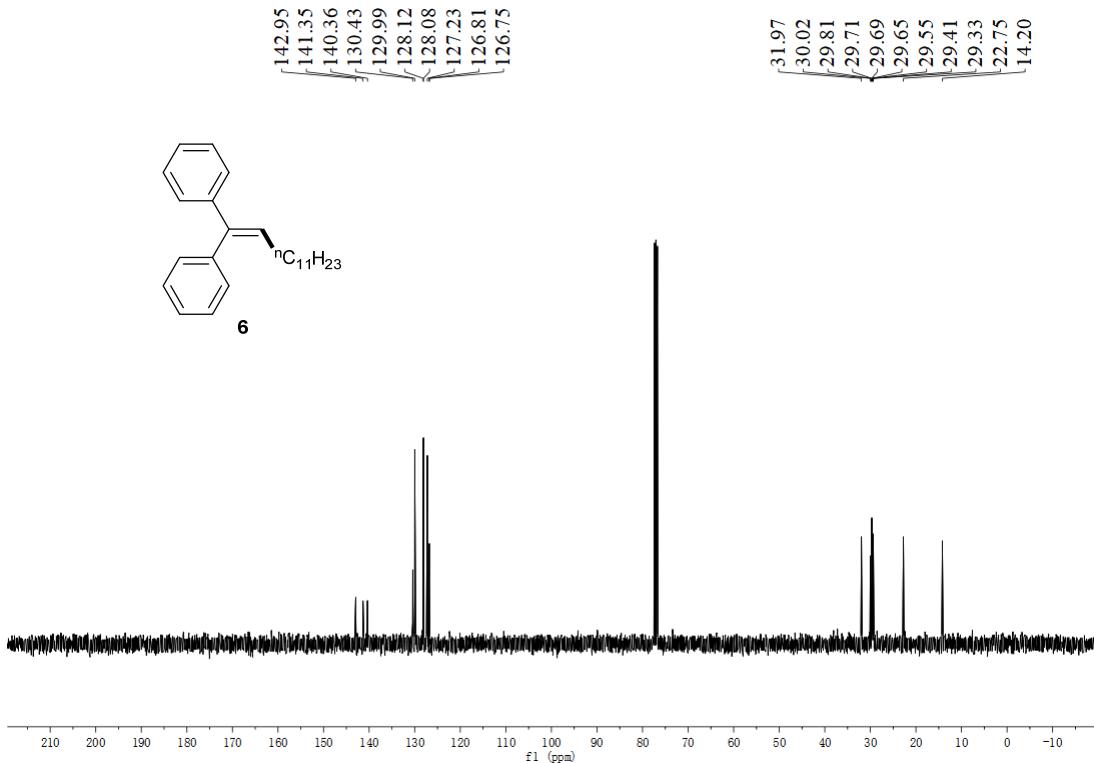
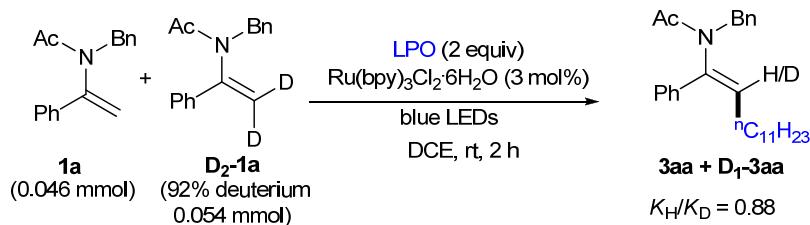


Figure S4. ^{13}C NMR of the adduct formed by $\cdot\text{C}_{11}\text{H}_{23}$ and 1,1-diphenylethylene.

3.2 Intermolecular Kinetic Isotopic Effect (KIE) Study



Enamide **D₂-1a** was prepared according to the literatures,³ as a light yellow oil with 92% deuterium. Under N₂, the mixture of enamides **1a** (0.046 mmol), enamides **D₂-1a** (0.054 mmol), LPO (2 equiv), Ru(bpy)₃Cl₂·6H₂O (3 mol%) and DCE (2 mL) were added to a Schlenk tube and sealed. The mixture was stirred at room temperature under 440-450nm blue LEDs for 2 hours. Then, the solvent was evaporated under reduced pressure, and the residue was purified by silica gel flash column chromatography to obtain product **3aa+D₁-3aa** as colourless oil (11.7 mg, 29% yield). A KIE value of 0.88 was observed.

