Supporting Information

Visible light induced 3-position-selective addition of arylpropiolic acids with ethers *via* $C(sp^3)$ -H functionalization

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1. General information

¹H NMR spectra were recorded on Bruker advance III (500 MHz). ¹³C NMR spectra were recorded on Bruker advance III (126 MHz). ¹H and ¹³C chemical shifts are reported relative to the corresponding residual solvent signals (CDCl₃: δ_H 7.26 ppm; δ_C 77.16 ppm). Data for ¹H NMR spectra are reported as follows: chemical shifts, multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet), coupling constant (Hz) and integration. Data for ¹³C NMR spectra are reported in terms of chemical shift. High-resolution mass spectrometry (HRMS) was performed using a ThermoFisher Q-Exactive instrument. Unless otherwise noted, all reagents were obtained commercially and used without further purification. Purification of the reaction products was carried out by flash column chromatography using 200–300 mesh silica gel. Thin layer chromatography (TLC) was visualized by exposure to UV light (254 nm). Reactions were performed under an atmosphere of N₂ using glassware that was dried under vacuum.

2. General methods for preparation of arylcarboxylic acids (1b-10)¹

$$R \xrightarrow{Pd(PPh_3)_4(5 \text{ mol}\%)} + = COOH \xrightarrow{DBU (2 \text{ equiv.})} R \xrightarrow{Pd(PPh_3)_4(5 \text{ mol}\%)} R \xrightarrow{Pd(PPh_3$$

To a 100 mL of round-bottomed flask were added Pd(PPh₃)₄ (577.8 mg, 5 mol%), aryl iodide (10.0 mmol, 1.0 equiv.), DBU (3.040 g, 20 mmol, 2.0 equiv.) and DMSO (15 mL). Then the solution of propiolic acid (771 mg, 11 mmol, 1.1 equiv.) in DMSO (5 mL) was added dropwise. The flask was put into a preheated oil bath (35°C). After stirring for 10 h, the reaction mixture was cooled to room temperature. The reaction mixture was diluted with EtOAc, and extracted with saturated aqueous NaHCO₃ solution. The aqueous layer was separated, acidified to pH=2.0 by cold HCl (1N), and extracted with CH₂Cl₂. The combined organic layers were dried with Na₂SO₄, filtered, and the solvent was removed under reduced pressure. The resulting crude product was purified by flash chromatography on silica gel (PE/EA = 2:1 with HOAc (1%, v/v)) to give **1b-1o**.

3. Photoredox reaction of arylpropiolic acids with ethers

$$Ar = COOH + R = COOH + R = COOH + R = COOH = COOH$$

A 25 mL Schlenk tube equipped with a magnetic stir bar was charged with arylpropiolic acid (0.25 mmol, 1.0 equiv.), $Ir(bpy)_4Cl_2$ (6 mg, 0.005 mmol, 0.02 equiv.), TBHP (0.1 mL, 0.5 mmol, 2 equiv., 5.5 M in decane) and ether (3 mL). The tube was sealed with a septum, evacuated, and

backfilled with nitrogen three times. The mixture was stirred at room temperature for 12 h with an 18 W blue LED light. Then 0.1 mL DBU was added and stirred at room temperature for 30 min. The reaction mixture was diluted with EtOAc, and extracted with saturated aqueous NaHCO₃ solution. The aqueous layer was separated, acidified to pH = 2.0 by cold HCl (1 N), and extracted with CH₂Cl₂. The combined organic layers were dried over Na₂SO₄, filtered, and the solvent was removed under reduced pressure. The resulting crude product was purified by silica gel column chromatography or preparative TLC plate.

4. Transformation of Compound 3a

4.1 Esterification of 3a



To a solution of **3a** (218 mg, 1 mmol) in MeOH (5 mL) was added dropwise $SOCl_2$ (0.15 mL, 2 mmol) at 0°C under a nitrogen atmosphere. Then the reaction was warmed to room temperature and stirred overnight. After the reaction was complete, the solvent was concentrated under reduced pressure and the crude product was purified by silica gel column chromatography.

4.2 Amidation of 3a



A 10 mL Schlenk tube equipped with a magnetic stir bar was charged with **3a** (109 mg, 0.5 mmol). Then SOCl₂ (0.5 mL) was added slowly by a syringe. The mixture was stirred at room temperature for 4 h. After evaporation of the excess SOCl₂, the residue was added dropwise to 25% aq. NH₃ solution (4.0 mL) at 0°C, and the mixture was stirred at room temperature for 30 min. Then the solution was extracted with ethyl acetate. The combined organic layers were dried with Na₂SO₄, filtered, and the solvent was removed under reduced pressure. The resulting crude product was purified by silica gel column chromatography.

4.3 Reduction of ester 4



To a 25 mL round-bottom flask was added LiAlH₄ (190 mg, 5 mmol) and anhydrous THF (5 mL), and the solution of **4** (116 mg, 0.5 mmol) in THF (2 mL) was added dropwise under a nitrogen atmosphere at 0°C. The reaction was stirred at room temperature, and the reaction was monitored by TLC. After the substrate **4** was disappeared, ice water (0.19 mL) was added dropwise at 0°C, and then a 15% aqueous NaOH solution (0.19 mL) was added and the reaction mixture was stirred for 2 h, then 0.6 mL of water was added. After that, anhydrous sodium sulfate was added to remove water. After filtration over celite, the filtrate was concentrated under reduced pressure. The target product was obtained by silica gel column chromatography (PE/EtOAc).

