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Supporting Information

N-Iodosuccinimide and dioxygen in air enabled synthesis of 10-phenanthrenols under sunlight

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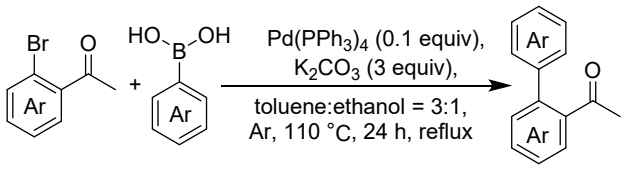
1. General information

All reagents were purchased from commercial suppliers and used without further purification. Flash chromatography was carried out with silica gel (200-300 mesh). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR (400 MHz, 101 MHz and 377 MHz, respectively) spectra were measured in $CDCl_3$, DMSO-d_6, Ethanol-d_6 recorded on Bruker Avance DPX 400 MHz spectrometer. All chemical shifts (δ) were reported in ppm and coupling constants (*J*) in Hz. NMR Spectra recorded in $CDCl_3$ were referenced to tetramethylsilane at 0 ppm for ^{1}H or referenced to residual $CHCl_3$ at 77.16 ppm for ^{13}C. NMR Spectra recorded in DMSO-d_6 were referenced to residual DMSO at 2.50 ppm for ^{1}H or 39.52 ppm for ^{13}C. NMR Spectra recorded in Ethanol-d_6 were referenced to residual ethanol at 3.56 ppm for ^{1}H. The following abbreviations are used: m (multiplet), s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), *etc*. The high resolution mass spectra (HRMS) were measured on a Bruker Daltonics APEX II 47e spectrometer by ESI. Substrates **1a**–**1z**,[1] **1aa**,[2] **1ab**,[3] **1ac**,[4] **1ad**,[1] **1ae**,[1] **1af**,[4] **1ag**–**1aj**[5] and compound **[I]-1a**[6] were prepared by the known methods.

2. Substrates preparation

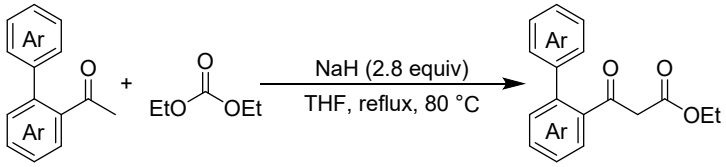
2.1 Synthesis of 1a–1z

Scheme S1



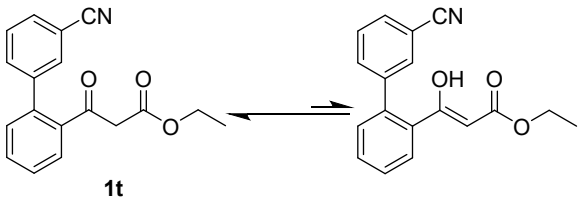
To a stirred solution of aryl bromide (4.0 mmol, 1.0 equiv) and aryl boronic acid (4.8 mmol, 1.2 equiv) in a toluene:ethanol = 3:1 (40 mL) mixture was added potassium carbonate (12.0 mmol, 3.0 equiv) and tetrakis(triphenylphosphine) palladium (0.4 mmol, 0.1 equiv). The resulting suspension was heated at 110 °C under an atmosphere of Ar for 24 h. The solvent was removed under reduced pressure and the crude residue was redissolved in water (100 mL) and extracted with EtOAc (3 × 30 mL). The combined organic layers were washed with water and brine. The filtrate was concentrated under reduced pressure and purified by column chromatography.

Scheme S2



To a dried three-necked flask equipped with a dropping funnel, a condenser, and a magnetic stirrer was added NaH (60% in mineral oil, 11.2 mmol, 2.8 equiv), diethyl carbonate (11.2 mmol, 2.8 equiv), and dry THF (10 mL). The mixture was heated to 80 °C under an atmosphere of Ar. A solution of ketone (4.0 mmol, 1.0 equiv) in dry THF (50 mL) was added dropwise from the dropping funnel over 30–60 min. After the addition, the mixture was heated to reflux until the biphenyl ketone is completely consumed (12 h). When the reaction was cooled to room temperature, glacial acetic acid was added dropwise and a heavy, pasty solid appeared. Ice-water was added until the solid was dissolved completely. The THF layer was separated, and the water layer was extracted with EtOAc (3 × 30 mL). The combined organic solution was washed with water and brine. After evaporation of the solvent, the mixture was distilled under reduced pressure. The crude residue was purified by column chromatography to furnish the desired compound **1a**–**1z**.

Ethyl 3-(3'-cyano-[1,1'-biphenyl]-2-yl)-3-oxopropanoate

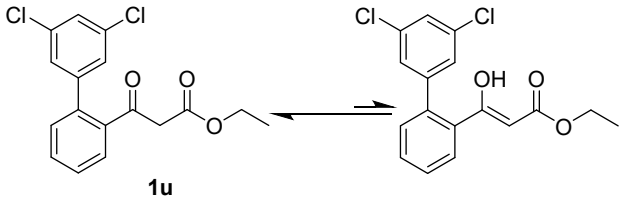


1t

keto:*enol* = 77:23

Pale yellow liquid (1.0 g, 83%, *keto*:*enol* = 77:23); $\mathbf{R_f}$ = 0.38 (petroleum ether/ethyl acetate 5:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.24 (s, 0.3H, *enol*), 7.70–7.43 (m, 9.1H, *keto* + *enol*), 7.36–7.31 (m, 1.3H, *keto* + *enol*), 5.13 (s, 0.3H, *enol*), 4.20 (q, *J* = 6.8 Hz, 0.6H, *enol*), 4.13 (q, *J* = 6.8 Hz, 2H, *keto*), 3.58 (s, 2H, *keto*), 1.28 (t, *J* = 7.2 Hz, 0.9H, *enol*), 1.21 (t, *J* = 7.2 Hz, 3H, *keto*); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.6, 173.3, 172.4, 166.9, 142.4, 141.9, 139.1, 138.6, 138.1, 133.8, 133.4, 133.2, 132.2, 132.0, 131.9, 131.3, 131.1, 130.9, 130.7, 130.6, 129.4, 129.4, 129.1, 128.9, 128.5, 128.3, 118.8, 118.6, 112.9, 112.5, 93.0, 61.5, 60.5, 48.5, 14.3, 14.1; **ESI-HRMS**: m/z Calcd for $C_{18}H_{16}NO_3^+$ [M+H]$^+$: 294.1125, found 294.1115.

Ethyl 3-(3',5'-dichloro-[1,1'-biphenyl]-2-yl)-3-oxopropanoate

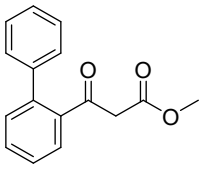


1u

keto:*enol* = 71:29

White solid (1.2 g, 90%, *keto*:*enol* = 71:29); $\mathbf{R_f}$ = 0.47 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.26 (s, 0.4H, *enol*), 7.65 (d, *J* = 7.6 Hz, 1H, *keto*), 7.58–7.54 (m, 1.4H, *keto* + *enol*), 7.50–7.29 (m, 4.6H, *keto* + *enol*), 7.25 (d, *J* = 8.0 Hz, 2.8H, *keto* + *enol*), 5.14 (s, 0.4H, *enol*), 4.21 (q, *J* = 7.2 Hz, 0.8H, *enol*), 4.13 (q, *J* = 6.8 Hz, 2H, *keto*), 3.55 (s, 2H, *keto*), 1.29 (t, *J* = 7.2 Hz, 1.2H, *enol*), 1.21 (t, *J* = 6.8 Hz, 3H, *keto*); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.9, 173.2, 172.5, 166.9, 144.1, 143.4, 138.5, 138.4, 138.3, 135.3, 134.7, 133.8, 131.8, 130.8, 130.7, 130.5, 129.3, 128.9, 128.6, 128.4, 128.0, 127.4, 127.2, 93.0, 61.6, 60.5, 48.7, 14.3, 14.1; **ESI-HRMS**: m/z Calcd for $C_{17}H_{15}Cl_2O_3^+$ [M+H]$^+$: 337.0393, found 337.0381.

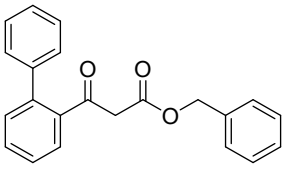
2.2 Synthesis of methyl 3-([1,1'-biphenyl]-2-yl)-3-oxopropanoate (1aa)



1aa

To a dried three-necked flask equipped with a dropping funnel, a condenser, and a magnetic stirrer was added NaH (60% in mineral oil, 11.2 mmol, 2.8 equiv), dimethyl carbonate (11.2 mmol, 2.8 equiv), and dry THF (10 mL). The mixture was heated to 80 °C under an atmosphere of Ar. A solution of 1-([1,1'-biphenyl]-2-yl)ethan-1-one (4.0 mmol, 1.0 equiv) in dry THF (50 mL) was added dropwise from the dropping funnel over 30–60 min. After the addition, the mixture was heated to reflux until the 1-([1,1'-biphenyl]-2-yl)ethan-1-one is completely consumed (12 h). When the reaction was cooled to room temperature, glacial acetic acid was added dropwise and a heavy, pasty solid appeared. Ice-water was added until the solid was dissolved completely. The THF layer was separated, and the water layer was extracted with EtOAc (3 × 30 mL). The combined organic solution was washed with water and brine. After evaporation of the solvent, the mixture was distilled under reduced pressure. The crude residue was purified by column chromatography to furnish the compound **1aa** as a white solid.

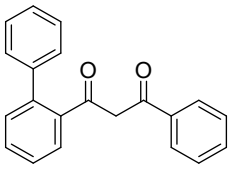
2.3 Synthesis of benzyl 3-([1,1'-biphenyl]-2-yl)-3-oxopropanoate (1ab)



1ab

A mixture of benzyl alcohol (4.0 mmol, 1.0 equiv), ethyl 3-([1,1'-biphenyl]-2-yl)-3-oxopropanoate (4.0 mmol, 1.0 equiv), DMAP (4.0 mmol, 1.0 equiv) was stirred with oven-dried 4 Å molecular sieves (20 g) in dry toluene (30 mL) at 100–105 °C for 48 h. The reaction mixture was cooled to room temperature, and filtered to remove the molecular sieves. The solvents were removed under reduced pressure, and EtOAc (60 mL) and water (60 mL) were added to the residue. The layers were separated, and filtered, and concentrated. The crude product was purified by column chromatography to furnish the compound **1ab** as a pale yellow liquid.

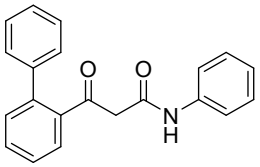
2.4 Synthesis of 1-([1,1'-biphenyl]-2-yl)-3-phenylpropane-1,3-dione (1ac)



1ac

To a stirred solution of 1-([1,1'-biphenyl]-2-yl)ethan-1-one (4.0 mmol, 1.0 equiv) in toluene (30 mL) pre-cooled at 0 °C, LiHMDS (1 M in toluene, 6.0 mmol, 1.5 equiv) was dropwise added over 10 min. After stirring at 0 °C for 10 min, benzoyl chloride (99%, 8.0 mmol, 2.0 equiv) was added in one portion. The reaction mixture was then allowed to stir at room temperature for an additional 2 min, quenched by glacial acetic acid and diluted with ethyl acetate. The solution was washed with water and brine, and concentrated under reduced pressure. The crude residue was purified by column chromatography to furnish the compound **1ac** as a pale yellow solid.

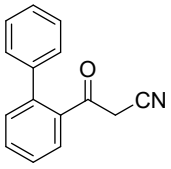
2.5 Synthesis of 3-([1,1'-biphenyl]-2-yl)-3-oxo-*N*-phenylpropanamide (1ad)



1ad

To ethyl 3-([1,1'-biphenyl]-2-yl)-3-oxopropanoate (4.0mmol, 1.0 equiv) in a round bottom flask, aniline (4.0mmol, 1.0 equiv) was added. To this reaction mixture, xylene (10 mL) was added, and the whole of the reaction mixture was heated at 165 °C under an atmosphere of Ar on an oil bath for 24 h. The reaction mixture was removed from heating, allowed to attain ambient temperature, distilled under reduced pressure. And the resultant residue was purified by column chromatography to furnish compound **1ad** as a pale yellow liquid.

2.6 Synthesis of 3-([1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (1ae)

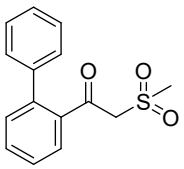


1ae

To a suspension of NaH (2.0 equiv.) in dry THF, under Ar was added acetonitrile (2 mL), followed by methyl [1,1'-biphenyl]-2-carboxylate (1.0 equiv). The reaction mixture was heated to 80 °C for 24 h. Then

cooled to 0°C, quenched with water. The THF layer was separated, and the water layer was extracted with EtOAc. The combined organic solution was washed with water and brine. After evaporation of the solvent, the mixture was distilled under reduced pressureor subjected chromatography to give **1ae** as a yellow solid.

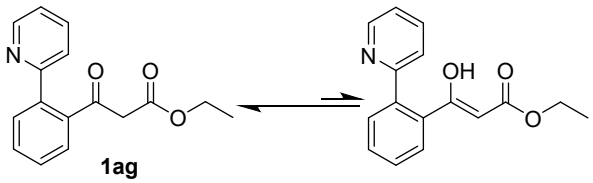
2.7 Synthesis of 1-([1,1'-biphenyl]-2-yl)-2-(methylsulfonyl)ethan-1-one (1af)



1af

Under Ar protection, *n*-BuLi (2.76 mL, 2.0 M in cyclohexane, 5.52 mmol) was dropwise added to a stirred solution of dimethyl sulfone (314.8 mg, 99%, 3.31 mmol) in THF (16 mL) at 0 °C. The resultant white cloudy solution was continued to stir at 0 °C for 40 min, followed by slowly adding with a solution of ethyl biphenyl-2-carboxylate (624.3 mg, 2.76 mmol) in THF (11.5 mL) over 5 min. The reaction mixture was then allowed to stir at rt for 36 hours, quenched by H_2O (15 mL), and diluted with ethyl acetate (350 mL). The organic layer was separated and washed with saturated aqueous NH_4Cl solution (50 mL x 2), water (50 mL) and brine (50 mL). After concentration, the crude mixture was subjected to chromatography to provide **1af** as a white solid.