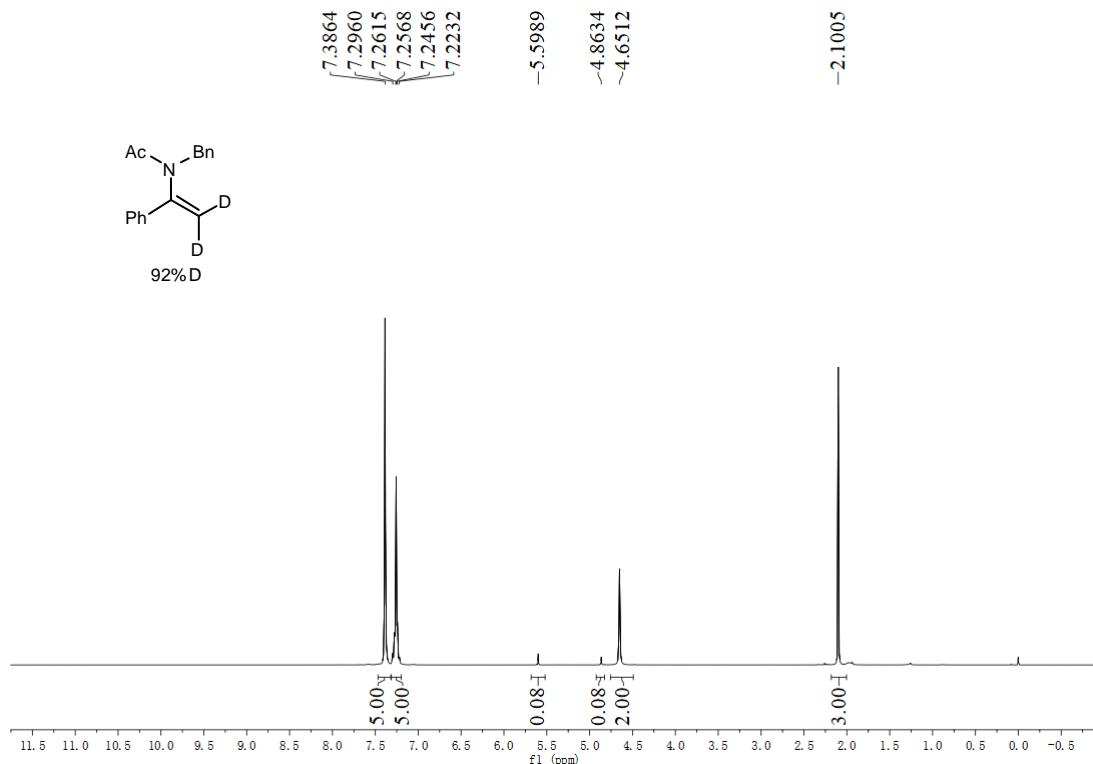


Figure S5. ¹H NMR of enamide **D₂-1a**

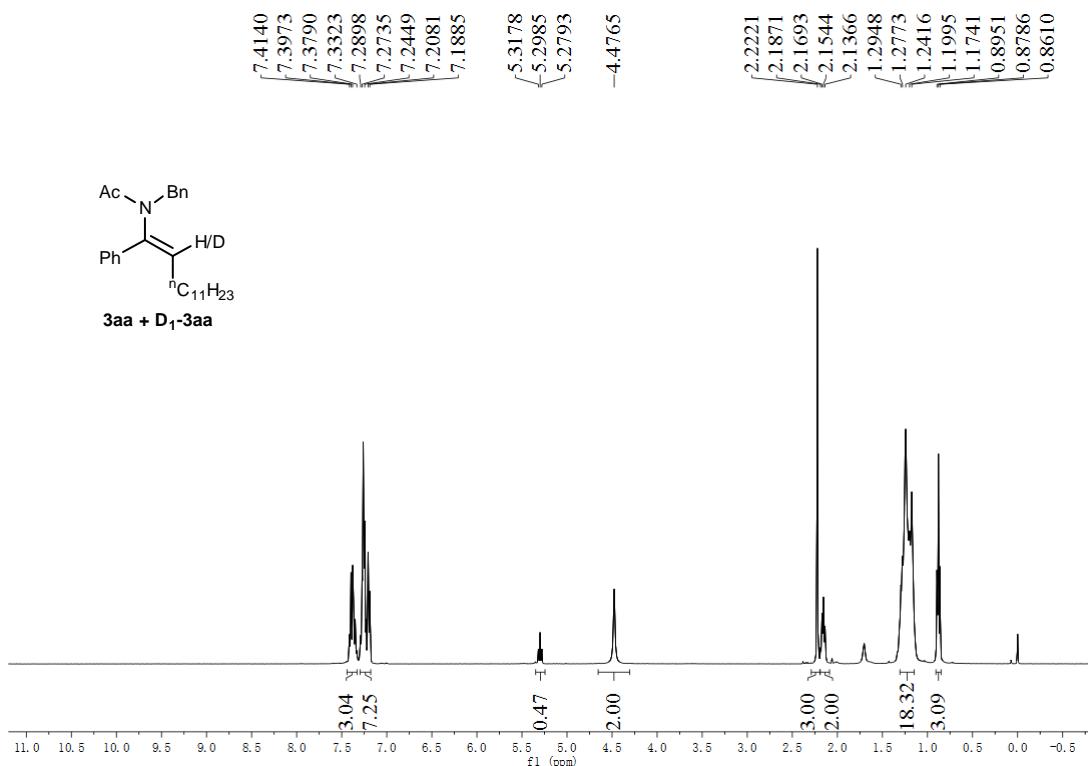


Figure S6. ¹H NMR spectrum of **3aa** and **D₁-3aa**

3.3 Fluorescence Quenching Experiments

Fluorescence quenching experiments were measured on an Agilent Technologies Cary Eclipse Fluorescence Spectrophotometer. The Ru(bpy)₃Cl₂·H₂O photocatalyst was excited at 450 nm and the emission spectrum max = 600 nm was recorded. The concentration of photocatalyst stock solution was 1 × 10⁻⁵ M in DCE. LPO with different concentrations were added into the solution of photocatalyst in DCE (3 mL) at room temperature. After being stirred with a thin glass rod, the emission spectrum was collected. Linear regression of I₀/I against concentration is done in Origin 2018 (Figure S7).

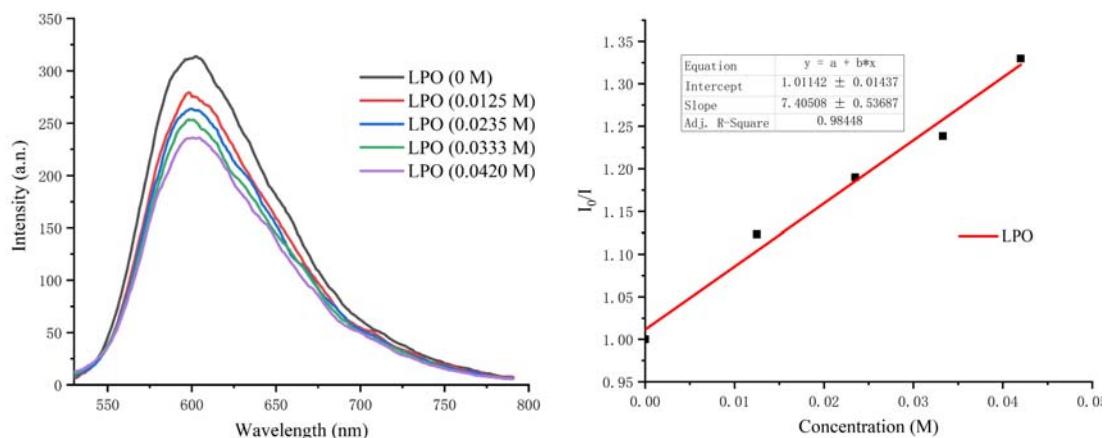


Figure S7. Stern-Volmer fluorescence quenching of the excited Ru(bpy)₃Cl₂·H₂O with LPO.

3.4 Cyclic voltammetry study

Cyclic voltammetric investigations were performed on the DonghuaDH7000C electrochemical workstation with the conventional three-electrode system. The electrodes are first polished with sandpaper, then with alumina powder until the surface of the electrodes is mirror-like. Finally, the electrodes are washed with distilled water and ultrasonication. The solvent exhaust employs a nitrogen blast for 1 h. A glassy carbon electrode was used as the working electrode, a saturated calomel electrode (SCE) and platinum electrode were used as the reference electrode and counter electrode, respectively. LPO (1 mM) was tested with tetrabutylammonium hexafluorophosphate (0.1 M) as the supporting electrolyte in 40 mL MeCN. Solutions were kept under positive pressure of nitrogen during the measurements. Cyclic voltammetry (CV) with the following settings: Scan Rates = 0.1 V/s, Sweep Segments = 10, Sample Interval = 0.001 V, Quiet Time = 2 sec.

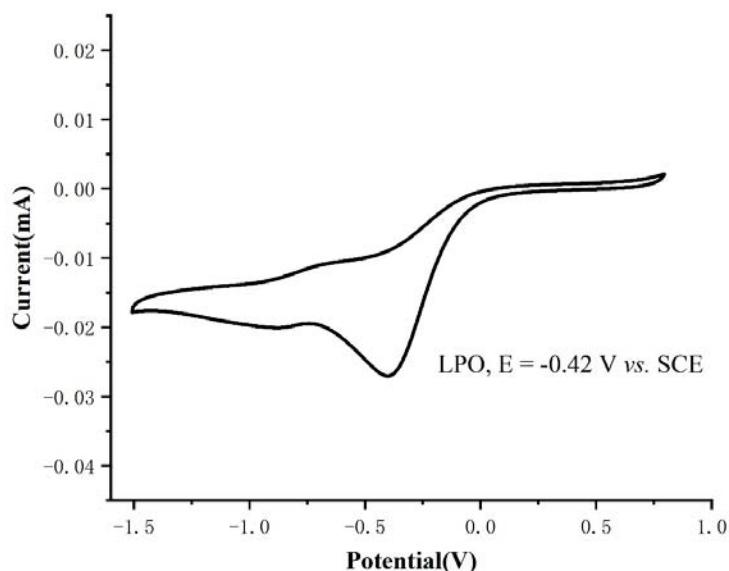
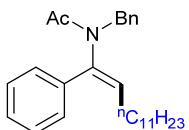
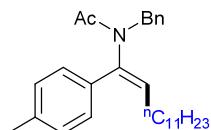