4.4 Oxidation of allyl alcohol 6



The compound **6** (61 mg, 0.3 mmol) and NaHCO₃ (100 mg, 1.2 mmol) were suspended in dry CH₂Cl₂ (0.5 mL) and cooled to 0°C prior to the addition of DMP (190 mg, 0.45 mmol). After 30 minutes the mixture was allowed to be warmed to room temperature and let stirring overnight. The reaction was quenched by diluting with CH₂Cl₂ (5 mL) and pouring into saturated Na₂S₂O₃ (aq.). The aqueous layer was extracted with CH₂Cl₂ (2 × 10 mL) and the combined organic layers were dried over Na₂SO₄. Removal of the solvent under reduced pressure gave the crude product which was purified by flash chromatography (PE/EtOAc).

4.5 Esterification of 3a with diosgenin



To a solution of 3a (83 mg, 0.2 mmol), 8 (652 mg, 0.24 mmol) and DMAP (2 mg, 0.01 mmol) in CH₂Cl₂ (5 mL) was added dropwise a solution of DCC (50 mg, 0.24 mmol) in CH₂Cl₂ (5 mL) at 0°C under a nitrogen atmosphere. Then the reaction was warmed to room temperature and stirred for another 2 h. The reaction was monitored by TLC to establish the consumption of starting material. After the reaction was complete, the solid was removed by filtration. The filtrate was concentrated under reduced pressure and the crude product was purified by silica gel column chromatography to provide the desired product.

5. Characterization data for the products



3-phenyl-3-(tetrahydrofuran-2-yl)acrylic acid (3a)

¹H NMR (CDCl₃, 500 MHz): $\delta = 8.66$ (br, 3H), 7.88 (s, 2H), 7.42 – 7.28 (m, 15H), 6.97 (s, 1H), 4.93 (t, J = 7.8 Hz, 2H), 4.76 – 4.72 (m, 1H), 4.14 – 4.08 (m, 2H), 4.06 – 4.00 (m, 1H), 3.92-3.89 (m, 1H), 3.81 – 3.85 (m, 2H), 2.23 – 2.27 (m, 1H), 2.12 – 2.19 (m, 6H), 2.05 – 1.94 (m, 5H).

¹³C NMR (CDCl₃, 126 MHz): δ = 172.29, 170.92, 143.67, 135.16, 134.46, 134.20, 133.95, 131.50, 129.25, 129.00, 128.72, 128.51, 128.30, 128.25, 79.78, 75.27, 69.07, 68.84, 31.89, 31.42, 26.77, 25.64;

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₃H₁₅O₃⁺: 219.1016, found: 219.1015.



3-(tetrahydrofuran-2-yl)-3-(o-tolyl)acrylic acid (3b)

¹H NMR (CDCl₃, 500 MHz): δ = 7.91 (s, 2H), 7.27 – 7.12 (m, 12H), 7.07 – 7.08 (m, 2H), 4.78 – 4.81 (m, 1H), 4.70 - 4.73 (m, 2H), 4.12 – 4.03 (m, 3H), 3.89 – 3.93 (m, 1H), 3.83 – 3.78 (m, 2H), 2.29 (s, 6H), 2.27 (s, 3H), 2.22 – 2.24 (m, 1H), 2.06 – 2.12 (m, 5H), 2.02 – 1.88 (m, 6H).

¹³C NMR (126 MHz, CDCl₃): δ = 169.53, 143.20, 137.00, 135.88, 135.73, 135.33, 134.46, 133.88, 131.33, 130.32, 129.83, 129.03, 128.58, 128.39, 128.23, 125.74, 125.70, 79.48, 76.04, 69.01, 68.90, 32.22, 31.79, 26.42, 25.68, 20.11, 20.04.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₄H₁₇O₃⁺: 233.1172, found: 233.1174.



3-(tetrahydrofuran-2-yl)-3-(*m*-tolyl)acrylic acid (3c)

¹H NMR (CDCl₃, 500 MHz): $\delta = 8.07$ (br, 3H), 7.85 (s, 2H), 7.26 – 7.29 (m, 2H), 7.16 – 7.19 (m, 6H), 7.08 – 7.10 (m, 5H), 6.90 (s, 1H), 4.91 – 4.94 (m, 2H), 4.72 – 4.75 (m, 1H), 4.13 – 4.06 (m, 2H), 4.06 – 3.98 (m, 1H), 3.93 – 3.86 (m, 1H), 3.80 – 3.85 (m, 2H), 2.37 (s, 6H), 2.32 (s, 3H), 2.21 – 2.25 (m, 1H), 2.15 (m, 6H), 2.03 – 1.92 (m, 5H).

¹³C NMR (CDCl₃, 126 MHz): δ = 170.57, 143.75, 138.23, 137.82, 135.14, 134.48, 134.09, 133.94, 131.24, 129.94, 129.79, 129.48, 129.12, 128.41, 128.22, 126.24, 125.75, 80.01, 75.51, 69.05, 68.84, 31.84, 31.57, 26.67, 25.69, 21.49, 21.41.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₄H₁₇O₃⁺: 233.1172, found: 233.1173.



(Z)-3-(tetrahydrofuran-2-yl)-3-(p-tolyl)acrylic acid (Z-3d)

¹H NMR (CDCl₃, 500 MHz): $\delta = 11.05$ (br, 1H), 7.85 (s, 1H), 7.21 (d, J = 7.8 Hz, 2H), 7.17 (d, J = 7.9 Hz, 2H), 4.99 – 4.95 (m, 1H), 4.19 – 4.15 (m, 1H), 3.88 – 3.84 (m, 1H), 2.38 (s, 3H), 2.27 – 2.22 (m, 1H), 2.19 – 2.13 (m, 1H), 2.10 – 1.97 (m, 2H).