2.8 Synthesis of ethyl 3-oxo-3-(2-(pyridin-2-yl)phenyl)propanoate (1ag)



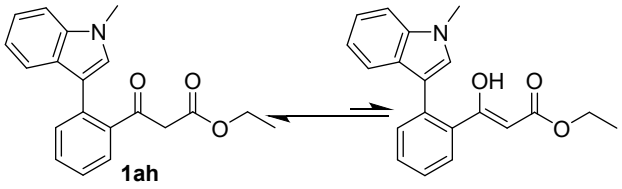
1ag

keto:*enol* = 91:9

In an argon-filled glove box, to a 100 mL Schlenk tube was charged sequentially $Pd(OAc)_2$ (0.08 mmol), P*t*Bu_3•HBF_4 (0.12 mmol), 2-bromopyridine (4 mmol), (2-acetylphenyl)boronic acid (4.8 mmol), and 35 mL of dioxane/H_2O (4:1). The mixture was stirred at RT for 15 min, and then a solution of NaOH (6.5 mmol) in 6 mL of degassed H_2O was added to initiate the Suzuki reaction. The Schlenk tube was capped tightly and the reaction mixture was stirred vigorously at 80 °C for 12 h. At the end of the reaction, the organic phase was separated and the aqueous phase was further extracted with Et_2O. The combined organic extracts were concentrated on a rotary evaporator. The resulting residue was purified by silica gel flash chromatography to obtain the desired coupling product 1-(2-(pyridin-2-yl)phenyl)ethan-1-one. The next operation is the same as

Scheme S2 to provide **1ag**. Yellow liquid (0.92 g, 85%, *keto*:*enol* = 91:9); $\mathbf{R_f}$ = 0.39 (petroleum ether/ethyl acetate 1:2); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.22 (s, 0.1H, *enol*), 8.63 (s, 1.1H, *keto* + *enol*), 7.77 (t, *J* = 7.6 Hz, 1.1H, *keto* + *enol*), 7.69 (d, *J* = 7.2 Hz, 2.2H, *keto* + *enol*), 7.60–7.45 (m, 3.3H, *keto* + *enol*), 7.28–7.25 (m, 1.1H, *keto* + *enol*), 5.21 (s, 0.1H, *enol*), 4.18 (q, *J* = 6.4 Hz, 0.2H, *enol*), 4.11 (q, *J* = 7.2 Hz, 2H, *keto*), 3.56 (s, 2H, *keto*), 1.27 (t, *J* = 6.8 Hz, 0.3H, *enol*), 1.20 (t, *J* = 6.8 Hz, 3H, *keto*); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.3, 174.3, 172.6, 167.4, 158.6, 156.1, 149.4, 149.1, 140.6, 139.9, 137.9, 137.1, 136.1, 133.9, 130.6, 130.5, 130.3, 129.1, 128.9, 128.4, 128.3, 128.0, 123.6, 122.6, 122.1, 121.9, 92.1, 61.1, 60.2, 49.4, 14.2, 14.1; **ESI-HRMS**: m/z Calcd for $C_{16}H_{16}NO_3^+$ $[M+H]^+$: 270.1125, found 270.1127.

2.9 Synthesis of ethyl 3-(2-(1-methyl-1*H*-indol-3-yl)phenyl)-3-oxopropanoate (1ah)



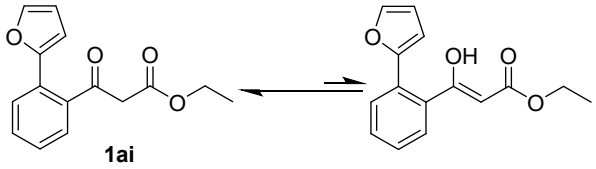
1ah

keto:*enol* = 95:5

In an argon-filled glove box, to a 100 mL Schlenk tube was charged sequentially $Pd(OAc)_2$ (0.08 mmol), P*t*Bu_3•HBF_4 (0.12 mmol), 3-bromo-1-methyl-1*H*-indole (4 mmol), (2-acetylphenyl)boronic acid (4.8 mmol), and 35 mL of dioxane. The mixture was prestirred at RT for 15 min, and then a solution of NaOH (6.5 mmol) in 6 mL of degassed H_2O was added to initiate the Suzuki reaction. The Schlenk tube was capped tightly and the reaction mixture was stirred vigorously at 80 °C for 12 h. At the end of the reaction, the organic phase was separated and the aqueous phase was further extracted with Et_2O. The combined organic extracts were concentrated on a rotary evaporator. The resulting residue was purified by silica gel flash chromatography to obtain the desired coupling product 1-(2-(1-methyl-1H-indol-3-yl)phenyl)ethan-1-one. The next operation is the same as Scheme S2 to provide **1ah**. Yellow liquid (770 mg, 60%, *keto*:*enol* = 95:5); $\mathbf{R_f}$ = 0.27 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.35 (s, 0.05H, *enol*), 7.70 (d, *J* = 8.0 Hz, 1.05H, *keto* + *enol*) 7.64–7.52 (m, 3.15H, *keto* + *enol*), 7.40–7.29 (m, 3.15H, *keto* + *enol*), 7.24–7.14 (m, 1.05H, *keto* + *enol*), 7.04 (s, 1.05H, *keto* + *enol*), 5.14 (s, 0.05H, *enol*), 4.13 (q, *J* = 7.2 Hz, 0.1H, *enol*), 4.01 (q, *J* = 6.8 Hz, 2H, *keto*), 3.83 (s, 3H, *keto*), 3.81 (s, 0.15H, *enol*), 3.32 (s, 2H, *keto*), 1.21 (t, *J* = 7.2 Hz, 0.15H, *enol*), 1.13 (t, *J* = 6.8 Hz, 3H, *keto*); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.3, 175.0, 172.9, 167.4, 140.1, 137.3, 137.0, 134.2,

133.9, 133.2, 131.5, 131.2, 130.6, 130.1, 129.3, 128.6, 127.8, 127.1, 126.6, 126.5, 126.3, 122.7, 121.9, 120.5, 119.8, 119.7, 115.0, 114.0, 109.8, 109.4, 92.2, 61.1, 60.1, 48.4, 33.1, 14.3, 14.1; **ESI-HRMS**: m/z Calcd for $C_{20}H_{20}NO_3^+$ $[M+H]^+$: 332.1438, found 332.1431.

2.10 Synthesis of ethyl 3-(2-(furan-2-yl)phenyl)-3-oxopropanoate (1ai)

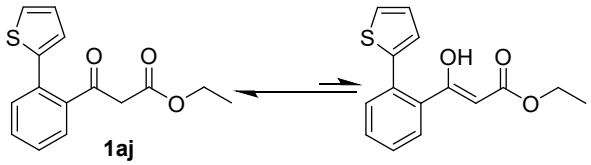


1ai

keto:*enol* = 87:13

In an argon-filled glove box, to a 100 mL Schlenk tube was charged sequentially $Pd(OAc)_2$ (0.08 mmol), P*t*Bu_3•HBF_4 (0.12 mmol), 2-bromofuran (4 mmol), (2-acetylphenyl)boronic acid (4.8 mmol), and 35 mL of *n*BuOH/H_2O (4:1). The mixture was prestirred at RT for 15 min, and then a solution of NaOH (6.5 mmol) in 6 mL of degassed H_2O was added to initiate the Suzuki reaction. The Schlenk tube was capped tightly and the reaction mixture was stirred vigorously at r.t. for 12 h. At the end of the reaction, the organic phase was separated and the aqueous phase was further extracted with Et_2O. The combined organic extracts were concentrated on a rotary evaporator. The resulting residue was purified by silica gel flash chromatography to obtain the desired coupling product 1-(2-(furan-2-yl)phenyl)ethan-1-one. The next operation is the same as Scheme S2 to provide **1ai**. Yellow liquid (720 mg, 70%, *keto*:*enol* = 87:13); $\mathbf{R_f}$ = 0.41 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.36 (s, 0.15H, *enol*), 7.67 (d, *J* = 7.6 Hz, 0.15H, *enol*), 7.60 (d, *J* = 7.6 Hz, 1H, *keto*), 7.53–7.46 (m, 3.3H, *keto* + *enol*), 7.40–7.26 (m, 1.3H, *keto* + *enol*), 6.63–6.60 (m, 1.15H, *keto* + *enol*), 6.52–6.46 (m, 1.15H, *keto* + *enol*), 5.23 (s, 0.15H, *enol*), 4.26 (q, *J* = 7.2 Hz, 0.3H, *enol*), 4.13 (q, *J* = 7.2 Hz, 2H, *keto*), 3.55 (s, 2H, *keto*), 1.32 (t, *J* = 7.2 Hz, 0.45H, *enol*), 1.22 (t, *J* = 7.2 Hz, 3H, *keto*); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.9, 174.8, 172.9, 167.2, 152.1, 151.8, 143.6, 142.6, 138.0, 132.4, 130.9, 130.2, 129.5, 129.5, 128.4, 128.1, 128.1, 128.0, 127.7, 127.5, 112.2, 111.7, 109.0, 108.7, 92.0, 61.4, 60.4, 48.7, 14.4, 14.1; **ESI-HRMS**: m/z Calcd for $C_{15}H_{15}O_4^+$ $[M+H]^+$: 259.0965, found 259.0968.

2.11 Synthesis of ethyl 3-oxo-3-(2-(thiophen-2-yl)phenyl)propanoate (1aj)

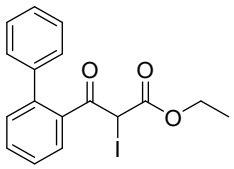


1aj

keto:*enol* = 83:17

In an argon-filled glove box, to a 100 mL Schlenk tube was charged sequentially $Pd(OAc)_2$ (0.08 mmol), P*t*Bu_3•HBF_4 (0.12 mmol), 2-bromothiophene (4 mmol), (2-acetylphenyl)boronic acid (4.8 mmol), and 35 mL of *n*BuOH/H_2O (4:1). The mixture was prestirred at RT for 15 min, and then a solution of NaOH (6.5 mmol) in 6 mL of degassed H_2O was added to initiate the Suzuki reaction. The Schlenk tube was capped tightly and the reaction mixture was stirred vigorously at r.t. for 12 h. At the end of the reaction, the organic phase was separated and the aqueous phase was further extracted with Et_2O. The combined organic extracts were concentrated on a rotary evaporator. The resulting residue was purified by silica gel flash chromatography to obtain the desired coupling product 1-(2-(thiophen-2-yl)phenyl)ethan-1-one. The next operation is the same as Scheme S2 to provide **1aj**. Yellow liquid (820 mg, 75%, *keto*:*enol* = 83:17); $\mathbf{R_f}$ = 0.41 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.31 (s, 0.2H, *enol*), 7.54–7.49 (m, 3.6H, *keto* + *enol*), 7.44–7.32 (m, 2.4H, *keto* + *enol*), 7.12–7.09 (m, 1.2H, *keto* + *enol*), 7.05–7.02 (m, 1.2H, *keto* + *enol*) , 5.20 (s, 0.2H, *enol*), 4.21 (q, *J* = 6.8 Hz, 0.4H, *enol*), 4.08 (q, *J* = 6.8 Hz, 2H, *keto*), 3.41 (s, 2H, *keto*), 1.29 (t, *J* = 6.8 Hz, 0.6H, *enol*), 1.18 (t, *J* = 7.2 Hz, 3H, *keto*); **^{13}C NMR** (101 MHz, CDCl3) δ 199.2, 174.1, 172.7, 167.1, 141.9, 140.9, 140.0, 134.2, 133.4, 132.5, 131.2, 131.1, 130.7, 130.1, 129.4, 128.5, 128.3, 128.2, 127.8, 127.5, 127.3, 126.6, 126.0, 92.9, 61.3, 60.4, 48.6, 14.3, 14.1; **ESI-HRMS**: m/z Calcd for $C_{15}H_{15}O_3S^+$ $[M+H]^+$: 275.0736, found 275.0739.

2.12 Synthesis of ethyl 3-([1,1'-biphenyl]-2-yl)-2-iodo-3-oxopropanoate ([I]-1a)

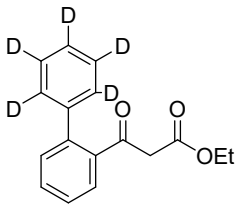


[I]-1a

To the i-PrOH solution of ethyl 3-([1,1'-biphenyl]-2-yl)-3-oxopropanoate (**1a**, 1.0 mmol), *N*-halosuccinamide (NIS) (1.05 mmol) was added, and the resultant mixture was stirred at room temperature for 3 h in dark. After completion of the reaction as indicated by TLC, the reaction mixture was washed with NH_4Cl

solution. The product was extracted with ethyl acetate, dried over sodium sulphate, and purified by column chromatography to afford [I]-**1a**. Yellow liquid; yield 335 mg, 85%; $\mathbf{R_f}$ = 0.36 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.2 Hz, 1H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.44–7.35 (m, 7H), 4.91 (s, 1H), 4.10–4.06 (m, 2H), 1.17 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 195.8, 166.2, 140.3, 139.6, 136.4, 131.7, 130.4, 130.3, 129.3, 128.8, 128.5, 127.6, 62.8, 26.5, 13.8; **ESI-HRMS**: m/z Calcd for $C_{17}H_{16}IO_3^+$ $[M+H]^+$: 395.0139, found 395.0126.