Figure S8. Cyclic voltammetry plots of LPO. Scan direction: from -1.5 V to 0.8 V, then back to -1.5 V

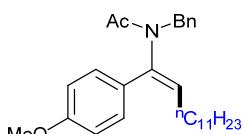
4. Characterization Data for the Products



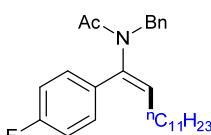
(E)-N-benzyl-N-(1-phenyltridec-1-en-1-yl)acetamide (3aa) (61.2 mg, 75%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.35-7.26 (m, 3H), 7.22-7.12 (m, 7H), 5.23 (t, $J = 7.7$ Hz, 1H), 4.41 (s, 2H), 2.16 (s, 3H), 2.09 (q, $J = 7.5$ Hz, 2H), 1.25-1.11 (m, 18H), 0.81 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 138.1, 137.6, 135.0, 133.1, 129.0, 128.6, 128.5, 128.4, 128.2, 127.1, 49.0, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 28.5, 22.7, 22.3, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{40}\text{NO} [\text{M}+\text{H}^+]$: 406.3104, found 406.3098.



(E)-N-benzyl-N-(1-(p-tolyl)tridec-1-en-1-yl)acetamide (3ba) (62 mg, 74%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.28-7.13 (m, 9H), 5.25 (t, $J = 7.7$ Hz, 1H), 4.47 (s, 2H), 2.38 (s, 3H), 2.21 (s, 3H), 2.15 (q, $J = 7.5$ Hz, 2H), 1.34-1.17 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 138.3, 138.1, 137.7, 132.6, 132.5, 132.1, 129.2, 129.0, 128.5, 128.1, 127.1, 49.0, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 28.5, 22.7, 22.3, 21.3, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{42}\text{NO} [\text{M}+\text{H}^+]$: 420.3261, found 420.3255.

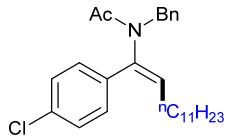


(E)-N-benzyl-N-(1-(4-methoxyphenyl)tridec-1-en-1-yl)acetamide (3ca) (56 mg, 64%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.29-7.17 (m, 7H), 6.92 (d, $J = 8.5$ Hz, 2H), 5.22 (t, $J = 7.6$ Hz, 1H), 4.48 (s, 2H), 3.84 (s, 3H), 2.21 (s, 3H), 2.15 (q, $J = 7.3$ Hz, 2H), 1.29-1.16 (m, 18H), 0.88 (t, $J = 6.7$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 171.0, 159.6, 137.7, 137.7, 132.0, 130.0, 129.1, 128.2, 127.3, 127.2, 113.9, 55.4, 49.0, 32.0, 29.7, 29.6, 29.5, 29.4, 29.4, 29.3, 28.6, 22.7, 22.3, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{42}\text{NO}_2 [\text{M}+\text{H}^+]$: 436.3210, found 436.3209.

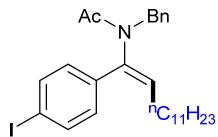


(E)-N-benzyl-N-(1-(4-fluorophenyl)tridec-1-en-1-yl)acetamide (3da) (60.1 mg, 71%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.29-7.17 (m, 7H), 7.07 (t, $J = 8.6$

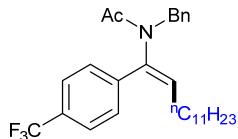
Hz, 2H), 5.30 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.21 (s, 3H), 2.12 (q, $J = 7.4$ Hz, 2H), 1.32-1.15 (m, 18H), 0.88 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 162.5 (d, $J_{\text{C}-\text{F}} = 249.5$ Hz), 137.4 (d, $J_{\text{C}-\text{F}} = 26.3$ Hz), 133.1, 131.1 (d, $J_{\text{C}-\text{F}} = 4.0$ Hz), 130.4 (d, $J_{\text{C}-\text{F}} = 8.1$ Hz), 129.0, 128.3, 127.2, 115.6 (d, $J_{\text{C}-\text{F}} = 21.6$ Hz), 45.0, 31.9, 29.6, 29.6, 29.5, 29.4, 29.4, 29.2, 28.5, 22.7, 22.3, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{39}\text{FNO} [\text{M}+\text{H}^+]$: 424.3010, found 424.3006.



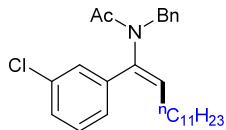
(E)-N-benzyl-N-(1-(4-chlorophenyl)tridec-1-en-1-yl)acetamide (3ea, 65 mg, 74%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.37-7.35 (m, 2H), 7.29-7.15 (m, 7H), 5.33 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.20 (s, 3H), 2.13 (q, $J = 7.5$ Hz, 2H), 1.31-1.17 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.7, 137.4, 137.2, 134.2, 133.6, 129.9, 129.0, 128.8, 128.2, 127.2, 49.1, 31.9, 29.6, 29.5, 29.3, 29.2, 28.5, 22.7, 22.3, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{39}\text{ClNO} [\text{M}+\text{H}^+]$: 440.2715, found 440.2707.



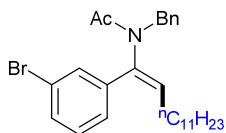
(E)-N-benzyl-N-(1-(4-iodophenyl)tridec-1-en-1-yl)acetamide (3fa, 66 mg, 62%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.71 (d, $J = 8.4$ Hz, 2H), 7.28-7.16 (m, 5H), 6.97 (d, $J = 8.4$ Hz, 2H), 5.33 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.19 (s, 3H), 2.12 (q, $J = 7.5$ Hz, 2H), 1.25-1.17 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.7, 137.7, 137.4, 137.3, 134.6, 133.8, 130.4, 129.0, 128.2, 127.2, 94.2, 49.1, 31.9, 29.6, 29.5, 29.3, 29.2, 28.5, 22.7, 22.3, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{39}\text{INO} [\text{M}+\text{H}^+]$: 532.2071, found 532.2064.



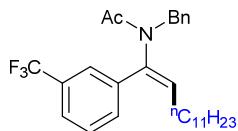
(E)-N-benzyl-N-(1-(4-(trifluoromethyl)phenyl)tridec-1-en-1-yl)acetamide (3ga, 70 mg, 74%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.64 (d, $J = 8.2$ Hz, 2H), 7.35 (d, $J = 8.1$ Hz, 2H), 7.28-7.17 (m, 5H), 5.43 (t, $J = 7.8$ Hz, 1H), 4.50 (s, 2H), 2.21 (s, 3H), 2.16 (q, $J = 7.6$ Hz, 2H), 1.30-1.19 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.7, 138.9, 137.3, 137.1, 135.0, 134.9, 130.4 (q, $J_{\text{C}-\text{F}} = 32.6$ Hz), 129.0, 128.9, 128.2, 127.3, 125.5, 123.9 (q, $J_{\text{C}-\text{F}} = 273.3$ Hz), 49.2, 31.9, 29.6, 29.5, 29.3, 29.2, 28.5, 22.6, 22.2, 22.1, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{39}\text{F}_3\text{NO} [\text{M}+\text{H}^+]$: 474.2978, found 474.2973.