¹³C NMR (CDCl₃, 126 MHz): δ = 168.56, 143.40, 139.47, 131.53, 129.43, 129.41, 129.34, 76.33, 68.99, 32.09, 26.14, 21.50.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₄H₁₇O₃⁺: 233.1172, found: 233.1171.



(E)-3-(tetrahydrofuran-2-yl)-3-(p-tolyl)acrylic acid (E-3d)

¹H NMR (CDCl₃, 500 MHz): δ = 7.30 (d, *J* = 7.9 Hz, 2H), 7.13 (d, *J* = 7.9 Hz, 2H), 6.90 (s, 1H), 4.70 - 4.64 (m, 1H), 4.07 - 4.00 (m, 1H), 3.92 - 3.88 (m, 1H), 2.34 (s, 3H), 2.26 - 2.20 (m, 1H), 2.03 - 1.92 (m, 3H).

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₄H₁₇O₃⁺: 233.1172, found: 233.1171.



3-(o-methoxyphenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (3e)

¹H NMR (CDCl₃, 500 MHz): $\delta = 10.03$ (br, 2.7H), 7.98 (s, 1.7H), 7.37 – 7.33 (m, 1.7), 7.32 – 7.30 (m, 1H), 7.29 – 7.25 (m, 1.7H), 7.17 – 7.16 (m, 1H), 7.08 (s, 1H), 6.97 – 6.94 (m, 1.7H), 6.91- 6.88 (m, 3H), 6.86 – 6.84 (m, 1H), 4.87- 4.84 (m, 1.7H), 4.78 – 4.75 (m, 1H), 4.15 – 4.07 (m, 1.7H), 4.06 – 4.02 (m, 1H), 3.92 – 3.88 (m, 1H), 3.84 – 3.78 (m, 11H), 2.28 – 2.22 (m, 1H), 2.17 – 2.09 (m, 5H), 2.04 – 1.93 (m, 5H).

¹³C NMR (CDCl₃, 126 MHz): δ = 172.19, 170.35, 157.79, 156.98, 140.39, 133.79, 131.37, 130.75, 130.16, 130.11, 129.84, 124.50, 123.58, 120.37, 120.21, 110.72, 110.59, 80.07, 75.99, 68.97, 68.81, 55.56, 55.29, 31.96, 31.46, 26.62, 25.73.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₄H₁₇O₄⁺: 249.1121, found: 249.1120.



3-(m-methoxyphenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (3f)

¹H NMR (CDCl₃, 500 MHz): δ = 7.83 (s, 2H), 7.32 – 7.29 (m, 2H), 7.23 – 7.20 (m, 1H), 6.98 – 6.93 (m, 2H), 6.91 – 6.89 (m, 2H), 6.88 (s, 2H), 6.87 (s, 1H), 6.84 – 6.81 (m, 3H), 4.94 – 4.91 (m, 2H), 4.75 – 4.72 (m 1H), 4.12- 4.00 (m, 2H), 3.93 – 3.87 (m, 1H), 3.93 – 3.87 (m, 1H), 3.82 (s, 8H), 3.77 (s, 3H), 2.27 – 2.20 (m, 1H), 2.18 – 2.11 (m, 6H), 2.03 – 1.93 (m, 6H)

¹³C NMR (CDCl₃, 126 MHz): δ = 171.92, 170.11, 159.58, 159.43, 143.20, 136.62, 135.87, 134.69, 133.20, 131.69, 129.61, 129.33, 121.55, 121.31, 114.69, 114.59, 114.36, 113.81, 80.00, 77.42, 75.64, 69.08, 68.87, 55.39, 55.28, 31.83, 31.63, 26.62, 25.72.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₄H₁₇O₄⁺: 249.1121, found: 249.1124.



(Z)-3-(p-methoxyphenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (Z-3g)

¹H NMR (CDCl₃, 500 MHz): δ = 7.84 (s, 1H), 7.28 (d, *J* = 8.4 Hz, 2H), 6.93 (d, *J* = 8.7 Hz, 2H), 5.04 - 4.96 (m, 1H), 4.18 - 4.11 (m, 1H), 3.88 - 3.82 (m, 4H), 2.23 - 2.11 (m, 3H), 2.07 - 2.01 (m, 1H).

¹³C NMR (CDCl₃, 126 MHz): δ = 170.32, 160.44, 143.40, 131.28, 129.00, 126.90, 114.11, 75.81, 69.01, 55.46, 31.60, 26.56.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₄H₁₇O₄⁺: 249.1121, found: 249.1122.



(E)-3-(p-methoxyphenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (E-3g)

¹H NMR (CDCl₃, 500 MHz): δ = 7.39 (d, *J* = 8.8 Hz, 2H), 6.88 (s, 1H), 6.84 (d, *J* = 8.8 Hz, 2H), 4.70 - 4.67 (m, 1H), 4.05 - 4.01 (m, 1H), 3.91 - 3.87 (m, 1H), 3.80 (s, 3H), 2.28 - 2.20 (m, 1H), 2.03 - 1.92 (m, 3H);

¹³C NMR (CDCl₃, 126 MHz): δ = 171.02, 160.00, 135.20, 131.01, 130.84, 127.37, 113.80, 80.90, 68.88, 55.39, 31.88, 25.85.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₄H₁₇O₄⁺: 249.1121, found: 249.1122.