2.13 Synthesis of ethyl 3-([1,1'-biphenyl]-2-yl-2',3',4',5',6'-*d*$_5$)-3-oxopropanoate (D$_5$-1a)



D$_5$-**1a**

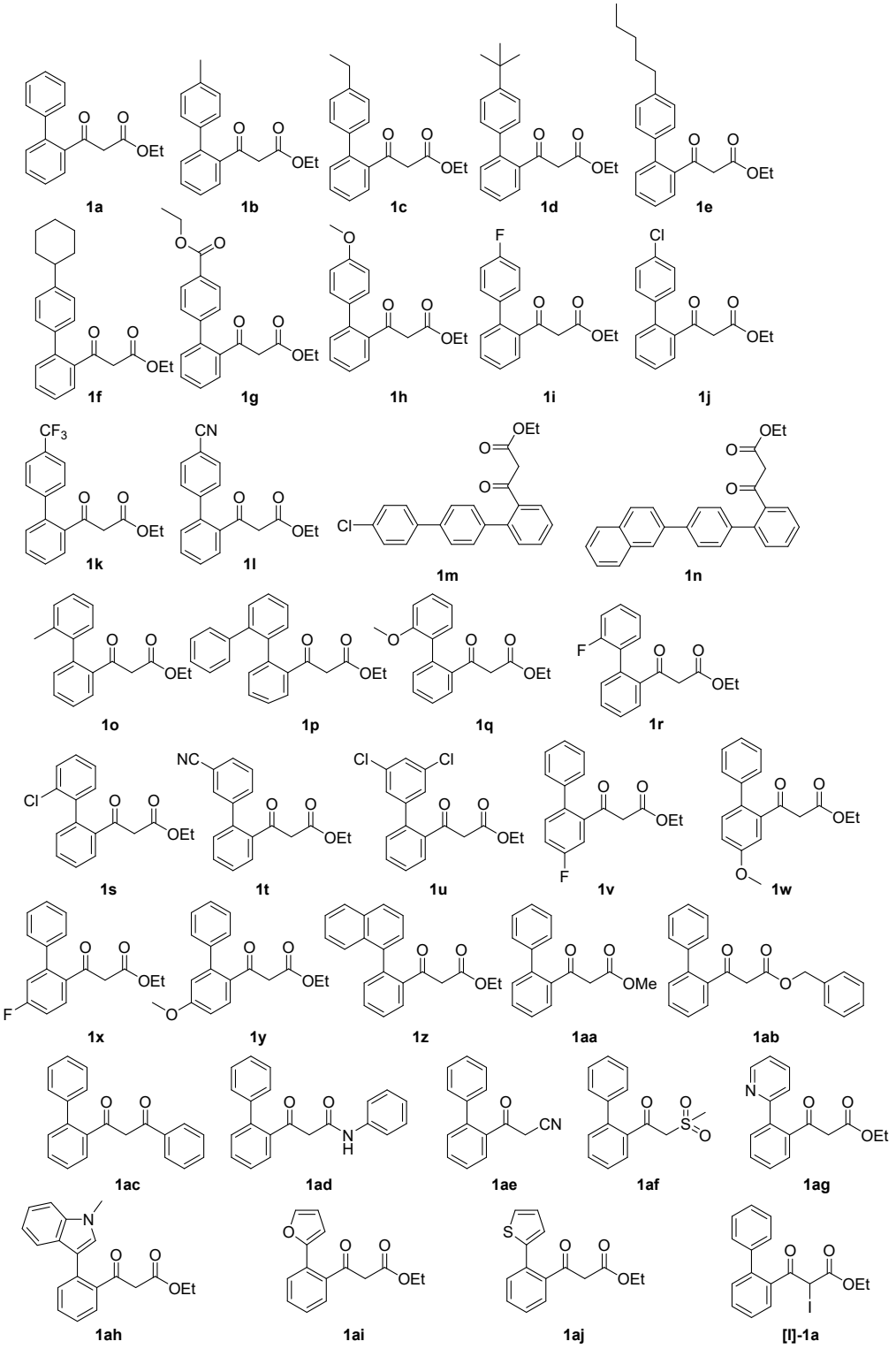
To a stirred solution of 1-(2-bromophenyl)ethan-1-one (4.0 mmol, 1.0 equiv) and (phenyl-*d*$_5$)boronic acid (4.8 mmol, 1.2 equiv) in a toluene:ethanol = 3:1 (40 mL) mixture was added potassium carbonate (12.0 mmol, 3.0 equiv) and tetrakis(triphenylphosphine) palladium (0.4 mmol, 0.1 equiv). The resulting suspension was heated at 110 °C under an atmosphere of Ar for 24 h. The solvent was removed under reduced pressure and the crude residue was redissolved in water (100 mL) and extracted with EtOAc (3 × 30 mL). The combined organic layers were washed with water and brine. The filtrate was concentrated under reduced pressure and purified by column chromatography.

To a dried three-necked flask equipped with a dropping funnel, a condenser, and a magnetic stirrer was added NaH (60% in mineral oil, 11.2 mmol, 2.8 equiv), diethyl carbonate (11.2 mmol, 2.8 equiv), and dry THF (10 mL). The mixture was heated to 80 °C under an atmosphere of Ar. A solution of 1-([1,1'-biphenyl]-2-yl-2',3',4',5',6'-*d*$_5$)ethan-1-one (4.0 mmol, 1.0 equiv) in dry THF (50 mL) was added dropwise from the dropping funnel over 30–60 min. After the addition, the mixture was heated to reflux until the 1-([1,1'-biphenyl]-2-yl-2',3',4',5',6'-*d*$_5$)ethan-1-one is completely consumed (12 h). When the reaction was cooled to room temperature, glacial acetic acid was added dropwise and a heavy, pasty solid appeared. Ice-water was added until the solid was dissolved completely. The THF layer was separated, and the water layer was extracted with EtOAc (3 × 30 mL). The combined organic solution was washed with water and brine. After evaporation of the solvent, the mixture was distilled under reduced pressure. The crude residue was purified by column

chromatography to furnish the compound D_5-**1a** 0.9 g, 82% yield. Pale yellow liquid, (*keto*:*enol* = 87:13); $\mathbf{R_f}$ = 0.47 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.26 (s, 0.15H, *enol*),7.61–7.58 (m, 1.15H, *keto* + *enol*), 7.53 (t, *J* = 7.6 Hz, 1H, *keto*), 7.48–7.36 (m, 2.45H, *keto* + *enol*), 5.06 (s, 0.15H, *enol*), 4.16 (q, *J* = 7.2 Hz, 0.3H, *enol*), 4.05 (q, *J* = 7.2 Hz, 2H, *keto*), 3.27 (s, 2H, *keto*), 1.25 (t, *J* = 7.2 Hz, 0.45H, *enol*), 1.15 (t, *J* = 6.8 Hz, 3H, *keto*); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.0, 174.2, 172.7, 167.1, 141.0, 140.8, 140.6, 140.0, 139.5, 133.8, 131.4, 130.9, 130.4, 130.2, 129.1, 128.9, 128.7, 128.6, 128.5, 128.4, 128.2, 128.0, 127.7, 127.6, 127.3, 92.8, 61.2, 60.3, 48.8, 14.3, 14.1; **ESI-HRMS**: m/z Calcd for $C_{17}H_{12}D_5O_3^+$ $[M+H]^+$: 274.1486, found 274.1476.

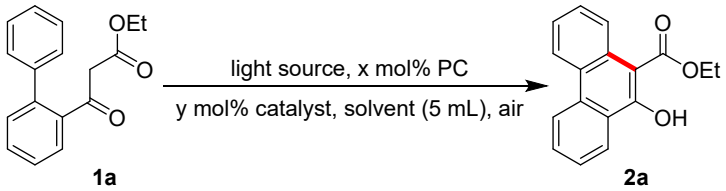
2.14 Involved Substrates

Table S1. Involved Substrates



3. Reaction optimization

Table S2. Optimization of the Reaction Conditions[a]



1a **2a**

Entry	PC (x mol%)	Catalyst (y mol%)	Solvent (mL)	light source (nm)	Time (h)	Yield (%) [b]
1	–	NIS (100)	i-PrOH	sunlight	7	53
2	–	NIS (50)	i-PrOH	sunlight	7	57
3	–	NIS (20)	i-PrOH	sunlight	7	67
4	–	NIS (10)	i-PrOH	sunlight	7	79
5	**–**	**NIS (5)**	**i-PrOH**	**sunlight**	**7**	**87**
6	–	NIS (1)	i-PrOH	sunlight	7	trace
7	–	I_2	i-PrOH	sunlight	7	80
8	–	HI	i-PrOH	sunlight	7	65
9	–	TBAI	i-PrOH	sunlight	7	12
10	–	KI	i-PrOH	sunlight	7	20
11	–	NIS (5)	DMSO	sunlight	24	79
12	–	NIS (5)	CH_3CN	sunlight	24	18
13	–	NIS (5)	MeOH	sunlight	24	trace
14	–	NIS (5)	Toluene	sunlight	24	18
15	–	NIS (5)	DCM	sunlight	24	15
16	–	NIS (5)	THF	sunlight	16	82
17	–	NIS (5)	EA	sunlight	16	65
18	–	NIS (5)	Acetone	sunlight	24	60
19	–	NIS (5)	EtOH	sunlight	16	77
20	–	NIS (5)	i-PrOH	3W blue LEDs (445)	7	90
21	–	NIS (5)	i-PrOH	3W green LEDs (525)	24	NR
22	**–**	**NIS (10)**	**THF**	**sunlight**	**12**	**93**
23	–	NIS (5)	i-PrOH	dark	7	NR
24[c]	–	NIS (5)	i-PrOH	dark	7	NR
25	–	–	i-PrOH	sunlight	7	NR
26	–	NIS (5)	i-PrOH	sunlight	7	NR
27	–	NBS (5)	i-PrOH	sunlight	7	15
28	–	NCS (5)	i-PrOH	sunlight	7	10
29	EY (3)	–	CH_3CN	3W green LEDs (525)	8	trace
30	EY (3)	–	DMF	3W green LEDs (525)	8	trace
31	EY (3)	–	DMF	3W green LEDs (525)	24	trace
32	EY (3)	–	DMF	3W green LEDs (525)	8	36
33	EY (3)	–	DMSO	3W green LEDs (525)	8	trace
34	$Ir(ppy)_3$ (3)	–	CH_3CN	3W blue LEDs (465)	24	NR

35	$Ir(ppy)_3$ (3)	–	DMF	3W blue LEDs (465)	24	NR
36	$Ir(ppy)_3$ (3)	–	DMSO	3W blue LEDs (465)	24	trace
37	$Ru(bpy)_3$ (3)	–	CH_3CN	3W blue LEDs (465)	24	trace
38	$Ru(bpy)_3$ (3)	–	DMF	3W blue LEDs (465)	24	trace
39	$Ru(bpy)_3$ (3)	–	DMSO	3W blue LEDs (465)	24	NR

[a]Reaction conditions: corresponding **1a**, photocatalyst (x mol%), cocatalyst (y mol%) in 5 mL solvent in air atmosphere, irradiation with light source, at rt. [b]Isolated yields. [c]50 °C.

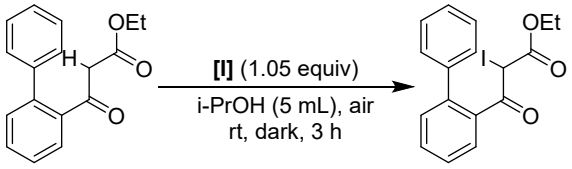
4. General experimental procedure

Mathod A: A 10 mL Pyrex tube was charged with substrate **1a**–**1n**, **1t**–**1ac** (0.1 mmol, 1 equiv) and NIS (0.005 mmol, 1.12 mg, 0.05 equiv) in i-PrOH (5 mL). The sample was then irradiated by sunlight for 7 h. Upon completion of the reaction, the solvent was then removed under vacuum. The residue was purified with chromatography column on silica gel using mixtures of petroleum ether and ethyl acetate to give the corresponding products. The identity and purity of the product **2a**–**2n**, **2t**–**2ac** was confirmed by ^{1}H NMR, ^{13}C NMR or ^{19}F NMR spectroscopic analysis. **Mathod B**: A 10 mL Pyrex tube was charged with substrate **1o**–**1s** (0.1 mmol, 1 equiv) and NIS (0.01 mmol, 2.24 mg, 0.1 equiv) in THF (5 mL). The sample was then irradiated by sunlight for 12 h. Upon completion of the reaction, the solvent was then removed under vacuum. The residue was purified with chromatography column on silica gel using mixtures of petroleum ether and ethyl acetate to give the corresponding products. The identity and purity of the product **2o**–**2s** was confirmed by ^{1}H NMR, ^{13}C NMR or ^{19}F NMR spectroscopic analysis.

The irradiation was maintained from 9:00 am till 16:00 pm (Longitude: 116.338229, Latitude: 39.994285). The ambient temperature and the light intensity are reported later. Other procedures are the same as the general method described above.

5. Mechanism study

5.1 Synthesis of [I]-1a



1a
0.1 mmol

[I]-1a

[I]	Yield
NIS	85%
I_2	NR
I_2 with 4 equiv H_2O	NR
HI	NR
TBAI	NR
KI	NR

Fig. S1 Synthesis of [I]-**1a**.