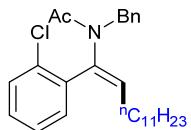
(E)-N-benzyl-N-(1-(3-chlorophenyl)tridec-1-en-1-yl)acetamide (3ha) (59.7 mg, 68%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.33-7.11 (m, 9H), 5.35 (t, $J = 7.7$ Hz, 1H), 4.49 (s, 2H), 2.20 (s, 3H), 2.14 (q, $J = 7.5$ Hz, 2H), 1.29-1.18 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.7, 137.4, 137.1, 137.0, 134.6, 134.3, 129.8, 129.0, 128.5, 128.2, 127.2, 126.8, 49.2, 31.9, 29.6, 29.5, 29.3, 29.2, 28.4, 22.7, 22.3, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{39}\text{ClNO} [\text{M}+\text{H}^+]$: 440.2715, found 440.2707.



(E)-N-benzyl-N-(1-(3-bromophenyl)tridec-1-en-1-yl)acetamide (3ia) (71.4 mg, 74%), yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.49-7.46 (m, 1H), 7.35 (t, $J = 1.7$ Hz, 1H), 7.29-7.15 (m, 7H), 5.35 (t, $J = 7.7$ Hz, 1H), 4.49 (s, 2H), 2.20 (s, 3H), 2.14 (q, $J = 7.5$ Hz, 2H), 1.29-1.18 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.7, 137.4, 137.3, 136.9, 134.3, 134.2, 131.4, 130.0, 129.0, 128.2, 127.3, 122.7, 49.3, 31.9, 29.6, 29.5, 29.3, 29.2, 28.4, 22.7, 22.2, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{39}\text{BrNO} [\text{M}+\text{H}^+]$: 484.2210, found 484.2202.

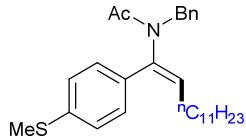


(E)-N-benzyl-N-(1-(3-(trifluoromethyl)phenyl)tridec-1-en-1-yl)acetamide (3ja) (65 mg, 69%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.60 (d, $J = 7.8$ Hz, 1H), 7.51 (t, $J = 7.7$ Hz, 1H), 7.43-7.40 (m, 2H), 7.29-7.16 (m, 5H), 5.44 (t, $J = 7.8$ Hz, 1H), 4.51 (s, 2H), 2.22 (s, 3H), 2.14 (q, $J = 7.5$ Hz, 2H), 1.31-1.19 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.7, 137.2, 137.1, 136.1, 134.6, 134.5, 131.8, 131.0 ($J_{\text{C}-\text{F}} = 32.3$ Hz), 129.1, 129.0, 128.3, 127.3, 125.3, 125.1, 123.8 (q, $J_{\text{C}-\text{F}} = 273.9$ Hz), 49.3, 31.9, 29.6, 29.5, 29.3, 29.2, 28.4, 22.7, 22.3, 22.2, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{39}\text{F}_3\text{NO} [\text{M}+\text{H}^+]$: 474.2978, found 474.2973.

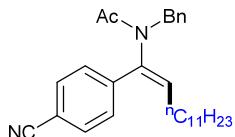


(E)-N-benzyl-N-(1-(2-chlorophenyl)tridec-1-en-1-yl)acetamide (3ka) (48 mg, 55%), colorless oil; ^1H NMR (300 MHz, CDCl_3) δ 7.41 (d, $J = 7.6$ Hz, 1H), 7.31-7.18 (m, 7H), 7.11 (d, $J = 7.0$ Hz, 1H), 5.52 (t, $J = 7.6$ Hz, 1H), 4.43 (s, 2H), 2.37 (s, 3H), 1.93

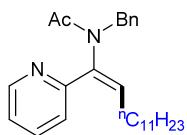
(q, $J = 7.4$ Hz, 2H), 1.30-1.16 (m, 18H), 0.87 (t, $J = 6.5$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 171.0, 137.7, 136.3, 133.7, 133.5, 132.0, 130.2, 129.8, 128.4, 128.1, 126.9, 126.6, 48.5, 31.9, 29.6, 29.5, 29.3, 29.3, 29.2, 28.8, 28.6, 22.7, 22.6, 22.6, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{39}\text{ClNO} [\text{M}+\text{H}^+]$: 440.2715, found 440.2707.



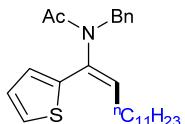
(E)-N-benzyl-N-(1-(4-(methylthio)phenyl)tridec-1-en-1-yl)acetamide (3la, 59.6 mg, 66%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.26-7.15 (m, 9H), 5.27 (t, $J = 7.6$ Hz, 1H), 4.48 (s, 2H), 2.52 (s, 3H), 2.20 (s, 3H), 2.18-2.14 (m, 2H), 1.25-1.18 (m, 18H), 0.88 (t, $J = 6.6$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.9, 139.2, 137.6, 137.6, 133.0, 131.6, 129.0, 128.2, 127.2, 126.0, 49.1, 31.9, 29.6, 29.6, 29.5, 29.4, 29.4, 29.3, 28.6, 22.7, 22.3, 15.4, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{41}\text{NOS} [\text{M}+\text{H}^+]$: 452.2982, found 452.2976.



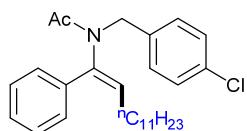
(E)-N-benzyl-N-(1-(4-cyanophenyl)tridec-1-en-1-yl)acetamide (3ma, 58.5 mg, 68%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.68 (d, $J = 8.4$ Hz, 2H), 7.33 (d, $J = 8.4$ Hz, 2H), 7.29-7.24 (m, 3H), 7.16-7.14 (m, 2H), 5.47 (t, $J = 7.8$ Hz, 1H), 4.49 (s, 2H), 2.19 (s, 3H), 2.15 (q, $J = 7.6$ Hz, 2H), 1.30-1.18 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.9, 139.9, 137.0, 136.8, 136.0, 132.4, 129.2, 129.0, 128.4, 127.5, 118.4, 112.1, 49.5, 31.9, 29.6, 29.5, 29.3, 29.3, 29.2, 29.1, 28.6, 24.8, 22.7, 22.2, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{38}\text{N}_2\text{O} [\text{M}+\text{H}^+]$: 431.3057, found 431.3051.



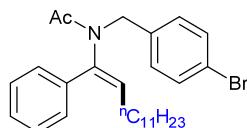
(E)-N-benzyl-N-(1-(pyridin-2-yl)tridec-1-en-1-yl)acetamide (3na, 68.2 mg, 84%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 8.67 (dd, $J = 4.7, 0.8$ Hz, 1H), 7.69-7.65 (m, 1H), 7.27-7.16 (m, 7H), 5.51 (t, $J = 7.7$ Hz, 1H), 4.58 (s, 2H), 2.38 (q, $J = 7.5$ Hz, 2H), 2.16 (s, 3H), 1.31-1.19 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 171.5, 154.4, 149.7, 137.6, 137.5, 137.3, 136.2, 129.2, 128.2, 127.2, 123.0, 122.6, 50.0, 31.9, 29.6, 29.5, 29.3, 29.2, 28.3, 22.7, 22.2, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{27}\text{H}_{38}\text{N}_2\text{O} [\text{M}+\text{H}^+]$: 407.3057, found 407.3048.