3-(o-fluorophenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (3h)

¹H NMR (CDCl₃, 500 MHz): $\delta = 9.36$ (br, 2H), 7.85 (s, 1H), 7.37 – 7.33 (m, 2H), 7.28 – 7.24 (m, 2H), 7.18 – 7.16 (m, 1H), 7.12 – 6.99 (m, 4H), 4.82 – 4.79 (m, 2H), 4.08 – 4.03 (m, 2H), 3.92 (q, J = 7.1 Hz, 1H), 3.84 – 3.81 (m, 1H), 2.31 – 2.26 (m, 1H), 2.22 – 2.13 (m, 3H), 2.05 – 1.92 (m, 4H). ¹³C NMR (CDCl₃, 126 MHz): $\delta = 171.74$, 170.31, 161.25, 161.04, 159.26, 159.06, 136.65, 136.13, 135.55, 135.47, 133.64, 132.82, 131.04, 130.98, 130.65, 130.62, 130.29, 130.26, 130.10, 130.03, 128.35, 124.16, 124.09, 124.06, 123.88, 123.86, 123.62, 123.50, 122.58, 122.46, 117.30, 117.13, 115.94, 115.76, 115.54, 115.37, 79.40, 75.86, 69.15, 68.94, 32.14, 31.23, 26.69, 25.65.

HRMS (ESI): *m*/*z* [M+Na]⁺ calcd for C₁₃H₁₄FO₃⁺: 237.0921, found: 237.0922.



3-(*m*-fluorophenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (3i)

¹H NMR (CDCl₃, 500 MHz): δ = 8.29 (br, 3H), 7.80 (s, 1.8H), 7.38 – 7.34 (m, 1.8H), 7.28 – 7.25 (m, 1H), 7.14 – 7.12 (m, 1H), 7.10 – 7.04 (m, 4.6H), 7.02 – 7.00 (m 1.8H), 6.98 – 6.94 (m, 1H),

6.92 (s, 1H), 4.92 – 4.85 (m, 1.8H), 4.77 – 4.74 (m, 1H), 4.12 – 4.05 (m, 1.8H), 4.06 – 4.00 (m, 1H), 3.93 – 3.88 (m, 1H), 3.89 – 3.82 (m, 1.8H), 2.28 – 2.23 (m, 1H), 2.22 – 2.11 (m, 5H), 2.04 – 1.93 (m, 5H).

¹³C NMR (CDCl₃, 126 MHz): = 171.64, 170.44, 163.60, 163.58, 161.64, 141.97, 137.54, 136.64, 136.58, 135.44, 132.80, 132.65, 132.64, 130.20, 130.14, 129.80, 129.74, 125.04, 125.01, 124.54, 124.51, 116.10, 115.99, 115.93, 115.82, 115.56, 115.39, 115.21, 115.04, 79.56, 75.27, 69.16, 68.92, 31.92, 31.50, 26.73, 25.65.

HRMS (ESI): *m*/*z* [M+Na]⁺ calcd for C₁₃H₁₄FO₃⁺: 237.0921, found: 237.0919.



3-(*p*-fluorophenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (3j)

¹H NMR (CDCl₃, 500 MHz): δ = 7.83 (s, 1.7 H), 7.37 (dd, *J* = 8.7, 5.4 Hz, 2H), 7.29 (dd, *J* = 8.5, 5.4 Hz, 3.3H), 7.09 (t, *J* = 8.6 Hz, 3.3H), 7.00 (t, *J* = 8.4 Hz, 2H), 6.93 (s, 1H), 4.89 (t, *J* = 7.7 Hz, 2H), 4.73 (t, *J* = 6.9 Hz, 1H), 4.13 – 4.09 (m, 2H), 4.05 – 4.01 (m, 1H), 3.93 – 3.90 (m, 1H), 3.88 – 3.82 (m, 2H), 2.28 – 2.24 (m, 1H), 2.18 – 2.10 (m, 5H), 2.05 – 1.86 (m, 6H).

¹³C NMR (CDCl₃, 126 MHz): = 171.92, 170.62, 164.07, 163.68, 162.08, 161.70, 142.52, 133.78, 133.68, 131.43, 131.35, 131.28, 130.81, 130.75, 130.57, 130.54, 130.36, 115.81, 115.64, 115.39, 115.22, 79.84, 75.33, 69.13, 68.91, 31.99, 31.47, 26.72, 25.72.

HRMS (ESI): *m*/*z* [M+Na]⁺ calcd for C₁₃H₁₄FO₃⁺: 237.0921, found: 239.0922.



(Z)-3-(p-chlorophenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (Z-3k)

¹H NMR (CDCl₃, 500 MHz): δ = 7.78 (s, 1H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 4.88 – 4.84 (m, 1H), 4.19 – 4.13 (m, 1H), 3.88 – 3.84 (m, 1H), 2.21 – 2.15 (m, 2H), 2.08 – 2.00 (m, 2H).

¹³C NMR (CDCl₃, 126 MHz): = 168.32, 141.92, 133.30, 131.97, 131.86, 130.81, 130.71, 69.13, 32.02, 26.22.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₃H₁₄ClO₃⁺: 253.0626, found: 253.0624.



(E)-3-(p-chlorophenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (E-3k)

¹H NMR (CDCl₃, 500 MHz): δ = 7.44 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 7.8 Hz, 2H), 6.89 (s, 1H), 4.70 – 4.67 (m, 1H), 4.06 – 4.01 (m, 1H), 3.93 – 3.89 (m, 1H), 2.29 – 2.22 (m, 1H), 2.04 – 1.97 (m, 2H), 1.95 – 1.88 (m, 1H).

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₃H₁₄ClO₃⁺: 253.0626, found: 253.0622.