5.2 UV−vis absorption spectra

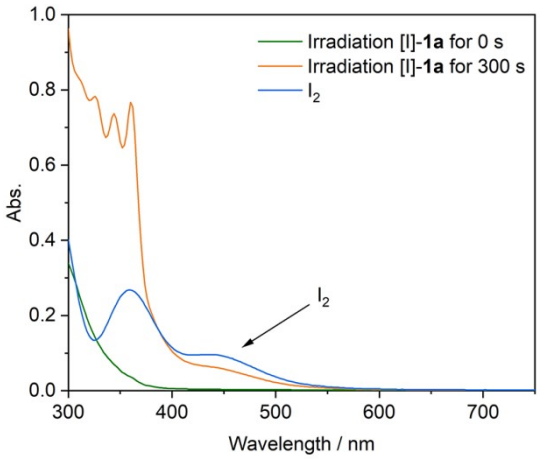


Fig. S2 UV−vis absorption spectra: i-PrOH solution of [I]-**1a** (0.1 mM) (labeled by green line); i-PrOH solution of [I]-**1a** (0.1 mM) irradiation with sunlight for 300 s (labeled by yellow line); i-PrOH solution of I_2 (0.1 mM) (labeled by blue line).

5.3 Light off/on experiment

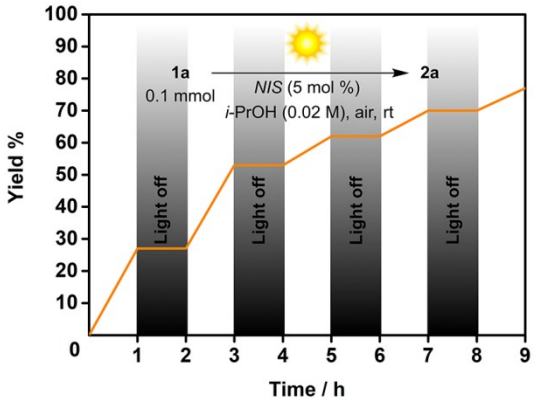
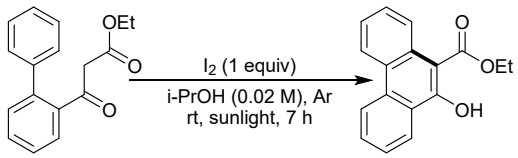


Fig. S3 Light off/on experiment.

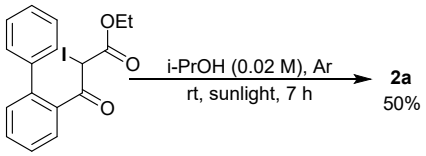
5.4 Control experiments

a) Elucidation of role of I ˙



1a 0.1 mmol — **2a** 23%

b) Elucidation of role of [I]-1a and O_2



2a 50%

[I]-1a 0.1 mmol

[I]-1a 0.1 mmol → (i-PrOH (0.02 M), air; rt, sunlight, 7 h) → **2a** 64%

c) Elucidation of role of $^1O_2$

1a 0.1 mmol → (NIS (5 mol %); DABCO (0.5 equiv); i-PrOH (0.02 M), air; rt, sunlight, 7 h) → **2a** 31%

d) Elucidation of role of H_2O_2

1a 0.1 mmol → (I_2 (1 equiv); i-PrOH (0.02 M), air; rt, dark, 3 h) → **[I]-1a** NR

1a 0.1 mmol → (I_2 (1 equiv); 30% H_2O_2 aqua (4 equiv); i-PrOH (0.02 M), air; rt, dark, 3 h) → **[I]-1a** 77%

Fig. S4 Control experiments.

5.5 ^{1}H spectra determination of H_2O_2

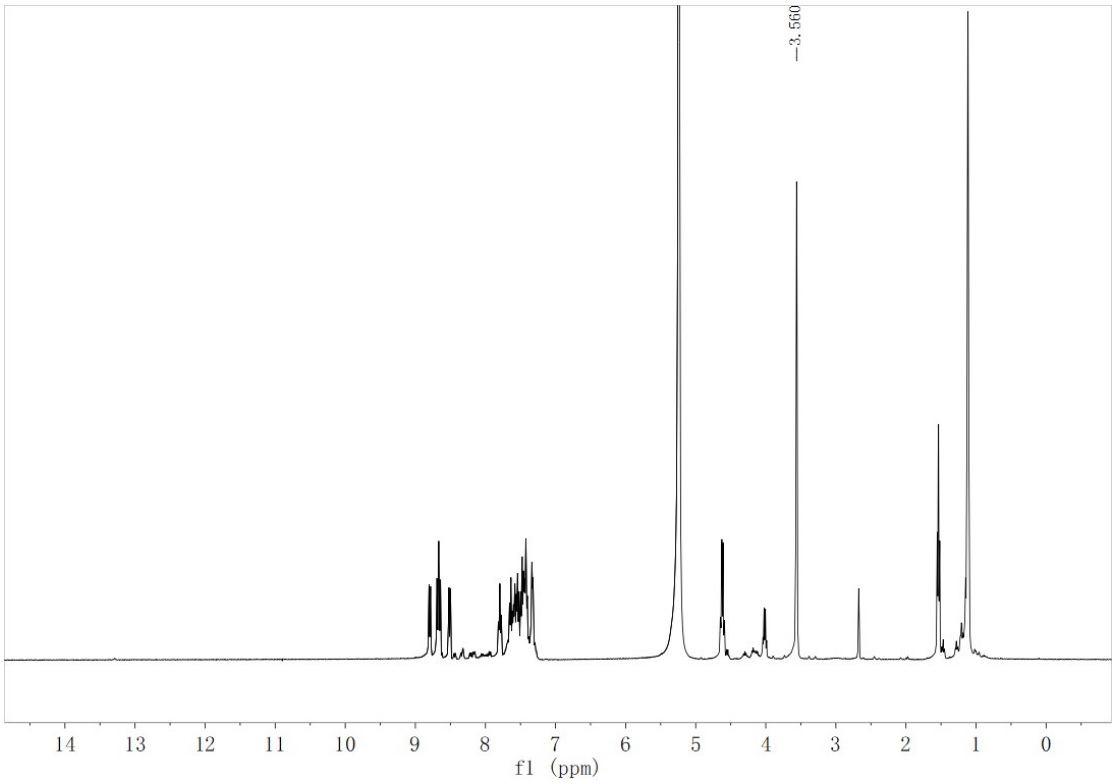


Fig. S5 ^{1}H spectra of the reaction mixture irradiation with sunlight for 5 h (Ethanol-d_6, 400 MHz). No H_2O_2 was detected.

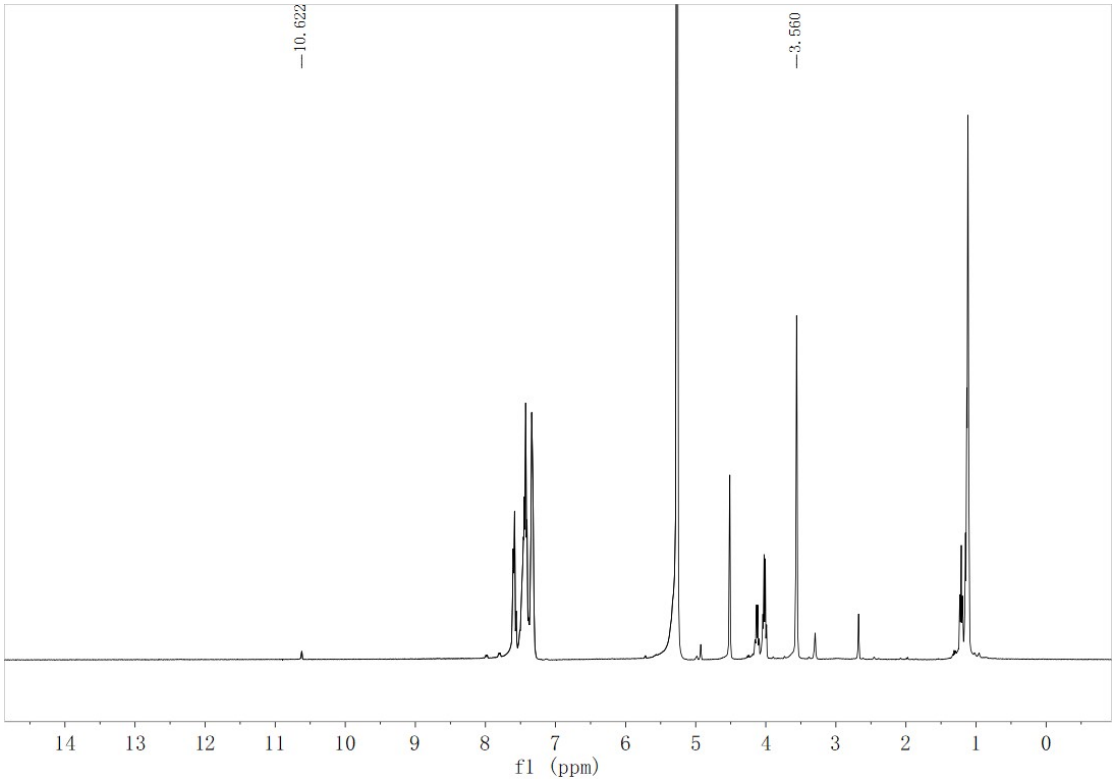


Fig. S6 ^{1}H spectra of the reaction mixture and 2.5 μL H_2O_2 (30% aqueous) in dark (Ethanol-d_6, 400 MHz). The peak at 10.62 ppm is due to H_2O_2.

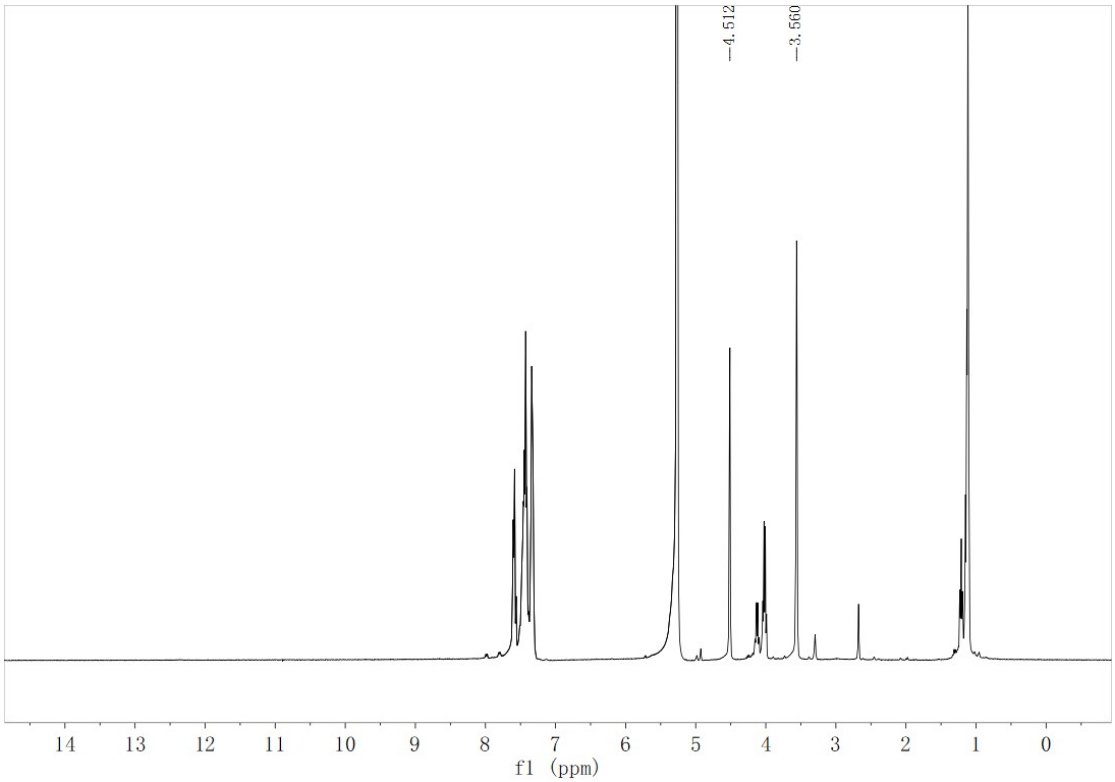


Fig. S7 ^{1}H spectra of the reaction mixture and 2.5 μL H_2O in dark (Ethanol-d_6, 400 MHz). The peak at 4.51 ppm is due to H_2O.

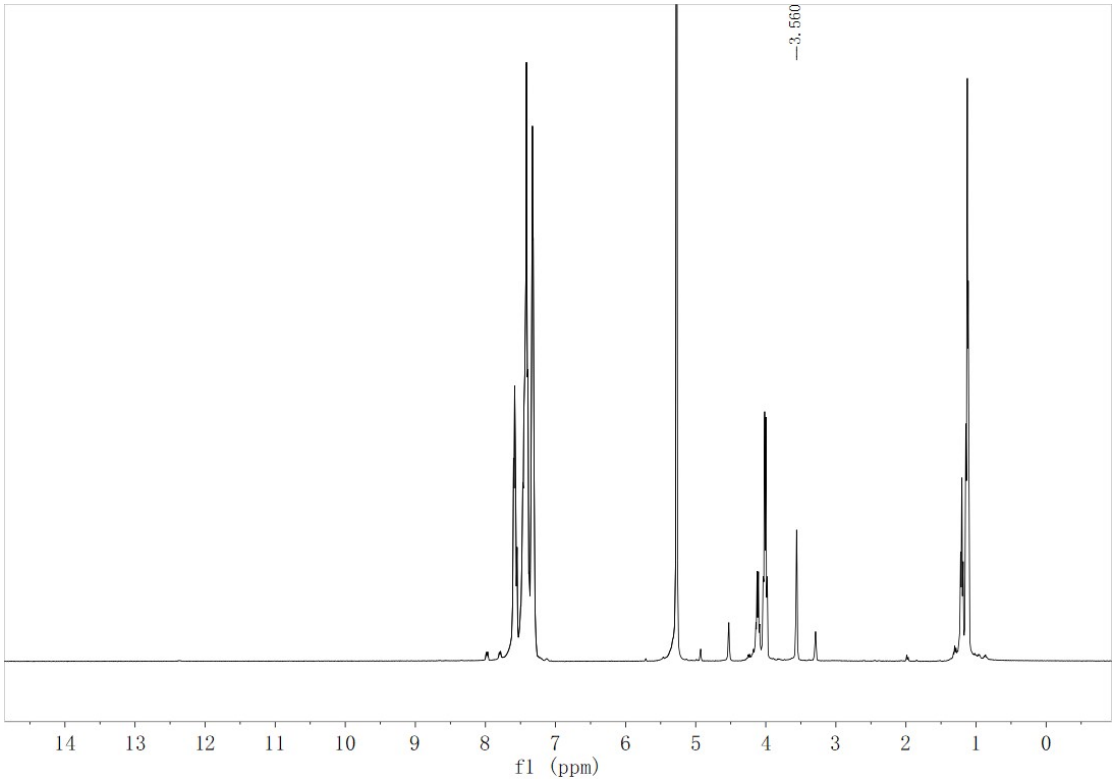


Fig. S8 ^{1}H spectra of **1a** (Ethanol-d_6, 400 MHz).