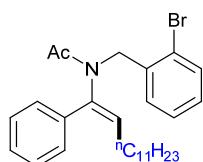
(E)-N-benzyl-N-(1-(thiophen-2-yl)tridec-1-en-1-yl)acetamide (3oa, 51.8 mg, 63%), yellow oil; ^1H NMR (300 MHz, CDCl_3) δ 7.28 (d, $J = 4.7$ Hz, 1H), 7.18-7.14 (m, 5H), 7.00-6.96 (m, 2H), 5.12 (t, $J = 7.6$ Hz, 1H), 4.56 (s, 2H), 2.22 (q, $J = 7.2$ Hz, 2H), 2.04 (s, 3H), 1.18-1.14 (m, 18H), 0.81 (t, $J = 6.2$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 138.8, 137.7, 133.9, 133.8, 132.6, 129.3, 128.2, 127.3, 127.1, 126.9, 126.3, 49.7, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.0, 28.6, 22.7, 21.8, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{38}\text{NOS} [\text{M}+\text{H}^+]$: 412.2669, found 412.2669.



(E)-N-(4-chlorobenzyl)-N-(1-phenyltridec-1-en-1-yl)acetamide (3pa, 59.6 mg, 68%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.33 (m, 3H), 7.27-7.22 (m, 4H), 7.13 (d, $J = 8.4$ Hz, 2H), 5.29 (t, $J = 7.7$ Hz, 1H), 4.43 (s, 2H), 2.22 (s, 3H), 2.17 (q, $J = 7.5$ Hz, 2H), 1.30-1.19 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.9, 138.0, 136.2, 134.8, 133.2, 133.0, 130.4, 128.6, 128.6, 128.3, 48.3, 31.9, 29.6, 29.6, 29.5, 29.4, 29.4, 29.3, 29.3, 28.5, 22.7, 22.2, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{39}\text{ClNO} [\text{M}+\text{H}^+]$: 440.2715, found 440.2709.

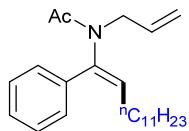


(E)-N-(4-bromobenzyl)-N-(1-phenyltridec-1-en-1-yl)acetamide (3qa, 72.5 mg, 75%), yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.33 (m, 5H), 7.27-7.23 (m, 2H), 7.07 (d, $J = 8.4$ Hz, 2H), 5.29 (t, $J = 7.7$ Hz, 1H), 4.41 (s, 2H), 2.22 (s, 3H), 2.17 (q, $J = 7.5$ Hz, 2H), 1.29-1.19 (m, 18H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.9, 138.0, 136.7, 134.8, 133.2, 131.3, 130.8, 128.6, 128.6, 121.2, 48.4, 31.9, 29.6, 29.6, 29.6, 29.4, 29.4, 29.4, 29.3, 28.5, 22.7, 22.2, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{39}\text{BrNO} [\text{M}+\text{H}^+]$: 484.2210, found 484.2203.

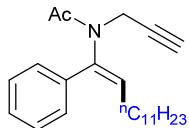


(E)-N-(2-bromobenzyl)-N-(1-phenyltridec-1-en-1-yl)acetamide (3ra, 68 mg, 70%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.47 (d, $J = 8.0$ Hz, 1H), 7.39-7.33 (m, 3H), 7.26-7.20 (m, 4H), 7.10-7.06 (m, 1H), 5.49 (t, $J = 7.7$ Hz, 1H), 4.68 (s, 2H), 2.25 (s, 3H), 2.16 (q, $J = 7.6$ Hz, 2H), 1.29-1.18 (m, 18H), 0.88 (t, $J = 6.8$ Hz, 3H);

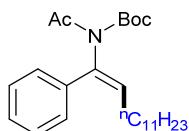
¹³C NMR (101 MHz, CDCl₃) δ 171.2, 138.3, 136.7, 134.9, 133.1, 132.6, 130.8, 128.7, 128.6, 128.5, 127.3, 124.0, 49.2, 31.9, 29.6, 29.6, 29.4, 29.4, 29.2, 28.5, 22.7, 22.3, 14.2; HRMS (ESI) *m/z* calcd for C₂₈H₃₉BrNO [M+H⁺]: 484.2210, found 484.2203.



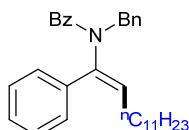
(E)-N-allyl-N-(1-phenyltridec-1-en-1-yl)acetamide (3sa, 50.2 mg, 71%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.33 (m, 3H), 7.28-7.26 (m, 2H), 5.83-5.73 (m, 1H), 5.60 (t, *J* = 7.6 Hz, 1H), 5.10-4.99 (m, 2H), 3.93 (d, *J* = 6.2 Hz, 2H), 2.27 (q, *J* = 7.5 Hz, 2H), 2.17 (s, 3H), 1.46-1.24 (m, 18H), 0.88 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 170.6, 138.7, 135.3, 133.2, 132.3, 128.6, 128.4, 117.5, 48.9, 31.9, 29.6, 29.5, 29.3, 29.3, 28.6, 22.6, 22.3, 22.2, 14.1; HRMS (ESI) *m/z* calcd for C₂₄H₃₈NO [M+H⁺]: 356.2948, found 356.2957.



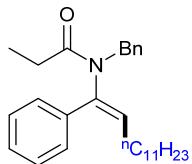
(E)-N-(1-phenyltridec-1-en-1-yl)-N-(prop-2-yn-1-yl)acetamide (3ta, 46.6 mg, 66%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.39-7.29 (m, 5H), 5.77 (t, *J* = 7.6 Hz, 1H), 4.15 (d, *J* = 2.2 Hz, 2H), 2.30 (q, *J* = 7.5 Hz, 2H), 2.15 (t, *J* = 2.5 Hz, 1H), 2.14 (s, 3H), 1.48-1.41 (m, 2H), 1.28-1.23 (m, 16H), 0.86 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 170.5, 138.1, 134.9, 132.9, 128.6, 128.5, 79.2, 71.4, 35.8, 31.9, 29.6, 29.5, 29.4, 29.3, 28.6, 22.7, 22.1, 14.1; HRMS (ESI) *m/z* calcd for C₂₄H₃₆NO [M+H⁺]: 354.2791, found 354.2793.



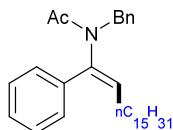
Tert-butyl (E)-acetyl(1-phenyltridec-1-en-1-yl)carbamate (**3ua**, 54 mg, 65%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.32-7.26 (m, 5H), 5.58 (t, *J* = 7.7 Hz, 1H), 2.50 (s, 3H), 2.24 (q, *J* = 7.3 Hz, 2H), 1.35 (s, 9H), 1.29-1.23 (m, 18H), 0.87 (t, *J* = 6.6 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 173.3, 153.0, 136.9, 135.3, 132.2, 128.7, 128.0, 127.7, 82.8, 31.9, 29.7, 29.6, 29.6, 29.5, 29.4, 29.2, 28.5, 27.8, 26.4, 22.7, 14.2; HRMS (ESI) *m/z* calcd for C₂₆H₄₁NO₃ [M+H⁺]: 416.3159, found 416.3154.