3-(p-(methoxycarbonyl)phenyl)-3-(tetrahydrofuran-2-yl)acrylic acid (31)

¹H NMR (CDCl₃, 500 MHz): $\delta = 10.11$ (br, 4H), 8.07 – 8.05 (m, 7H), 7.99 – 7.95 (m, 2H), 7.87 (s, 3.5H), 7.42 (d, J = 7.5 Hz, 2H), 7.37 (d, J = 7.8 Hz, 7H), 7.01 (s, 1H), 4.87 – 4.84 (m, 3.5H), 4.79 – 4.76 (m, 1H), 4.11 – 4.07 (m, 3.5H), 4.05 – 4.02 (m, 1H), 3.94 (s, 9H), 3.91 (s, 3H), 3.86 – 3.82 (m, 3H), 2.31 – 2.27 (m, 1H), 2.19 – 2.13 (m, 10H), 2.03 – 1.91 (m, 7H).

¹³C NMR (126 MHz, CDCl₃) δ 169.85, 166.90, 166.63, 142.05, 140.16, 139.10, 136.15, 133.13, 132.31, 132.23, 130.36, 129.76, 129.55, 129.13, 128.73, 128.66, 79.56, 75.51, 69.19, 68.97, 52.42, 52.25, 31.98, 31.73, 26.61, 25.71.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₅H₁₇O₅⁺: 277.1071, found: 277.1072.



3-(tetrahydrofuran-2-yl)-3-(p-(trifluoromethyl)phenyl)acrylic acid (3m)

¹H NMR (CDCl₃, 500 MHz): $\delta = 7.87$ (s, 1.8H), 7.66 (d, J = 8.0 Hz, 3.6H), 7.57 (d, J = 8.1 Hz, 2H), 7.46 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 3.6H), 7.03 (s, 1H), 4.82 (t, J = 7.7 Hz, 1.8H), 4.77 (t, J = 6.9 Hz, 1H), 4.11 – 4.02 (m, 2.8H), 3.94 – 3.90 (m, 1H), 3.86 – 3.83 (m, 1.8H), 2.33 – 2.26 (m, 1H), 2.19 – 2.11 (m, 6H), 2.05 – 1.89 (m, 6H).

¹³C NMR (CDCl₃, 126 MHz): = 171.64, 170.50, 141.78, 139.16, 138.19, 136.23, 133.72, 133.14, 130.93, 130.67, 129.42, 128.94, 125.55, 125.52, 125.49, 125.46, 125.25, 125.22, 125.19, 125.16, 125.07, 122.90, 79.36, 77.42, 77.16, 76.91, 75.26, 69.22, 68.99, 32.06, 31.58, 26.76, 25.69.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₄H₁₄F₃O₃⁺: 287.0890, found: 287.0889.



3-([1,1'-biphenyl]-4-yl)-3-(tetrahydrofuran-2-yl)acrylic acid (3n)

¹H NMR (CDCl₃, 500 MHz): δ = 7.92 (s, 1.5H), 7.63 – 7.54 (m, 11H), 7.48 – 7.37 (m, 12H), 7.00 (s, 1H), 5.01 (t, *J* = 7.6 Hz, 1.5H), 4.77 (t, *J* = 6.4 Hz, 1H), 4.15 – 4.10 (m, 1.5H), 4.10 – 4.01 (m, 1H), 3.93 – 3.89 (m, 1H), 3.88 – 3.83 (m, 1.5H), 2.32 – 2.23 (m, 1H), 2.22 – 2.14 (m, 5H), 2.06 – 1.94 (m, 5H).

¹³C NMR (CDCl₃, 126 MHz): = 171.92, 170.31, 143.26, 141.95, 141.20, 140.59, 140.26, 134.42, 134.06, 133.57, 133.40, 131.12, 129.93, 129.48, 129.03, 128.89, 127.91, 127.59, 127.26, 127.17, 127.13, 127.01, 80.20, 77.41, 75.69, 69.12, 68.94, 31.98, 31.68, 26.64, 25.78.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₉H₁₉O₃+: 295.1329, found: 295.1330.



3-(tetrahydrofuran-2-yl)-3-(thiophen-2-yl)acrylic acid (30)

¹H NMR (CDCl₃, 500 MHz): $\delta = 9.31$ (br, 3H), 7.97 (s, 2.4H), 7.53 (d, J = 5.2 Hz, 2.4H), 7.45 (d, J = 5.1 Hz, 1H), 7.37 (d, J = 2.5 Hz, 1H), 7.28 (d, J = 3.8 Hz, 2.4H), 7.12 – 7.10 (m, 2.4H), 7.04 – 7.03 (m, 1H), 5.36 – 5.33 (m, 2.4H), 4.77 (t, J = 7.0 Hz, 1H), 4.23 – 4.19 (m, 2.4H), 4.10 – 4.05 (m, 1H), 3.99 – 3.91 (m, 3.4H), 2.44 – 2.38 (m, 2.4H), 2.35 – 2.28 (m, 1H), 2.24 – 2.08 (m, 6H), 2.02 – 1.92 (m, 5H), 1.87 – 1.82 (m, 1H).

¹³C NMR (CDCl₃, 126 MHz): =170.38, 169.31, 137.55, 136.75, 134.81, 134.15, 130.84, 127.85, 126.74, 126.34, 79.93, 76.81, 69.08, 68.88, 32.70, 31.27, 26.21, 25.75.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₁H₁₃O₃S⁺: 225.0580, found: 225.0578.