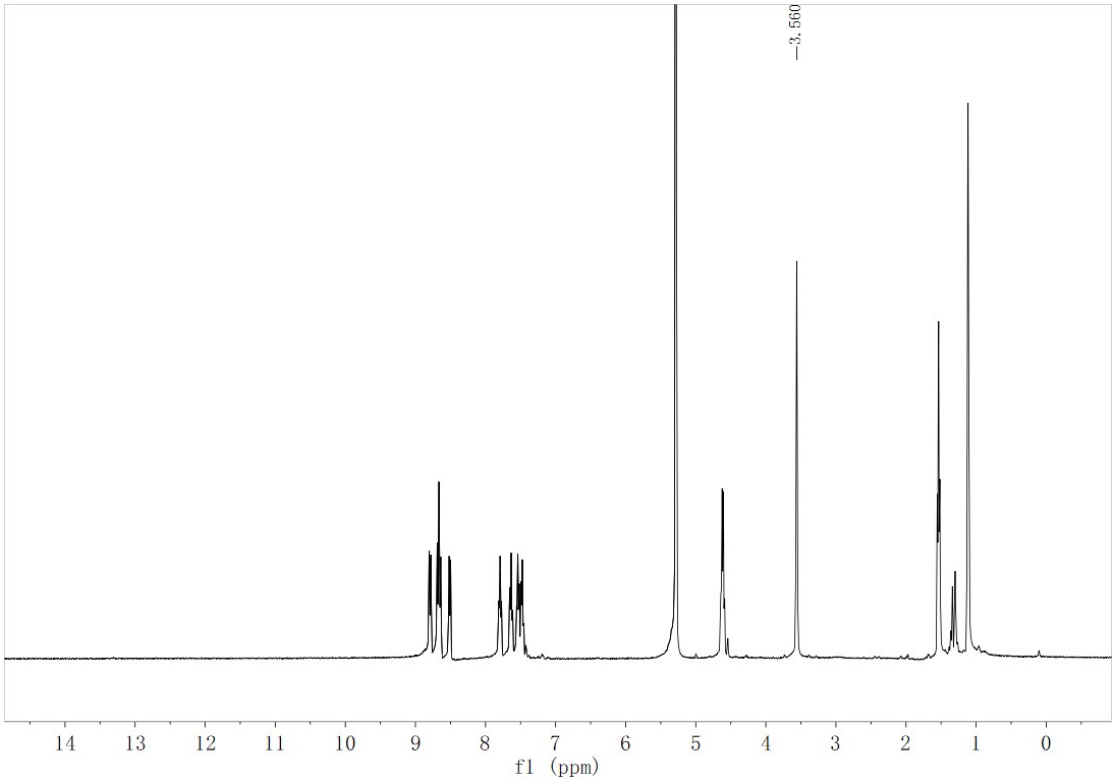


Fig. S9 ^{1}H spectra of **2a** (Ethanol-d_6, 400 MHz).

We did not detect H_2O_2 by *in situ* ^{1}H NMR analysis after the reaction mixture in Ethanol-d_6 was irradiated with sunlight for 5 hours in air at room temperature (Fig. S5), increased possibly due to the H_2O_2 participated in the catalytic cycle to convert I_2 to IOH.

5.6 Electron paramagnetic resonance (EPR) experiments

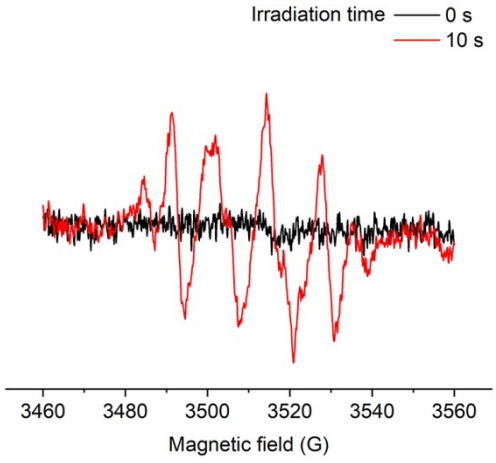


Fig. S10 Electron spin resonance (ESR) spectrum: solution of DMPO (0.56 M), **1a** (2×10^{-2} M) and NIS (1×10^{-3} M) in air-saturated i-PrOH without irradiation (labeled by black line); solution of DMPO (0.56 M), **1a** (2×10^{-2} M) and NIS (1×10^{-3} M) in air-saturated i-PrOH upon irradiation with sunlight for 10 s (labeled by red line).

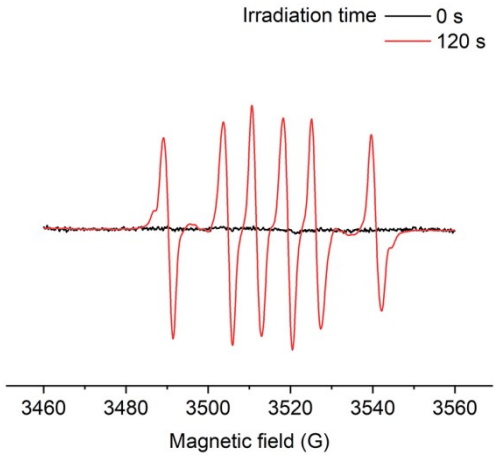


Fig. S11 Electron spin resonance (ESR) spectrum: solution of DMPO (0.48 M), **1a** (2×10^{-2} M) and NIS (1×10^{-3} M) in deaerated i-PrOH without irradiation (labeled by black line); solution of DMPO (0.48 M), **1a** (2×10^{-2} M) and NIS (1×10^{-3} M) in deaerated i-PrOH upon irradiation with sunlight for 120 s (labeled by red line).

5.7 ESI-HRMS spectra

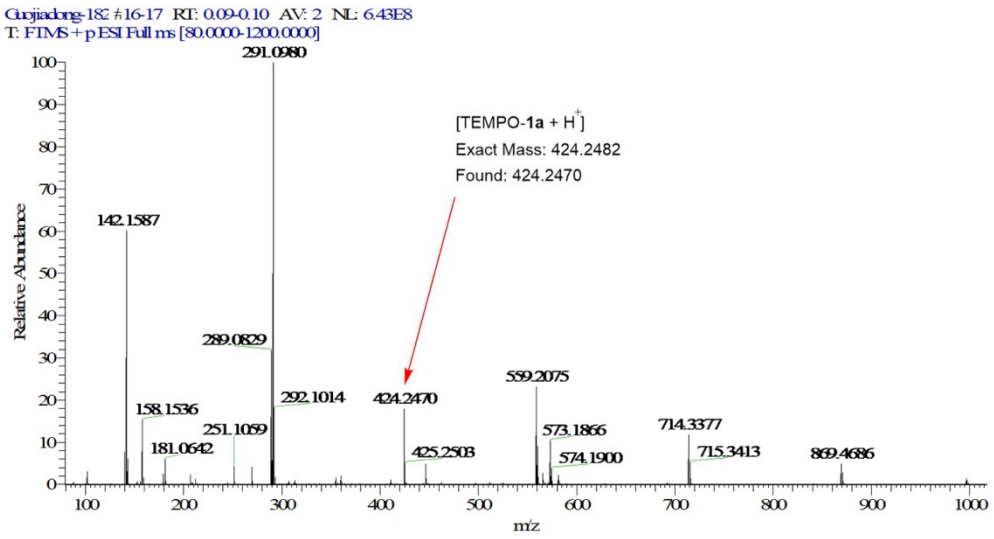
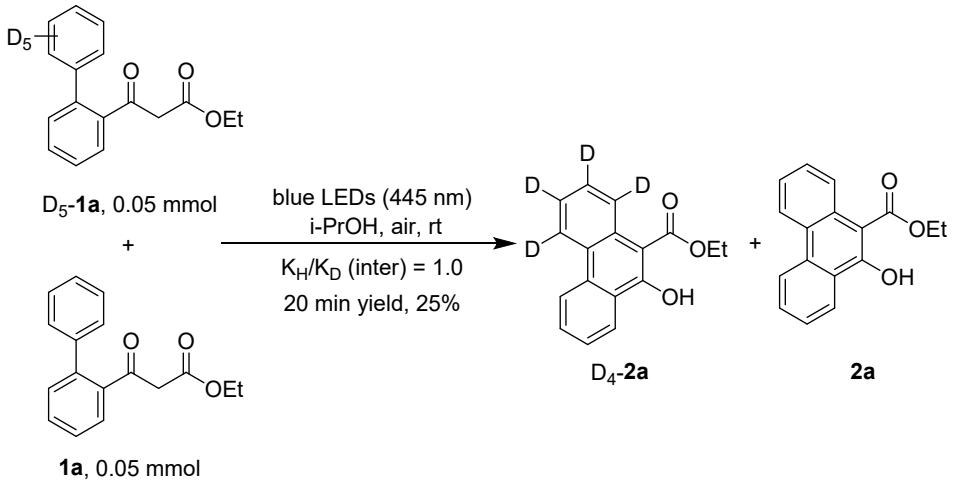


Fig. S12 ESI-HRMS spectra of TEMPO-**1a**.

5.8 Competing kinetic isotope effect (KIE) experiments

Intermolecular KIE with D_5-**1a** and **1a**



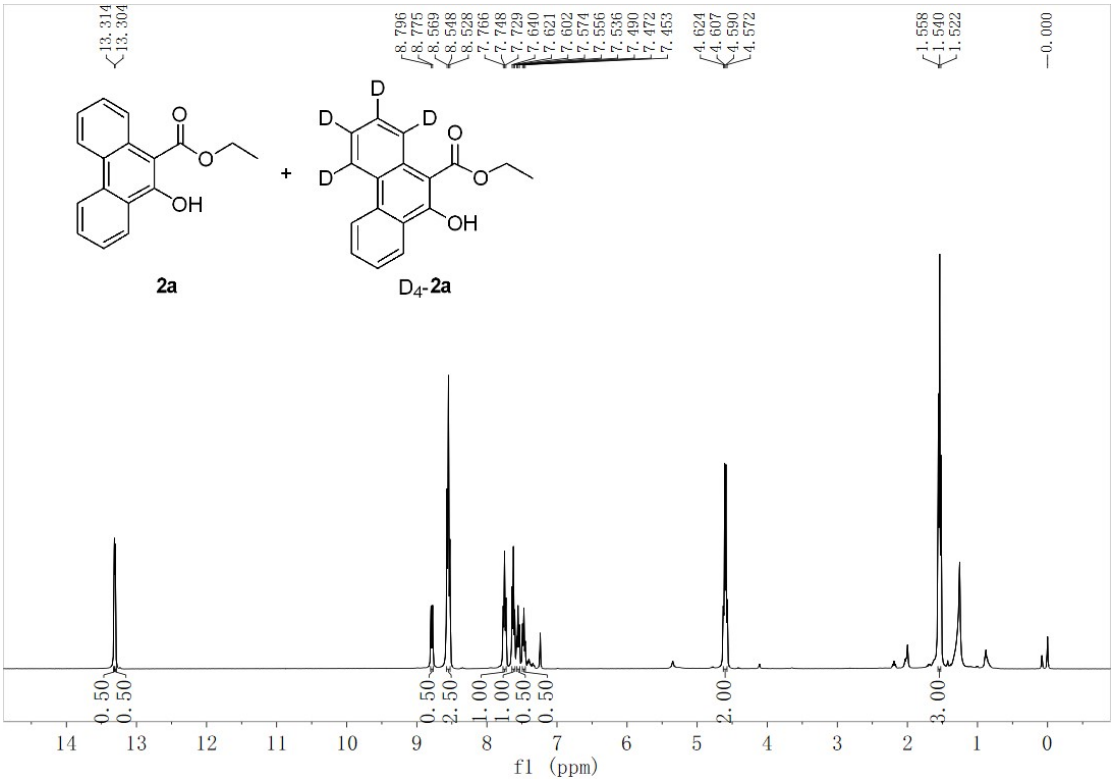


Fig. S13 Intermolecular KIE with D_5-**1a** and **1a**.

5.9 Scale-up experiment

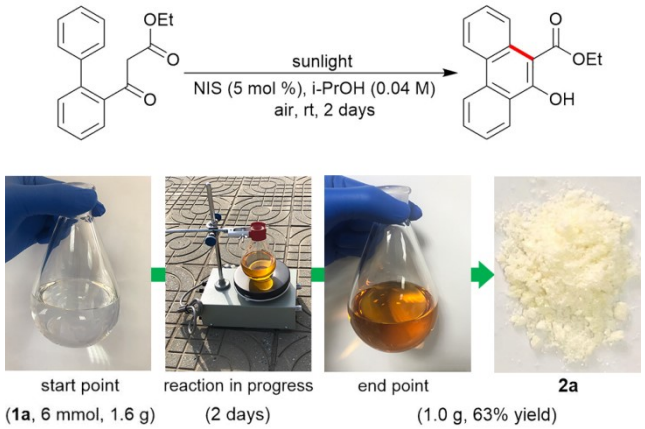


Fig. S14 Scale-up experiment.