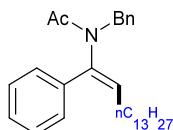
(E)-N-benzyl-N-(1-phenyltridec-1-en-1-yl)benzamide (**3va**, 68.2 mg, 73%), colorless oil; ^1H NMR (300 MHz, CDCl_3) δ 7.54 (d, $J = 7.3$ Hz, 2H), 7.37-7.23 (m, 13H), 5.03 (t, $J = 7.1$ Hz, 1H), 4.72 (s, 2H), 1.93 (q, $J = 7.1$ Hz, 2H), 1.24-1.02 (m, 14H), 0.90-0.79 (m, 7H); ^{13}C NMR (75 MHz, CDCl_3) δ 171.4, 138.5, 137.6, 136.9, 135.4, 133.2, 129.5, 129.0, 128.5, 128.2, 128.2, 127.8, 127.7, 127.2, 50.3, 31.9, 29.6, 29.4, 29.3, 28.9, 28.6, 22.7, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{33}\text{H}_{41}\text{NO}$ [$\text{M}+\text{H}^+$]: 468.3261, found 468.3265.



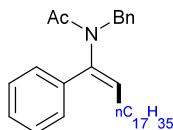
(E)-N-benzyl-N-(1-phenyltridec-1-en-1-yl)propionamide (**3wa**, 57.9 mg, 69%), colorless oil; ^1H NMR (300 MHz, CDCl_3) δ 7.40-7.33 (m, 3H), 7.25-7.20 (m, 7H), 5.29 (t, $J = 7.6$ Hz, 1H), 4.49 (s, 2H), 2.50 (q, $J = 7.4$ Hz, 2H), 2.17 (dd, $J = 14.4, 7.1$ Hz, 2H), 1.25-1.17 (m, 21H), 0.88 (t, $J = 6.4$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 174.3, 137.9, 137.7, 135.3, 133.0, 129.1, 128.6, 128.5, 128.4, 128.2, 127.1, 49.4, 31.9, 29.6, 29.5, 29.5, 29.4, 29.3, 29.2, 28.6, 27.2, 22.7, 14.1, 10.3; HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{41}\text{NO}$ [$\text{M}+\text{H}^+$]: 420.3261, found 420.3256.



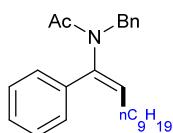
(E)-N-benzyl-N-(1-phenylheptadec-1-en-1-yl)acetamide (**3ab**, 62.4 mg, 68%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.34 (m, 3H), 7.29-7.18 (m, 7H), 5.30 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.22 (s, 3H), 2.16 (q, $J = 7.5$ Hz, 2H), 1.32-1.16 (m, 26H), 0.88 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.9, 138.1, 137.7, 135.1, 133.2, 129.0, 128.7, 128.5, 128.4, 128.2, 127.1, 49.0, 32.0, 29.7, 29.7, 29.7, 29.6, 29.5, 29.4, 29.4, 29.2, 28.5, 22.7, 22.3, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{32}\text{H}_{48}\text{NO}$ [$\text{M}+\text{H}^+$]: 462.3730, found 462.3724.



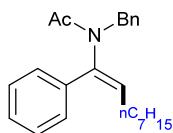
(E)-N-benzyl-N-(1-phenylpentadec-1-en-1-yl)acetamide (**3ac**, 61.2 mg, 71%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.34 (m, 3H), 7.29-7.18 (m, 7H), 5.30 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.22 (s, 3H), 2.16 (q, $J = 7.4$ Hz, 2H), 1.30-1.16 (m, 22H), 0.88 (t, $J = 6.7$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 138.1, 137.6, 135.1, 133.2, 129.0, 128.6, 128.5, 128.4, 128.2, 127.1, 49.0, 31.9, 29.7, 29.7, 29.6, 29.5, 29.4, 29.4, 29.2, 28.5, 22.7, 22.3, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{30}\text{H}_{44}\text{NO}$ [$\text{M}+\text{H}^+$]: 434.3417, found 434.3412.



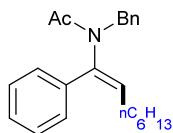
(E)-N-benzyl-N-(1-phenylnonadec-1-en-1-yl)acetamide (3ad, 47.2 mg, 48%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.34 (m, 3H), 7.29-7.18 (m, 7H), 5.30 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.22 (s, 3H), 2.16 (q, $J = 7.4$, 2H), 1.32-1.15 (m, 30H), 0.89-0.86 (m, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 138.1, 137.6, 135.1, 133.2, 133.1, 129.0, 128.6, 128.5, 128.2, 127.1, 49.0, 31.9, 29.7, 29.5, 29.4, 29.2, 28.5, 22.7, 22.3, 22.2, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{34}\text{H}_{52}\text{NO} [\text{M}+\text{H}^+]$: 490.4043, found 490.4046.



(E)-N-benzyl-N-(1-phenylundec-1-en-1-yl)acetamide (3ae, 54.4 mg, 72%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.33 (m, 3H), 7.29-7.18 (m, 7H), 5.30 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.22 (s, 3H), 2.16 (q, $J = 7.6$ Hz, 2H), 1.30-1.18 (m, 14H), 0.87 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 138.1, 137.6, 135.1, 133.1, 129.0, 128.6, 128.5, 128.4, 128.2, 127.1, 49.0, 31.8, 29.5, 29.4, 29.2, 29.2, 28.5, 22.6, 22.3, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{36}\text{NO} [\text{M}+\text{H}^+]$: 378.2791, found 378.2782.

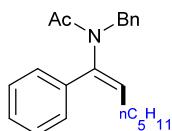


(E)-N-benzyl-N-(1-phenylnon-1-en-1-yl)acetamide (3af, 57.1 mg, 82%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.32 (m, 3H), 7.29-7.19 (m, 7H), 5.30 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.22 (s, 3H), 2.16 (q, $J = 7.5$ Hz, 2H), 1.30-1.18 (m, 10H), 0.85 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 138.1, 137.6, 135.1, 133.1, 129.0, 128.6, 128.5, 128.4, 128.2, 127.1, 49.0, 31.7, 29.4, 29.1, 29.0, 28.5, 22.6, 22.3, 14.1; HRMS (ESI) m/z calcd for $\text{C}_{24}\text{H}_{32}\text{NO} [\text{M}+\text{H}^+]$: 350.2478, found 350.2482.

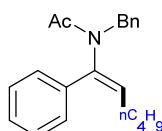


(E)-N-benzyl-N-(1-phenyloct-1-en-1-yl)acetamide (3ag, 52.7 mg, 79%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.32 (m, 3H), 7.29-7.19 (m, 7H), 5.30 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 2.22 (s, 3H), 2.16 (q, $J = 7.5$ Hz, 2H), 1.30-1.15 (m, 8H), 0.84 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 138.1, 137.6, 135.1, 133.1, 133.1, 129.0, 128.6, 128.5, 128.4, 128.2, 127.1, 49.0, 31.6, 29.4, 28.9, 28.5,

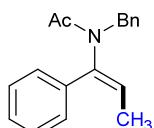
22.5, 22.3, 14.0; HRMS (ESI) m/z calcd for C₂₃H₃₀NO [M+H⁺]: 336.2322, found 336.2318.