3-(naphthalen-1-yl)-3-(tetrahydrofuran-2-yl)acrylic acid (3p)

¹H NMR (CDCl₃, 500 MHz): $\delta = 8.34$ (s, 2H), 7.92 – 7.83 (m, 9H), 7.81 – 7.79 (m, 1H), 7.60 (s, 1H), 7.55 – 7.53 (m, 4H), 7.50 - 7.47 (m, 4H), 7.42 – 7.41 (m, 1H), 7.29 - 7.28 (m, 2H), 4.90 (t, J = 6.4 Hz, 1H), 4.73 (t, J = 7.9 Hz, 2H), 4.11 – 4.07 (m, 2H), 3.99 – 3.94 (m, 1H), 3.78 – 3.74 (m, 3H), 2.38 – 2.33 (m, 1H), 2.13 – 2.00 (m, 7H), 1.97 – 1.88 (m, 4H).

¹³C NMR (CDCl₃, 126 MHz): = 168.95, 142.15, 136.04, 134.79, 133.52, 133.46, 133.44, 132.79, 131.92, 131.39, 131.30, 129.43, 128.64, 128.61, 126.91, 126.62, 126.38, 126.25, 126.09, 125.45, 125.15, 124.96, 124.62, 79.54, 77.41, 76.47, 69.02, 69.01, 32.35, 32.03, 26.29, 25.83.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₇H₁₇O₃⁺: 269.1172, found: 269.1172.



4-ethoxy-3-phenylpent-2-enoic acid (3q)

¹H NMR (CDCl₃, 500 MHz): δ = 7.91 (s, 1.4H), 7.44 – 7.29 (m, 10H), 7.26 - 7.25 (m, 2H), 6.86 (s, 1H), 4.75 – 4.71 (m, 1.4H), 4.26 – 4.22 (m, 1H), 3.70 – 3.64 (m, 1H), 3.56 – 3.47 (m, 2.4H), 3.41 – 3.35 (m, 1.4H), 1.56 (d, *J* = 6.7 Hz, 4.6H), 1.46 (d, *J* = 6.5 Hz, 3H), 1.24 – 1.25 (m, 3H), 1.17 (t, *J* = 7.1 Hz, 5H).

¹³C NMR (CDCl₃, 126 MHz): = 142.16, 139.49, 131.83, 130.35, 128.14, 127.42, 127.16, 126.08, 125.77, 124.28, 123.67, 123.02, 79.42, 75.80, 68.32, 68.27, 32.54, 32.47, 26.38, 26.02.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₃H₁₇O₃⁺: 221.1172, found: 221.1173.



4-butoxy-3-phenylhept-2-enoic acid (3r)

¹H NMR (CDCl₃, 500 MHz): $\delta = 10.84$ (br, 3H), 7.93 (s, 1H), 7.42 – 7.39 (m, 8H), 7.35 – 7.28 (m, 12H), 6.84 (s, 3H), 4.55 – 4.52 (m, 1H), 4.03 – 4.00 (m, 3H), 3.67 – 3.63 (m, 3H), 3.54 – 3.50 (m, 1H), 3.41 – 3.36 (m, 3H), 3.27 – 3.23 (m, 1H), 1.98 – 1.92 (m, 1H), 1.76 – 1.66 (m, 7H), 1.63 – 1.56 (m, 7H), 1.56 – 1.47 (m, 7H), 1.45 – 1.38 (m, 12H), 1.37 – 1.29 (m, 4H), 0.96 – 0.91 (m, 25H).

¹³C NMR (CDCl₃, 126 MHz): = 171.39, 168.89, 143.29, 136.27, 134.84, 134.44, 133.21, 131.26, 129.29, 129.15, 128.95, 128.76, 128.72, 128.45, 82.54, 77.42, 76.38, 70.05, 69.42, 37.48, 36.93, 31.96, 31.57, 26.58, 19.52, 19.30, 19.21, 19.13, 14.02, 14.00, 13.86.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₇H₂₅O₃⁺: 277.1798, found: 277.1797.



4-(tert-butoxy)-3-phenylbut-2-enoic acid (3s)

¹H NMR (CDCl₃, 500 MHz): $\delta = 10.87$ (br 2), 8.10 (d, J = 7.3 Hz, 1H), 8.01 (s, 1.6H), 7.60 – 7.57 (m, 3.2H), 7.48 (d, J = 7.6 Hz, 1H), 7.41 – 7.39 (m, 5H), 7.34 – 7.29 (m, 3H), 7.05 (s, 1H), 4.27 (s, 3.2H), 4.26 (d, J = 1.6 Hz, 2H), 1.31 (s, 14.8H), 1.28 (s, 9H).

¹³C NMR (CDCl₃, 126 MHz): = 172.59, 145.90, 137.95, 135.27, 134.81, 133.87, 130.31, 130.09, 129.65, 128.99, 128.58, 128.54, 128.27, 74.60, 74.57, 63.99, 56.81, 27.76, 27.65.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₄H₁₉O₃⁺: 235.1329, found: 235.1326.



(Z)-4,5-dimethoxy-3-phenylpent-2-enoic acid (Z-3t)

¹H NMR (CDCl3, 500 MHz): δ = 8.05 (s, 1H), 7.43 – 7.36 (m, 5H), 4.68 – 4.65 (m, 1H), 3.92 – 3.88 (m, 1H), 3.76 – 3.74 (m, 1H), 3.45 (s, 3H), 3.28 (s, 3H).

¹³C NMR (126 MHz, CDCl₃): δ = 137.26, 134.55, 130.77, 129.00, 128.98, 128.51, 81.99, 75.92, 59.55, 57.66.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₃H₁₇O₄⁺: 237.1121, found: 237.1121.