Scale up experiment: A 250 mL eggplant-type flask was loaded with substrate **1a** (6 mmol, 1.6 g, 1 equiv), NIS (0.3 mmol, 67.5 mg, 0.05 equiv) in i-PrOH (0.04 M, 150 mL). The sample was then irradiated by sunlight for 20 h (2 days). Upon completion of the reaction, the solvent was then removed under vacuum. The residue was purified with chromatography column on silica gel using mixtures of petroleum ether and ethyl acetate to give the corresponding products **2a** (1.0 g, 63% yield).

5.10 Supplementary data

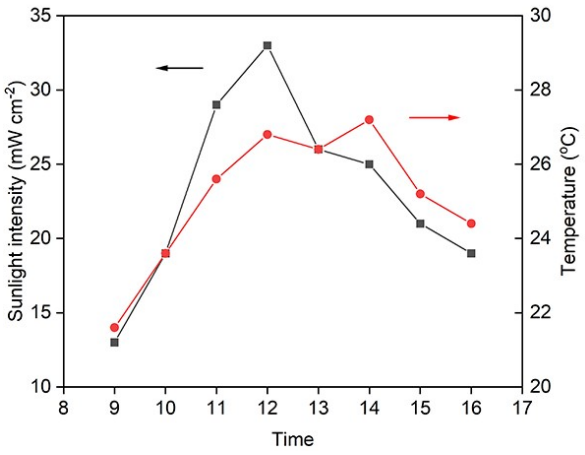
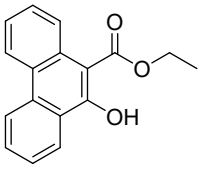


Figure S15. The variation of sunlight intensity (■) and ambient temperature (•) during sunlight photocatalysis.

6. Characterization Data for Compounds

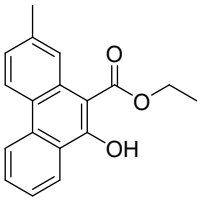
Ethyl 10-hydroxyphenanthrene-9-carboxylate



2a

White solid; yield 23.2 mg, 87%; $\mathbf{R_f}$ = 0.36 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.32 (s, 1H), 8.79 (d, *J* = 8.4 Hz, 1H), 8.58–8.53 (m, 3H), 7.76 (t, *J* = 7.2 Hz, 1H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 6.8 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 4.60 (q, *J* = 7.2 Hz, 2H), 1.54 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.1, 162.8, 133.8, 130.6, 129.6, 127.7, 127.0, 126.2, 126.1, 125.4, 125.1, 124.4, 123.0, 122.6, 101.7, 62.2, 14.5; **ESI-HRMS**: m/z Calcd for $C_{17}H_{13}O_3^-$ [M–H]$^-$: 265.0870, found 265.0870.

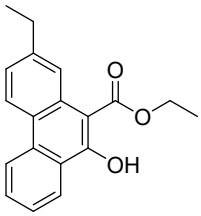
Ethyl 10-hydroxy-7-methylphenanthrene-9-carboxylate



2b

Pale yellow solid; yield 18.8 mg, 67%; $\mathbf{R_f}$ = 0.36 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.23 (s, 1H), 8.54 (s, 1H), 8.47 (t, *J* = 9.6 Hz, 2H), 8.37 (d, *J* = 8.4 Hz, 1H), 7.68 (t, *J* = 7.2 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 8.4 Hz, 1H), 4.55 (q, *J* = 7.2 Hz, 2H), 2.49 (s, 3H), 1.52 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.0, 162.8, 137.3, 133.8, 130.4, 129.6, 126.4, 126.1, 125.8, 125.0, 125.0, 124.0, 122.8, 122.3, 101.5, 62.0, 22.3, 14.4; **ESI-HRMS**: m/z Calcd for $C_{18}H_{15}O_3^-$ [M–H]$^-$: 279.1027, found 279.1028.

Ethyl 7-ethyl-10-hydroxyphenanthrene-9-carboxylate

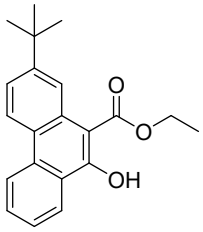


2c

Pale yellow solid; yield 22.1 mg, 75%; $\mathbf{R_f}$ = 0.36 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.26 (s, 1H), 8.59 (s, 1H), 8.47 (dd, *J* = 12.8 Hz, *J* = 8.4 Hz, 2H), 8.39 (d, *J* = 8.4 Hz, 1H), 7.67 (t, *J*

= 8.0 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 1H), 4.54 (q, *J* = 7.2 Hz, 2H), 2.80 (q, *J* = 7.6 Hz, 2H), 1.51 (t, *J* = 7.2 Hz, 3H), 1.33 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.0, 162.9, 143.5, 133.8, 130.4, 129.7, 126.4, 125.0, 125.0, 124.7 (×2), 124.2, 122.8, 122.3, 101.6, 62.0, 29.4, 15.4, 14.4; **ESI-HRMS**: m/z Calcd for $C_{19}H_{17}O_3^-$ $[M–H]^–$: 293.1183, found 293.1185.

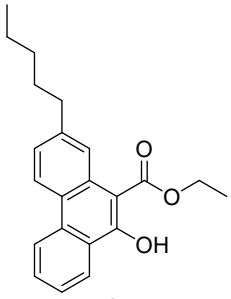
Ethyl 7-(*tert*-butyl)-10-hydroxyphenanthrene-9-carboxylate



2d

Pale yellow solid; yield 22.9 mg, 71%; **R_f** = 0.32 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.36 (s, 1H), 8.85 (s, 1H), 8.51–8.43 (m, 3H), 7.69 (t, *J* = 7.6 Hz, 1H), 7.57–7.50 (m, 2H), 4.56 (q, *J* = 7.2 Hz, 2H), 1.55 (t, *J* = 7.2 Hz, 3H), 1.43 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.3, 163.1, 150.4, 133.7, 130.5, 129.4, 126.5, 125.1, 125.1, 124.0, 122.7, 122.4, 122.4 (×2), 101.8, 62.0, 35.3, 31.6, 14.5; **ESI-HRMS**: m/z Calcd for $C_{21}H_{21}O_3^-$ $[M–H]^–$: 321.1496, found 321.1496.

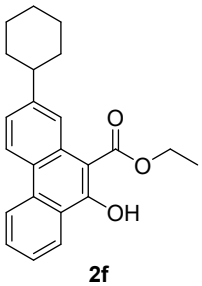
Ethyl 10-hydroxy-7-pentylphenanthrene-9-carboxylate



2e

White solid; yield 28.3 mg, 84%; **R_f** = 0.32 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.24 (s, 1H), 8.63 (s, 1H), 8.56–8.48 (m, 3H), 7.75 (t, *J* = 7.2 Hz, 1H), 7.60 (t, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 4.61 (q, *J* = 7.2 Hz, 2H), 2.79 (t, *J* = 7.2 Hz, 2H), 1.77–1.75 (m, 2H), 1.57 (t, *J* = 6.8 Hz, 3H), 1.39 (s, 4H), 0.92 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.1, 162.9, 142.3, 133.9, 130.5, 129.7, 126.5, 125.5, 125.3, 125.1, 125.0, 124.3, 122.8, 122.4, 101.6, 62.1, 36.6, 31.7, 31.1, 22.7, 14.4, 14.2; **ESI-HRMS**: m/z Calcd for $C_{22}H_{23}O_3^-$ $[M–H]^–$: 335.1653, found 335.1653.

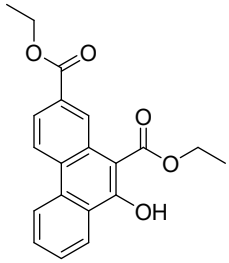
Ethyl 7-cyclohexyl-10-hydroxyphenanthrene-9-carboxylate



2f

White solid; yield 23.7 mg, 68%; $\mathbf{R_f}$ = 0.32 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.26 (s, 1H), 8.64 (s, 1H), 8.48 (dd, *J* = 11.2 Hz, *J* = 8.8 Hz, 2H), 8.40 (d, *J* = 8.4 Hz, 1H), 7.67 (t, *J* = 7.2 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 1H), 4.55 (q, *J* = 7.2 Hz, 2H), 2.67–2.61 (m, 1H), 1.94 (dd, *J* = 42.8 Hz, *J* = 10.8 Hz, 4H), 1.79 (d, *J* = 12.8 Hz, 1H), 1.53 (t, *J* = 7.2 Hz, 3H), 1.50–1.40 (m, 3H), 1.33–1.25 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.1, 162.9, 147.3, 133.8, 130.4, 129.7, 126.4, 125.0 (×2), 124.4, 124.0, 123.8, 122.8, 122.3, 101.7, 62.0, 45.0, 34.8, 27.1, 26.5, 14.5; **ESI-HRMS**: m/z Calcd for $C_{23}H_{23}O_3^-$ $[M–H]^–$: 347.1653, found 347.1652.

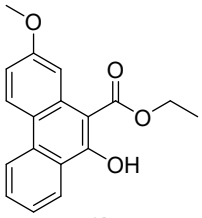
Diethyl 9-hydroxyphenanthrene-2,10-dicarboxylate



2g

White solid; yield 21.7 mg, 64%; $\mathbf{R_f}$ = 0.34 (petroleum ether/ethyl acetate 20:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.44 (s, 1H), 9.56 (s, 1H), 8.55 (t, *J* = 8.0 Hz, 3H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.78 (t, *J* = 7.2 Hz, 1H), 7.67 (t, *J* = 7.6 Hz, 1H), 4.61 (q, *J* = 6.8 Hz, 2H), 4.45 (q, *J* = 6.8 Hz, 2H), 1.62 (t, *J* = 6.8 Hz, 3H), 1.46 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.8, 167.1, 163.4, 133.0, 130.8, 129.2, 129.1, 129.1, 128.4, 128.0, 126.2, 125.2, 124.4, 123.2, 123.0, 101.6, 62.5, 61.2, 14.6, 14.3; **ESI-HRMS**: m/z Calcd for $C_{20}H_{17}O_5^-$ $[M–H]^–$: 337.1081, found 337.1082.

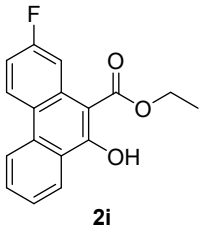
Ethyl 10-hydroxy-7-methoxyphenanthrene-9-carboxylate



2h

White solid; yield 23.1 mg, 78%; $\mathbf{R_f}$ = 0.33 (petroleum ether/ethyl acetate 20:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.43 (s, 1H), 8.50 (d, *J* = 8.4 Hz, 1H), 8.46 (d, *J* = 8.8 Hz, 2H), 8.33 (s, 1H), 7.72 (t, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.10 (d, *J* = 8.8 Hz, 1H), 4.59 (q, *J* = 6.8 Hz, 2H), 3.93 (s, 3H), 1.56 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.1, 163.8, 159.2, 134.0, 131.2, 130.7, 125.9, 125.2, 124.4, 124.3, 122.1, 120.4, 113.7, 108.2, 101.3, 62.1, 55.3, 14.5; **ESI-HRMS**: m/z Calcd for $C_{18}H_{15}O_4^-$ [M–H]$^-$: 295.0976, found 295.0973.

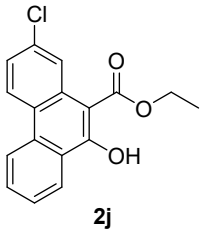
Ethyl 7-fluoro-10-hydroxyphenanthrene-9-carboxylate



2i

White solid; yield 25.6 mg, 90%; $\mathbf{R_f}$ = 0.30 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.49 (s, 1H), 8.46–8.35 (m, 4H), 7.69 (t, *J* = 6.8 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.14 (t, *J* = 8.0 Hz, 1H), 4.57 (q, *J* = 7.2 Hz, 2H), 1.54 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 164.1, 162.3 (d, $^1J_{C,F}$ = 244.6 Hz), 133.4, 131.3 (d, $^3J_{C,F}$ = 10.2 Hz), 130.8, 126.7, 125.2, 124.9, 124.8, 122.6 (d, $^4J_{C,F}$ = 1.3 Hz), 122.3, 112.6 (d, $^2J_{C,F}$ = 23.6 Hz), 111.6 (d, $^2J_{C,F}$ = 25.7 Hz), 101.0 (d, $^4J_{C,F}$ = 3.1 Hz), 62.4, 14.4; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -112.74 (s, 1F); **ESI-HRMS**: m/z Calcd for $C_{17}H_{12}FO_3^-$ [M–H]$^-$: 283.0776, found 283.0781.

Ethyl 7-chloro-10-hydroxyphenanthrene-9-carboxylate

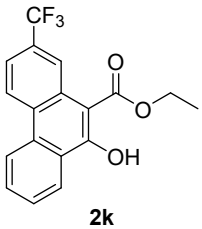


2j

White solid; yield 21.1 mg, 70%; $\mathbf{R_f}$ = 0.30 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.42 (s, 1H), 8.71 (s, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 8.33 (dd, *J* = 15.6 Hz, *J* = 8.4 Hz, 2H), 7.68 (t, *J* = 7.2 Hz, 1H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.33 (d, *J* = 8.8 Hz, 1H), 4.57 (q, *J* = 7.2 Hz, 2H), 1.54 (t, *J* = 6.8 Hz, 3H); **^{13}C**

NMR (101 MHz, $CDCl_3$) δ 172.6, 163.8, 133.8, 133.1, 130.8, 130.7, 127.1, 125.6, 125.2 (×2), 124.5, 124.4, 124.2, 122.4, 100.7, 62.4, 14.3; **ESI-HRMS**: m/z Calcd for $C_{17}H_{12}ClO_3^-$ $[M–H]^–$: 299.0480, found 299.0479.