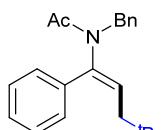
(E)-N-benzyl-N-(1-phenylhept-1-en-1-yl)acetamide (3ah, 52 mg, 81%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.32 (m, 3H), 7.29-7.19 (m, 7H), 5.30 (t, J = 7.7 Hz, 1H), 4.48 (s, 2H), 2.22 (s, 3H), 2.16 (q, J = 7.6 Hz, 2H), 1.33-1.12 (m, 6H), 0.83 (t, J = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 170.8, 138.1, 137.6, 135.0, 133.1, 129.0, 128.6, 128.5, 128.4, 128.2, 127.1, 49.0, 31.4, 29.1, 28.4, 22.4, 22.3, 14.0; HRMS (ESI) m/z calcd for C₂₂H₂₈NO [M+H⁺]: 322.2165, found 322.2166.



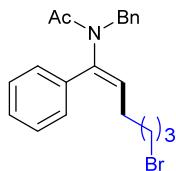
(E)-N-benzyl-N-(1-phenylhex-1-en-1-yl)acetamide (3ai, 48.4 mg, 79%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.32 (m, 3H), 7.29-7.19 (m, 7H), 5.30 (t, J = 7.7 Hz, 1H), 4.48 (s, 2H), 2.22 (s, 3H), 2.17 (q, J = 7.5 Hz, 2H), 1.31-1.15 (m, 4H), 0.81 (t, J = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 170.8, 138.1, 137.6, 135.0, 133.1, 129.0, 128.6, 128.5, 128.4, 128.2, 127.1, 49.0, 31.5, 28.2, 22.3, 22.3, 13.8; HRMS (ESI) m/z calcd for C₂₁H₂₆NO [M+H⁺]: 308.2009, found 308.2006.



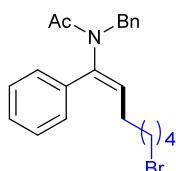
(E)-N-benzyl-N-(1-phenylprop-1-en-1-yl)acetamide⁴ (3aj, 36.6 mg, 69%), colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.40-7.35 (m, 3H), 7.28-7.18 (m, 7H), 5.44 (q, J = 7.2 Hz, 1H), 4.49 (s, 2H), 2.21 (s, 3H), 1.77 (d, J = 7.3 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 170.9, 139.3, 137.7, 134.8, 128.8, 128.7, 128.5, 128.4, 128.2, 127.1, 126.9, 126.8, 49.1, 22.2, 14.5;



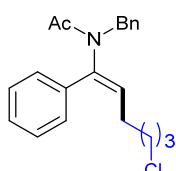
(E)-N-benzyl-N-(4,4-dimethyl-1-phenylpent-1-en-1-yl)acetamide⁵ (3ak, 35.2 mg, 55%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.34 (m, 3H), 7.29-7.20 (m, 7H), 5.40 (t, J = 7.7 Hz, 1H), 4.50 (s, 2H), 2.25 (s, 3H), 2.07 (d, J = 7.7 Hz, 2H), 0.77 (s, 9H); ¹³C NMR (101 MHz, CDCl₃) δ 170.7, 139.1, 137.7, 135.3, 130.4, 128.9, 128.8, 128.5, 128.4, 128.2, 127.2, 49.1, 42.1, 31.4, 29.4, 22.6;



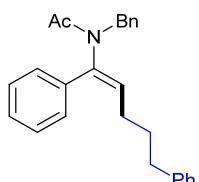
(E)-N-benzyl-N-(6-bromo-1-phenylhex-1-en-1-yl)acetamide (3al, 53.7 mg, 70%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.34 (m, 3H), 7.30-7.18 (m, 7H), 5.28 (t, $J = 7.7$ Hz, 1H), 4.49 (s, 2H), 3.29 (t, $J = 6.7$ Hz, 2H), 2.22 (s, 3H), 2.19 (q, $J = 7.6$ Hz, 2H), 1.75-1.68 (m, 2H), 1.48-1.40 (m, 2H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.6, 138.8, 137.6, 134.8, 131.9, 129.0, 128.6, 128.6, 128.6, 128.2, 127.2, 48.9, 33.3, 32.1, 27.8, 27.6, 22.3; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{25}\text{BrNO} [\text{M}+\text{H}^+]$: 386.1114, found 386.1111.



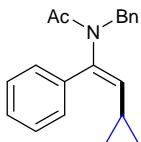
(E)-N-benzyl-N-(7-bromo-1-phenylhept-1-en-1-yl)acetamide (3am, 58.3 mg, 73%), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.34 (m, 3H), 7.29-7.18 (m, 7H), 5.29 (t, $J = 7.7$ Hz, 1H), 4.48 (s, 2H), 3.32 (t, $J = 6.7$ Hz, 2H), 2.23 (s, 3H), 2.21-2.17 (m, 2H), 1.77-1.72 (m, 2H), 1.33-1.29 (m, 4H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.7, 138.5, 137.6, 134.9, 132.4, 129.0, 128.6, 128.5, 128.2, 127.1, 49.0, 33.6, 32.4, 28.5, 28.3, 27.7, 22.3; HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{27}\text{BrNO} [\text{M}+\text{H}^+]$: 400.1271, found 400.1261.



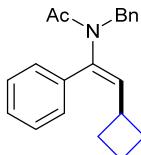
(E)-N-benzyl-N-(6-chloro-1-phenylhex-1-en-1-yl)acetamide (3an, 60 mg, 88%), yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.34 (m, 3H), 7.30-7.18 (m, 7H), 5.28 (t, $J = 7.7$ Hz, 1H), 4.49 (s, 2H), 3.42 (t, $J = 6.5$ Hz, 2H), 2.22 (s, 3H), 2.19 (q, $J = 7.6$ Hz, 2H), 1.66-1.59 (m, 2H), 1.48-1.40 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 170.7, 138.8, 137.6, 134.9, 132.0, 129.0, 128.6, 128.6, 128.2, 127.2, 49.0, 44.6, 31.9, 27.7, 26.6, 22.3; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{25}\text{ClNO} [\text{M}+\text{H}^+]$: 342.1619, found 342.1617.



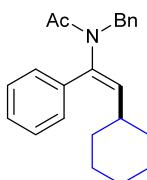
(E)-N-benzyl-N-(1,5-diphenylpent-1-en-1-yl)acetamide⁶ (**3ao**, 61.3 mg, 83%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.39-7.34 (m, 3H), 7.29-7.13 (m, 10H), 7.04 (d, *J* = 7.2 Hz, 2H), 5.32 (t, *J* = 7.7 Hz, 1H), 4.48 (s, 2H), 2.47 (t, *J* = 7.6 Hz, 2H), 2.22-2.17 (m, 5H), 1.64-1.57 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 170.8, 141.7, 138.7, 137.7, 135.0, 132.4, 129.0, 128.6, 128.6, 128.4, 128.3, 127.2, 125.9, 49.1, 35.4, 31.2, 28.0, 22.4.



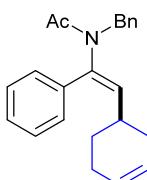
(E)-N-benzyl-N-(2-cyclopropyl-1-phenylvinyl)acetamide (**3ap**, 25 mg, 43%), colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.42-7.36 (m, 5H), 7.26-7.18 (m, 5H), 4.62 (d, *J* = 10.2 Hz, 1H), 4.49 (s, 2H), 2.19 (s, 3H), 1.72-1.60 (m, 1H), 0.75 (d, *J* = 7.7 Hz, 2H), 0.29 (d, *J* = 4.8 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 171.0, 137.6, 137.1, 137.1, 136.6, 135.4, 129.1, 128.6, 128.2, 128.1, 127.1, 49.2, 22.2, 10.9, 7.7; HRMS (ESI) *m/z* calcd for C₂₁H₂₅ClNO [M+H⁺]: 292.1696, found 292.1687.