(*E*)-4,5-dimethoxy-3-phenylpent-2-enoic acid (*E*-3t)

¹H NMR (CDCl₃, 500 MHz): δ = 7.43 – 7.39 (m, 2H), 7.36 – 7.31 (m, 3H), 6.94 (s, 1H), 4.23 – 4.21 (m, 1H), 3.66 – 3.63 (m, 2H), 3.45 (s, 3H), 3.45 (s, 3H).

¹³C NMR (126 MHz, CDCl₃): δ = 168.97, 145.58, 134.18, 129.63, 129.44, 128.93, 128.83, 77.24, 74.60, 59.68, 57.67.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₃H₁₇O₄⁺: 237.1121, found: 237.1121.



methyl 3-phenyl-3-(tetrahydrofuran-2-yl)acrylate (4)

¹H NMR (CDCl₃, 500 MHz): = 7.74 (s, 1H), 7.39 – 7.36 (m, 2H), 7.32 (m, 4H), 7.27 – 7.24 (m, 3H), 6.87 (s, 1H), 4.89 (m, 1H), 4.72 (m, 1H), 4.08 – 3.96 (m, 2H), 3.88 (m, 1H), 3.81 (d, J = 3.0 Hz, 4H), 3.66 (s, 4H), 2.37 – 2.26 (m, 1H), 2.24 – 2.05 (m, 4H), 2.00 – 1.90 (m, 4H), 1.87 (m, 1H). ¹³C NMR (CDCl₃, 126 MHz): = 169.13, 167.51, 141.70, 135.71, 135.02, 133.45, 131.84, 129.32, 128.68, 128.46, 128.41, 128.34, 128.13, 79.75, 75.05, 69.27, 68.89, 51.85, 31.88, 31.30, 27.37, 25.74.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₄H₁₇O₃⁺: 233,1172, found: 233,1171.



3-phenyl-3-(tetrahydrofuran-2-yl)prop-2-en-1-ol (6)

¹H NMR (CDCl₃, 500 MHz): = 7.36 – 7.32 (m, 7H), 7.27 (s, 1H), 7.24 (s, 1H), 7.21 – 7.18 (m, 3H), 6.71 (s, 2H), 6.65 (s, 1H), 4.88 – 4.80 (m, 2H), 4.60 – 4.53 (m, 1H), 4.46 (d, *J* = 12.2 Hz, 1H), 4.30 (s, 2H), 4.10 (d, *J* = 12.2 Hz, 2H), 4.00 (m, 3H), 3.86 (m, 2H), 3.73 (m, 2H), 2.14 (m, 3H), 2.09 – 1.95 (m, 6H), 1.95 – 1.84 (m, 4H).

¹³C NMR (CDCl₃, 126 MHz): = 169.13, 167.51, 141.70, 135.71, 135.40, 135.02, 133.51, 131.84,
129.32, 128.68, 128.46, 128.41, 128.34, 128.13, 79.75, 77.42, 77.16, 76.91, 75.05, 69.27, 68.89,
51.85, 31.88, 31.30, 27.37, 25.74.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₃H₁₇O₂⁺: 205.1223, found:205.1226.



3-phenyl-3-(tetrahydrofuran-2-yl)acrylamide (5)

¹H NMR (CDCl₃, 500 MHz): = 7.82 (dd, *J* = 7.2, 1.8 Hz, 2H), 7.70 (s, 5H), 7.44 – 7.41 (m, 4H), 7.37 (m, 12H), 7.35 – 7.29 (m, 11H), 7.27 (m, 14H), 6.68 (s, 1H), 6.32 (s, 5H), 6.22 (s, 1H), 5.97 (s, 1H), 4.83 (m, 5H), 4.61 (t, *J* = 7.0 Hz, 1H), 4.10 – 4.03 (m, 5H), 3.99 (m, 1H), 3.87 (m, 1H), 3.75 (m, 6H), 2.24 – 2.12 (m, 4H), 2.11 – 1.93 (m, 25H).

¹³C NMR (CDCl₃, 126 MHz): = 171.29, 169.98, 139.59, 135.23, 134.05, 131.93, 129.91, 128.95, 128.67, 128.60, 128.51, 128.43, 128.25, 127.44, 81.36, 77.41, 77.16, 76.90, 75.71, 68.76, 68.41, 31.19, 31.09, 26.04.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₃H₁₆NO₂⁺: 218.1176, found: 218.1177.



(Z)-3-phenyl-3-(tetrahydrofuran-2-yl)acrylaldehyde (Z-7)

¹H NMR (CDCl₃, 500 MHz): δ = 9.88 (s, 1H), 7.83 (s, 1H), 7.44 – 7.38 (m, 3H), 7.38 – 7.31 (m, 2H), 4.82 (t, J = 7.1 Hz, 1H), 4.05 (m, 1H), 3.90 (q, J = 7.4 Hz, 1H), 2.40 (m, 1H), 1.97 (m, 2H), 1.66 (m, 1H).

¹³C NMR (126 MHz, CDCl₃): δ = 137.26, 134.55, 130.77, 129.00, 128.98, 128.51, 81.99, 75.92, 59.55, 57.66.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₃H₁₅O₂⁺: 203.1067, found: 203.1066.



(E)-3-phenyl-3-(tetrahydrofuran-2-yl)acrylaldehyde (E-7)

¹H NMR (500 MHz, CDCl₃): δ = 9.64 (s, 1H), 7.52 – 7.38 (m, 6H), 4.93 (t, *J* = 7.8 Hz, 1H), 4.08 (m, 1H), 3.85 (m, 1H), 2.28 – 2.14 (m, 2H), 2.14 – 2.06 (m, 1H), 1.97 (m, 1H).