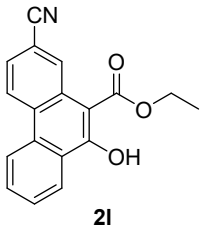
Ethyl 10-hydroxy-7-(trifluoromethyl)phenanthrene-9-carboxylate



2k

White solid; yield 29.1 mg, 87%; $\mathbf{R_f}$ = 0.30 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.42 (s, 1H), 9.05 (s, 1H), 8.44 (d, *J* = 8.0 Hz, 2H), 8.39 (d, *J* = 8.4 Hz, 1H), 7.71 (t, *J* = 6.8 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.55 (d, *J* = 8.4 Hz, 1H), 4.55 (q, *J* = 6.8 Hz, 2H), 1.54 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.5, 163.8, 132.7, 130.9, 129.1, 129.1 (d, $^2J_{C,F}$ = 32.0 Hz), 128.0, 127.9, 126.0, 125.2, 124.8 (d, $^1J_{C,F}$ = 273.2 Hz), 123.5, 123.5, 122.8, 120.1 (d, $^4J_{C,F}$ = 3.0 Hz), 101.1, 62.5, 14.1; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -62.41 (s, 3F); **ESI-HRMS**: m/z Calcd for $C_{18}H_{12}F_3O_3^-$ $[M–H]^–$: 333.0744, found 333.0744.

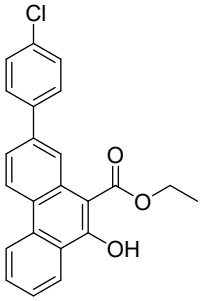
Ethyl 7-cyano-10-hydroxyphenanthrene-9-carboxylate



2l

White solid; yield 27.4 mg, 94%; $\mathbf{R_f}$ = 0.40 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.57 (s, 1H), 9.16 (s, 1H), 8.60–8.52 (m, 3H), 7.83 (t, *J* = 7.2 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.65 (d, *J* = 8.8 Hz, 1H), 4.65 (q, *J* = 7.2 Hz, 2H), 1.58 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 164.2, 132.6, 131.3, 131.2, 129.5, 128.8, 128.6, 126.4, 126.0, 125.5, 123.9, 123.1, 119.8, 111.1, 100.7, 62.8, 14.5; **ESI-HRMS**: m/z Calcd for $C_{18}H_{12}NO_3^-$ $[M–H]^–$: 290.0823, found 290.0818.

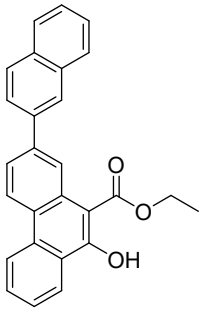
Ethyl 7-(4-chlorophenyl)-10-hydroxyphenanthrene-9-carboxylate



2m

White solid; yield 28.3 mg, 75%; $\mathbf{R_f}$ = 0.40 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.36 (s, 1H), 8.93 (s, 1H), 8.48–8.42 (m, 3H), 7.68 (t, *J* = 7.2 Hz, 1H), 7.58–7.54 (m, 4H), 7.41 (d, *J* = 8.0 Hz, 2H), 4.52 (q, *J* = 6.8 Hz, 2H), 1.50 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.9, 163.4, 140.0, 138.6, 133.6, 133.4, 130.6, 129.9, 129.1, 128.5, 127.0, 125.4, 125.4, 125.2, 124.5, 123.5, 122.8, 122.5, 101.4, 62.2, 14.4; **ESI-HRMS**: m/z Calcd for $C_{23}H_{16}ClO_3^-$ $[M–H]^–$: 375.0793, found 375.0794.

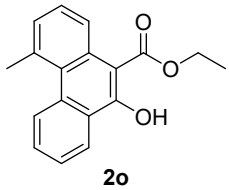
Ethyl 10-hydroxy-7-(naphthalen-2-yl)phenanthrene-9-carboxylate



2n

White solid; yield 32.2 mg, 82%; $\mathbf{R_f}$ = 0.35 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.41 (s, 1H), 9.21 (s, 1H), 8.60 (d, *J* = 8.4 Hz, 1H), 8.55 (t, *J* = 7.2 Hz, 2H), 8.17 (s, 1H), 7.96–7.82 (m, 5H), 7.75 (t, *J* = 7.2 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.52–7.48 (m, 2H), 4.59 (q, *J* = 6.8 Hz, 2H), 1.59 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.1, 163.4, 139.9, 138.9, 134.0, 133.6, 132.9, 130.7, 130.0, 128.7, 128.4, 127.8, 127.0, 126.5, 126.2, 126.1, 125.7, 125.4, 125.4, 125.2, 125.1, 123.6, 123.5, 122.6, 101.7, 62.2, 14.5; **ESI-HRMS**: m/z Calcd for $C_{27}H_{19}O_3^-$ $[M–H]^–$: 391.1340, found 391.1342.

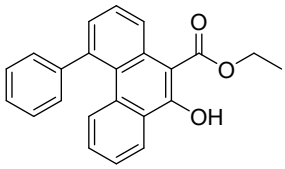
Ethyl 10-hydroxy-5-methylphenanthrene-9-carboxylate



2o

Pale yellow solid; yield 14.6 mg, 52%; $\mathbf{R_f}$ = 0.41 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.86 (s, 1H), 8.60–8.55 (m, 3H), 7.67 (t, *J* = 7.2 Hz, 1H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.31 (d, *J* = 7.2 Hz, 1H), 4.56 (q, *J* = 7.2 Hz, 2H), 2.99 (s, 3H), 1.49 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 161.4, 135.2, 134.5, 130.9, 129.1, 128.8, 127.7, 126.7, 126.5, 126.4, 126.3, 124.6, 124.0, 102.7, 62.1, 27.3, 14.5; **ESI-HRMS**: m/z Calcd for $C_{18}H_{15}O_3^-$ $[M–H]^–$: 279.1027, found 279.1029.

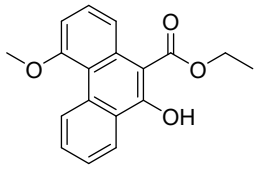
Ethyl 10-hydroxy-5-phenylphenanthrene-9-carboxylate



2p

White solid; yield 15.8 mg, 46%; $\mathbf{R_f}$ = 0.32 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.08 (s, 1H), 8.73 (d, *J* = 8.0 Hz, 1H), 8.48 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.43–7.30 (m, 7H), 7.14 (t, *J* = 7.2 Hz, 1H), 4.60 (q, *J* = 6.8 Hz, 2H), 1.53 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.8, 162.3, 145.6, 140.6, 133.7, 131.1, 129.2, 129.1, 129.0, 128.8, 128.2, 127.1, 126.5, 126.4, 126.4, 125.2, 125.0, 124.4, 102.1, 62.2, 14.5; **ESI-HRMS**: m/z Calcd for $C_{23}H_{17}O_3^-$ $[M–H]^–$: 341.1183, found 341.1182.

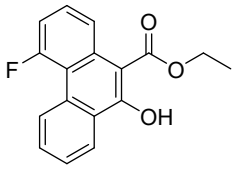
Ethyl 10-hydroxy-5-methoxyphenanthrene-9-carboxylate



2q

Pale yellow solid; yield 17.2 mg, 58%; $\mathbf{R_f}$ = 0.35 (petroleum ether/ethyl acetate 20:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.95 (s, 1H), 9.57 (d, *J* = 8.8 Hz, 1H), 8.58 (d, *J* = 8.0 Hz, 1H), 8.37 (d, *J* = 8.4 Hz, 1H), 7.74 (t, *J* = 7.2 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 1H), 7.03 (d, *J* = 8.0 Hz, 1H), 4.58 (q, *J* = 6.8 Hz, 2H), 4.08 (s, 3H), 1.52 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.8, 162.1, 158.8, 133.4, 132.0, 130.1, 128.6, 127.3, 126.3, 125.6, 124.3, 118.7, 117.0, 106.9, 102.3, 62.2, 55.9, 14.5; **ESI-HRMS**: m/z Calcd for $C_{18}H_{15}O_4^-$ $[M–H]^–$: 295.0976, found 295.0973.

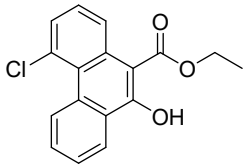
Ethyl 5-fluoro-10-hydroxyphenanthrene-9-carboxylate



2r

White solid; yield 14.3 mg, 50%; $\mathbf{R_f}$ = 0.38 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.21 (s, 1H), 9.02–9.00 (m, 1H),8.56 (t, *J* = 8.8 Hz, 2H), 7.77 (t, *J* = 7.6 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.46 (q, *J* = 8.0 Hz, 1H), 7.19 (dd, *J* = 14.0 Hz, *J* = 7.6 Hz, 1H), 4.59 (q, *J* = 7.2 Hz, 2H), 1.53 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 163.0, 161.7 (d, $^1J_{C,F}$ = 252.4 Hz), 132.1 (d, $^3J_{C,F}$ = 3.9 Hz), 131.5 (d, $^3J_{C,F}$ = 5.1 Hz), 130.9 (d, $^4J_{C,F}$ = 2.6 Hz), 127.7 (d, $^2J_{C,F}$ = 21.7 Hz), 127.5 (d, $^4J_{C,F}$ = 3.7 Hz), 127.2 (d, $^4J_{C,F}$ = 1.6 Hz), 125.8, 124.8, 121.8 (d, $^4J_{C,F}$ = 3.4 Hz), 115.8 (d, $^3J_{C,F}$ = 9.1 Hz), 111.5 (d, $^2J_{C,F}$ = 25.3 Hz), 101.7 (d, $^4J_{C,F}$ = 1.9 Hz), 62.4, 14.5; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -108.68 (s, 1F); **ESI-HRMS**: m/z Calcd for $C_{17}H_{12}FO_3^-$ $[M–H]^–$: 283.0776, found 283.0774.

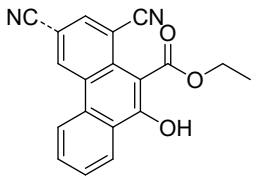
Ethyl 5-chloro-10-hydroxyphenanthrene-9-carboxylate



2s

Pale yellow solid; yield 19.3 mg, 64%; $\mathbf{R_f}$ = 0.41 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.00 (s, 1H), 9.59 (d, *J* = 8.4 Hz, 1H), 8.65 (d, *J* = 8.4 Hz, 1H), 8.57 (d, *J* = 8.0 Hz, 1H), 7.74 (t, *J* = 6.8 Hz, 1H), 7.67 (t, *J* = 7.6 Hz, 1H), 7.56 (d, *J* = 7.6 Hz, 1H), 7.40 (t, *J* = 8.0 Hz, 1H), 4.59 (q, *J* = 6.8 Hz, 2H), 1.52 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.3, 162.3, 132.6, 132.5, 131.7, 129.2, 128.5, 127.7, 127.4, 127.0, 126.5, 124.8, 124.5, 124.1, 102.1, 62.4, 14.5; **ESI-HRMS**: m/z Calcd for $C_{17}H_{12}ClO_3^-$ $[M–H]^–$: 299.0480, found 299.0480.

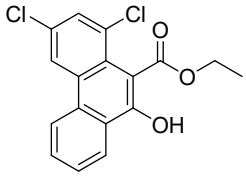
Ethyl 8-cyano-10-hydroxyphenanthrene-9-carboxylate and ethyl 6-cyano-10-hydroxyphenanthrene-9-carboxylate



2t:(**2t'**) = 1:2.2

White solid; yield 26.2 mg, 90%; $\mathbf{R_f}$ = 0.39 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.57 (s, 1H), 11.57 (s, 0.45H), 8.71 (d, *J* = 8.8 Hz, 1H), 8.63–8.60 (m, 1.45H), 8.43–8.40 (m, 2H), 8.29 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 7.2 Hz, 0.45H), 7.77–7.73 (m, 1.45H), 7.66–7.59 (m, 2.35H), 7.47 (t, *J* = 8.0 Hz, 0.45H), 4.59 (q, *J* = 6.8 Hz, 2.9H), 1.55 (t, *J* = 6.8 Hz, 3H), 1.42 (t, *J* = 7.2 Hz, 1.35H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.3, 170.4, 164.9, 160.9, 134.6, 132.5, 132.4, 132.3, 131.3, 130.9, 130.0, 129.0, 128.1, 128.0, 127.7, 127.1, 127.1, 126.7, 125.6, 125.5, 125.2, 125.0, 124.1, 122.5, 122.2, 119.5, 119.4, 109.6, 107.3, 102.4, 101.0, 62.7, 62.1, 14.4, 13.9; **ESI-HRMS**: m/z Calcd for $C_{18}H_{12}NO_3^-$ [M–H]$^-$: 290.0823, found 290.0819.

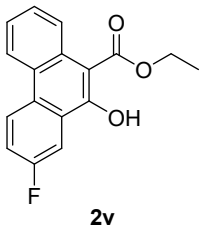
Ethyl 6,8-dichloro-10-hydroxyphenanthrene-9-carboxylate



2u

White solid; yield 18.1 mg, 54%; $\mathbf{R_f}$ = 0.46 (petroleum ether/ethyl acetate 20:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.55 (s, 1H), 8.45 (d, *J* = 8.4 Hz, 2H), 8.41 (d, *J* = 1.6 Hz, 1H), 7.78 (t, *J* = 7.2 Hz, 1H), 7.69 (t, *J* = 7.6 Hz, 1H), 7.59 (d, *J* = 2.0 Hz, 1H), 4.44 (q, *J* = 6.8 Hz, 2H), 1.36 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.7, 158.3, 132.1, 131.9, 130.7, 130.5, 129.5, 129.4, 128.4, 126.5, 125.3, 124.9, 123.0, 121.3, 103.3, 62.3, 14.1; **ESI-HRMS**: m/z Calcd for $C_{17}H_{11}Cl_2O_3^-$ [M–H]$^-$: 333.0091, found 333.0093.