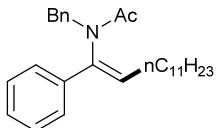
(E)-N-benzyl-N-(2-cyclobutyl-1-phenylvinyl)acetamide⁶ (**3aq**, 48.2 mg, 79%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.34 (m, 3H), 7.29-7.17 (m, 7H), 5.37 (d, *J* = 9.7 Hz, 1H), 4.48 (s, 2H), 3.22-3.12 (m, 1H), 2.18 (s, 3H), 2.11-2.05 (m, 2H), 1.86-1.70 (m, 4H); ¹³C NMR (101 MHz, CDCl₃) δ 170.8, 137.6, 137.6, 136.9, 135.2, 129.2, 128.5, 128.5, 128.2, 127.2, 49.1, 34.3, 29.3, 22.3, 18.6;



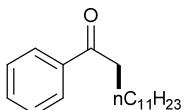
(E)-N-benzyl-N-(2-cyclohexyl-1-phenylvinyl)acetamide⁶ (**3ar**, 62.7 mg, 94%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.42-7.35 (m, 3H), 7.28-7.18 (m, 7H), 5.06 (d, *J* = 10.8 Hz, 1H), 4.45 (s, 2H), 2.37-2.27 (m, 1H), 2.21 (s, 3H), 1.64-1.56 (m, 5H), 1.21-1.08 (m, 3H), 1.02-0.93 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 170.7, 138.6, 137.5, 136.4, 135.3, 129.2, 128.6, 128.4, 128.1, 127.1, 48.8, 37.1, 32.5, 25.8, 25.2, 22.2.



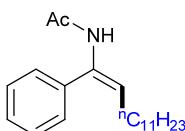
(E)-N-benzyl-N-(2-(cyclohex-3-en-1-yl)-1-phenylvinyl)acetamide (**3as**, 63 mg, 95%), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.34 (m, 3H), 7.29-7.18 (m, 7H), 5.63-5.55 (m, 2H), 5.15 (d, *J* = 10.8 Hz, 1H), 4.52-4.42 (m, 2H), 2.68-2.56 (m, 1H), 2.23 (s, 3H), 2.02-1.94 (m, 2H), 1.74-1.61 (m, 2H), 1.37-1.26 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 170.6, 137.5, 137.5, 137.2, 135.2, 129.2, 128.7, 128.6, 128.5, 128.1, 127.2, 126.9, 125.1, 48.8, 32.8, 31.0, 28.3, 23.9, 22.3; HRMS (ESI) *m/z* calcd for C₂₃H₂₆NO [M+H⁺]: 332.2009, found 332.2004.



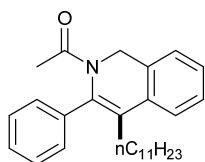
(Z)-N-benzyl-N-(1-phenyltridec-1-en-1-yl)acetamide (**Z-3aa**), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.31 (m, 5H), 7.26-7.22 (m, 5H), 5.95 (dd, *J* = 9.2, 5.4 Hz, 1H), 5.48 (d, *J* = 13.8 Hz, 1H), 3.60 (d, *J* = 13.8 Hz, 1H), 2.06 (s, 3H), 1.77-1.73 (m, 1H), 1.56-1.46 (m, 1H), 1.33-0.96 (m, 18H), 0.89 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 170.9, 137.8, 137.3, 136.3, 130.9, 130.0, 128.9, 128.2, 128.2, 127.5, 125.6, 48.8, 32.0, 29.7, 29.6, 29.5, 29.4, 29.4, 28.6, 28.6, 22.7, 21.3, 14.2; HRMS (ESI) *m/z* calcd for C₂₈H₄₀NO [M+H⁺]: 406.3104, found 406.3098.



1-Phenyltridecan-1-one (**4**), colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 8.00-7.95 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 2H), 2.96 (t, *J* = 7.4 Hz, 2H), 1.77-1.69 (m, 2H), 1.37-1.26 (m, 18H), 0.88 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 200.7, 137.1, 132.9, 128.6, 128.1, 38.7, 32.0, 29.7, 29.7, 29.6, 29.5, 29.4, 29.4, 24.4, 22.7, 14.2; HRMS (ESI) *m/z* calcd for C₁₉H₃₁O [M+H⁺]: 275.2369, found 275.2366.



(E)-N-(1-phenyltridec-1-en-1-yl)acetamide (**5**), colorless oil; ¹H NMR (300 MHz, DMSO) δ 9.09 (s, 1H), 7.39-7.30 (m, 3H), 7.24 (d, *J* = 7.1 Hz, 2H), 5.99 (t, *J* = 7.6 Hz, 0.78H), 5.81 (t, *J* = 7.1 Hz, 0.23H), 1.99-1.96 (m, 2H), 1.89 (s, 3H), 1.30-1.22 (m, 18H), 0.85 (t, *J* = 6.2 Hz, 3H); ¹³C NMR (75 MHz, DMSO) δ 168.7, 137.4, 135.0, 129.0, 128.6, 128.4, 127.9, 125.6, 119.4, 31.8, 30.2, 29.5, 29.4, 29.2, 29.2, 29.0, 27.9, 24.0, 22.6, 14.4; HRMS (ESI) *m/z* calcd for C₂₁H₃₃NO [M+H⁺]: 316.2635, found 316.2624.



1-(3-Phenyl-4-undecylisoquinolin-2(1H)-yl)ethan-1-one (6), colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.43-7.41 (m, 5H), 7.36-7.23 (m, 4H), 4.97 (s, 2H), 2.74 (t, $J = 7.3$ Hz, 2H), 1.46 (s, 3H), 1.38-1.05 (m, 18H), 0.87 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 171.3, 137.8, 135.6, 134.6, 133.5, 130.2, 128.5, 128.1, 128.0, 127.4, 125.4, 124.0, 46.2, 31.9, 29.6, 29.6, 29.5, 29.4, 29.2, 29.1, 28.9, 27.5, 24.3, 22.7, 14.2; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{38}\text{NO} [\text{M}+\text{H}^+]$: 404.2948, found 404.2949.

5. References

1. K. Tang, Y. Chen, J. Guan, Z. Wang, K. Chen, H. Xiang and H. Yang, *Org. Biomol. Chem.*, 2021, **19**, 7475.
2. W. Liu, M. Pu, J. He, T. Zhang, S. Dong, X. Liu, Y.-D. Wu and X. Feng, *J. Am. Chem. Soc.*, 2021, **143**, 11856.
3. W. Yu, J. Chen, K. Gao, Z. Liu and Y. Zhang, *Org. Lett.*, 2014, **16**, 4870.
4. X.-H. Chang, Z.-L. Wang, M. Zhao, C. Yang, J.-J. Li, W.-W. Ma and Y.-H. Xu, *Org. Lett.*, 2020, **22**, 1326.
5. X.-D. Su, B.-B. Zhang, Q. Liu, J.-T. Cheng, Z.-X. Wang and X.-Y. Chen, *Org. Lett.*, 2021, **23**, 8262.
6. J.-Y. Guo, T. Guan, J.-Y. Tao, K. Zhao and T.-P. Loh, *Org. Lett.*, 2019, **21**, 8395.

6. Copies of the ^1H NMR and ^{13}C NMR Spectra