¹³C NMR (126 MHz, CDCl₃): δ = 192.51, 143.81, 142.55, 134.09, 130.15, 129.27, 128.58, 76.38, 68.64, 32.86, 25.89.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₃H₁₅O₂⁺: 203.1067, found: 203.1066.



(4S,6aR,6bS,8aS,8bR,9S,11aR,12aS,12bS)-5',6a,8a,9-tetramethyl-

1,4,5,6,6a,6b,7,8,8a,8b,9,11,11a,12,12a,12b-hexadecahydro-3H-spiro[pentaleno[2,1-a|phenanthrene-10,2'-[1,3]dioxan]-4-yl 3-phenyl-3-(tetrahydrofuran-2-yl)acrylate (9)

¹H NMR (500 MHz, CDCl₃): $\delta = 7.71$ (d, J = 3.7 Hz, 1H), 7.37 (m, 2H), 7.33 (m, 5H), 7.29 (m, 3H), 6.68 (s, 1H), 5.41 (m, 1H), 5.34 (d, J = 6.9 Hz, 1H), 4.88 (t, J = 8.1 Hz, 1H), 4.75 (m 2H), 4.44 – 4.39 (m, 2H), 4.26 – 4.19 (m, 1H), 4.04 (m, 1H), 3.91 (m, 2H), 3.81 (m, 2H), 3.48 (m, 2H), 3.38 (m, 2H), 2.71 m, 1H), 2.50 – 2.40 (m, 2H), 2.40 – 2.28 (m, 3H), 2.25 – 2.11 (m, 5H), 2.05 – 1.89 (m, 13H), 1.87 (m, 4H), 1.78 (m, 10H), 1.70 – 1.51 (m, 19H), 1.45 (m, 8H), 1.38 – 1.25 (m, 6H), 1.24 – 1.11 (m, 9H), 0.97 (m, 8H), 0.84 – 0.76 (m, 11H).

¹³C NMR (126 MHz, CDCl₃): δ = 167.94, 166.45, 154.56, 141.30, 139.75, 139.63, 135.07, 134.81, 129.27, 128.93, 128.77, 128.53, 128.49, 128.36, 128.35, 128.12, 128.01, 122.54, 109.36, 80.89, 80.87, 79.64, 76.55, 75.05, 74.49, 74.34, 69.47, 69.20, 68.80, 66.91, 62.14, 56.50, 50.01, 41.68, 40.33, 40.31, 39.80, 38.26, 37.10, 36.85, 33.20, 32.77, 32.13, 31.92, 31.84, 31.48, 31.46, 31.25, 30.37, 29.15, 28.88, 27.94, 27.38, 26.73, 26.29, 26.16, 25.71, 25.49, 25.46, 24.73, 24.47, 20.90, 19.49, 17.24, 16.39, 14.63. HRMS (ESI): m/z [M+H]⁺ calcd for C₄₀H₅₅O₅⁺: 615.4044, found: 615.4047.



2,2,6,6-tetramethyl-1-((tetrahydrofuran-2-yl)oxy)piperidine (10)

¹H NMR (CDCl₃, 500 MHz): = 5.45 – 5.24 (m, 1H), 3.98 – 3.70 (m, 2H), 2.09 – 1.86 (m, 3H), 1.86 – 1.72 (m, 1H), 1.72 – 1.38 (m, 5H), 1.31 (d, J = 11.1 Hz, 1H), 1.22 (s, 3H), 1.16 – 0.97 (m, 9H). ¹³C NMR (CDCl₃, 126 MHz): = 109.76, 66.78, 60.27, 58.77, 40.25, 39.84, 34.04, 33.49, 31.37, 24.04, 20.60, 20.21, 17.40.

HRMS (ESI): m/z [M+H]⁺ calcd for C₁₃H₂₆NO₂⁺: 228.1958, found: 228.1959.

Reference

1. Pan, S.; Li, H.; Huang, Y.; Xu, X. H.; Qing, F. L. Org Lett, 2017, 19, 3247

6. ¹H NMR and ¹³C NMR Spectra of Compounds

¹H NMR spectrum of the product of 3a (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3a (CDCl₃, 126 MHz)





S10

¹H NMR spectrum of the product of 3b (CDCl₃, 500 MHz)

7.91 7.91 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.72



¹³C NMR spectrum of the product of 3b (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3c (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3c (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of Z-3d (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of Z-3d (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of *E*-3d (CDCl₃, 500 MHz)



¹H NMR spectrum of the product of 3e (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3e (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3f (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3f (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of Z-3g (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of Z-3g (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of *E*-3g (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of *E*-3g (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3h (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3h (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3i (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3i (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3j (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3j (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of Z-3k (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of Z-3k (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of *E*-3k (CDCl₃, 500 MHz)



¹H NMR spectrum of the product of 3l (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3l (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3m (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3m (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3n (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3n (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 30 (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 30 (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3p (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3p (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3q (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3q (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3r (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3r (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 3s (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 3s (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of Z-3t (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of Z-3t (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of *E*-3t (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of *E*-3t (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 4 (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 4 (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 5 (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 5 (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 6 (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 6 (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of Z-7 (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of Z-7 (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of *E*-7 (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of *E*-7 (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 9 (CDCl₃, 500 MHz)



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¹³C NMR spectrum of the product of 9 (CDCl₃, 126 MHz)



¹H NMR spectrum of the product of 10 (CDCl₃, 500 MHz)



¹³C NMR spectrum of the product of 10 (CDCl₃, 126 MHz)