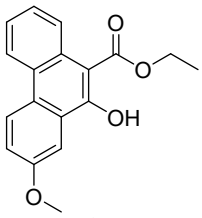
Ethyl 2-fluoro-10-hydroxyphenanthrene-9-carboxylate



2v

Pale yellow solid; yield 27.9 mg, 98%; $\mathbf{R_f}$ = 0.38 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.19 (s, 1H), 8.78 (d, *J* = 8.4 Hz, 1H), 8.56–8.53 (m, 1H), 8.48 (d, *J* = 8.0 Hz, 1H), 8.14 (d, *J* = 9.6 Hz, 1H), 7.56 (t, *J* = 6.8 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 2H), 4.62 (q, *J* = 7.2 Hz, 2H), 1.55 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.8, 161.7 (d, $^1J_{C,F}$ = 247.9 Hz), 161.6 (d, $^4J_{C,F}$ = 3.3 Hz), 130.2, 129.0, 127.5, 126.9 (d, $^3J_{C,F}$ = 8.7 Hz), 126.1, 125.8, 125.0 (d, $^3J_{C,F}$ = 8.3 Hz), 124.6, 122.7, 119.2 (d, $^2J_{C,F}$ = 23.8 Hz), 109.8 (d, $^2J_{C,F}$ = 22.8 Hz), 102.6, 62.3, 14.4; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -113.76 (s, 1F); **ESI-HRMS**: m/z Calcd for $C_{17}H_{12}FO_3^-$ [M–H]$^-$: 283.0776, found 283.0784.

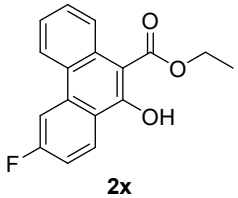
Ethyl 10-hydroxy-2-methoxyphenanthrene-9-carboxylate



2w

White solid; yield 21.1 mg, 71%; $\mathbf{R_f}$ = 0.37 (petroleum ether/ethyl acetate 20:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.39 (s, 1H), 8.77 (d, *J* = 8.4 Hz, 1H), 8.42 (d, *J* = 8.8 Hz, 2H), 7.86 (s, 1H), 7.53 (t, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 7.19 (d, *J* = 8.8 Hz, 1H), 4.57 (q, *J* = 6.8 Hz, 2H), 3.98 (s, 3H), 1.52 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.1, 163.3, 161.7, 135.9, 130.3, 127.8, 127.1, 126.2, 125.7, 123.9, 123.0, 119.6, 116.4, 104.3, 99.9, 61.9, 55.6, 14.5; **ESI-HRMS**: m/z Calcd for $C_{18}H_{15}O_4^-$[M–H]$^-$: 295.0976, found 295.0973.

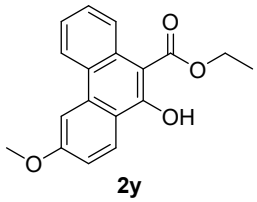
Ethyl 3-fluoro-10-hydroxyphenanthrene-9-carboxylate



2x

Pale yellow solid; yield 26.2 mg, 92%; $\mathbf{R_f}$ = 0.41 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.35 (s, 1H), 8.76 (d, *J* = 7.6 Hz, 1H), 8.49–8.48 (m, 1H), 8.35 (d, J = 7.2 Hz, 1H), 8.10 (d, J = 10.8 Hz, 1H), 7.55 (t, J = 7.6 Hz, 1H), 7.44 (t, J = 7.2 Hz, 1H), 7.33–7.29 (m, 1H), 4.59 (q, J = 7.2 Hz, 2H), 1.54 (t, J = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.0, 164.3 (d, $^1J_{C,F}$ = 251.0 Hz), 162.5, 136.1 (d, $^3J_{C,F}$ = 9.2 Hz), 130.1, 128.3, 128.0 (d, $^3J_{C,F}$ = 9.6 Hz), 126.2, 125.4 (d, $^4J_{C,F}$ = 3.6 Hz), 124.4, 123.1, 122.0, 115.8 (d, $^2J_{C,F}$ = 23.6 Hz), 107.9 (d, $^2J_{C,F}$ = 23.0 Hz), 101.1, 62.2, 14.5; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -108.12 (s, 1F); **ESI-HRMS**: m/z Calcd for $C_{17}H_{12}FO_3^-$ [M–H]$^-$: 283.0776, found 283.0783.

Ethyl 10-hydroxy-3-methoxyphenanthrene-9-carboxylate

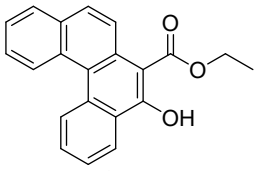


2y

White solid; yield 13.1 mg, 44%; $\mathbf{R_f}$ = 0.33 (petroleum ether/ethyl acetate 20:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.31 (s, 1H), 8.77 (d, *J* = 8.0 Hz, 1H), 8.47 (d, *J* = 8.8 Hz, 2H), 7.88 (d, *J* = 2.0 Hz, 1H), 7.49 (dt, *J* = 20.4 Hz,

J = 6.8 Hz, 2H), 7.37 (dd, *J* = 9.2 Hz, *J* = 2.0 Hz, 1H), 4.61 (q, *J* = 7.2 Hz, 2H), 3.98 (s, 3H), 1.55 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.2, 162.2, 158.8, 128.5, 128.1, 126.8, 126.6, 126.3, 126.1, 124.4, 124.3, 122.5, 121.3, 104.8, 102.2, 62.2, 55.7, 14.5; **ESI-HRMS**: m/z Calcd for $C_{18}H_{15}O_4^-$ [M–H]$^-$: 295.0976, found 295.0973.

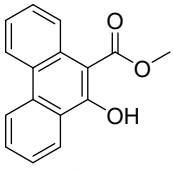
Ethyl 5-hydroxybenzo[*c*]phenanthrene-6-carboxylate



2z

Pale yellow solid; yield 10.2 mg, 32%; **R_f** = 0.38 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.99 (s, 1H), 8.85 (dd, *J* = 16.4 Hz, *J* = 8.4 Hz, 2H) 8.74 (d, *J* = 9.2 Hz, 1H), 8.63 (d, *J* = 8.0 Hz, 1H), 7.95 (d, *J* = 7.2 Hz, 1H), 7.85 (d, *J* = 9.2 Hz, 1H), 7.74 (t, *J* = 7.2 Hz, 1H), 7.64 (t, *J* = 8.0 Hz, 1H), 7.60–7.52 (m, 2H), 4.61 (q, *J* = 6.8 Hz, 2H), 1.54 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 161.8, 133.3, 131.9, 130.0, 129.5, 128.5, 128.5, 128.0, 128.0, 127.6, 126.3, 126.1, 125.8, 125.4, 124.6, 124.1, 122.8, 102.8, 62.3, 14.5; **ESI-HRMS**: m/z Calcd for $C_{21}H_{15}O_3^-$ [M–H]–: 315.1027, found 315.1027.

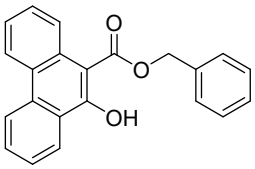
Methyl 10-hydroxyphenanthrene-9-carboxylate



2aa

Pale yellow solid; yield 14.2 mg, 56%; **R_f** = 0.38 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.21 (s, 1H), 8.69 (d, *J* = 8.4 Hz, 1H), 8.51 (d, *J* = 8.0 Hz, 3H), 7.71 (t, *J* = 7.2 Hz, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.44 (t, *J* = 7.2 Hz, 1H), 4.07 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.4, 162.9, 133.8, 130.6, 129.4, 127.7, 126.9, 126.2, 126.1, 125.3, 125.1, 124.4, 122.9, 122.5, 101.6, 52.6; **ESI-HRMS**: m/z Calcd for $C_{16}H_{11}O_3^-$ [M–H]$^-$: 251.0714, found 251.0712.

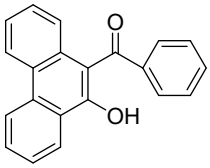
Benzyl 10-hydroxyphenanthrene-9-carboxylate



2ab

White solid; yield 26.9 mg, 82%; $\mathbf{R_f}$ = 0.38 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.19 (s, 1H), 8.74 (d, *J* = 8.4 Hz, 1H), 8.49 (t, *J* = 7.2 Hz, 3H), 7.69 (t, *J* = 7.2 Hz, 1H), 7.57 (t, *J* = 7.2 Hz, 1H) 7.50–7.32 (m, 7H), 5.53 (s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 163.0, 135.4, 133.8, 130.6, 129.5, 128.9, 128.6, 128.5, 127.7, 126.9, 126.2 (×2), 125.3, 125.1, 124.3, 122.9, 122.5, 101.5, 67.7; **ESI-HRMS**: m/z Calcd for $C_{22}H_{15}O_3^-$ $[M–H]^–$: 327.1027, found 327.1026.

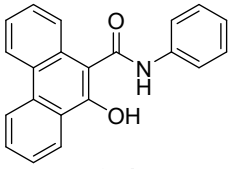
(10-hydroxyphenanthren-9-yl)(phenyl)methanone



2ac

Pale yellow solid; yield 13.5 mg, 45%; $\mathbf{R_f}$ = 0.47 (petroleum ether/ethyl acetate 20:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.74 (s, 1H), 8.61 (t, *J* = 9.2 Hz, 2H), 8.54 (d, *J* = 8.0 Hz, 1H), 7.82 (t, *J* = 7.2 Hz, 1H), 7.69 (t, *J* = 7.2 Hz, 1H), 7.63 (d, *J* = 7.2 Hz, 2H), 7.54 (t, *J* = 7.2 Hz, 1H), 7.40–7.33 (m, 4H), 7.17 (t, *J* = 7.2 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.3, 160.8, 140.5, 134.1, 132.6, 130.7, 130.4, 129.6, 128.6, 127.8, 127.2, 126.3, 126.0, 125.3, 125.2, 124.5, 123.0, 122.7, 111.1; **ESI-HRMS**: m/z Calcd for $C_{21}H_{13}O_2^-$ $[M–H]^–$: 297.0921, found 297.0917.

10-hydroxy-*N*-phenylphenanthrene-9-carboxamide

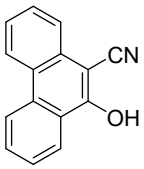


2ad

White solid; yield 7.9 mg, 25%; $\mathbf{R_f}$ = 0.58 (petroleum ether/ethyl acetate 6:1); **^{1}H NMR** (400 MHz, DMSO) δ 10.57 (s, 1H), 10.10 (s, 1H), 8.86 (d, *J* = 7.6 Hz, 1H), 8.80 (d, *J* = 7.6 Hz, 1H), 8.41 (d, *J* = 7.6 Hz, 1H), 7.85 (d, *J* = 7.6 Hz, 2H), 7.79–7.73 (m, 3H), 7.60–7.54 (m, 2H), 7.38 (t, *J* = 6.4 Hz, 2H), 7.12 (t, *J* = 6.8 Hz, 1H); **^{13}C NMR** (101 MHz, DMSO) δ 166.0, 147.2, 139.6, 131.0, 130.0, 128.7, 127.9, 127.4, 126.8, 126.1, 125.5, 124.3,

124.2, 123.5, 123.2, 123.0, 119.6, 116.7; **ESI-HRMS**: m/z Calcd for $C_{21}H_{14}NO_2^-$ [M–H]$^-$: 312.1030, found 312.1029.

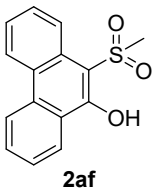
10-hydroxyphenanthrene-9-carbonitrile



2ae

Cream solid; yield 17.5 mg, 80%; $\mathbf{R_f}$ = 0.24 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, DMSO) δ 11.84 (s, 1H), 8.85 (d, *J* = 8.4 Hz, 1H), 8.78 (d, *J* = 8.0 Hz, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.88 (t, *J* = 7.2 Hz, 1H), 7.79–7.71 (m, 2H), 7.61 (t, *J* = 7.2 Hz, 1H); **^{13}C NMR** (101 MHz, DMSO) δ 158.8, 132.5, 130.4, 129.4, 128.7, 127.5, 125.6, 125.3, 124.8, 123.7, 123.6, 123.5, 123.5, 116.5, 90.2; **ESI-HRMS**: m/z Calcd for $C_{15}H_8NO^-$ [M–H]$^-$: 218.0611, found 218.0595.

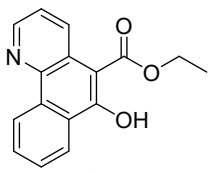
10-(methylsulfonyl)phenanthren-9-ol



2af

Pale yellow solid; yield 10.4 mg, 38%; $\mathbf{R_f}$ = 0.36 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 11.69 (s, 1H), 8.63–8.52 (m, 4H), 7.81 (t, *J* = 7.2 Hz, 1H), 7.66 (q, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 6.8 Hz, 1H), 3.35 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.4, 133.8, 131.1, 128.6, 127.6, 127.2, 126.5, 125.4, 125.3, 123.5, 123.4, 122.6, 108.7, 45.0; **ESI-HRMS**: m/z Calcd for $C_{15}H_{11}O_3S^-$ [M–H]$^-$: 271.0434, found 271.0421.

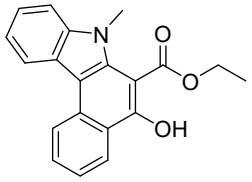
Ethyl 6-hydroxybenzo[*h*]quinoline-5-carboxylate



2ag

White solid; yield 2.9 mg, 11%; $\mathbf{R_f}$ = 0.16 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.54 (s, 1H), 9.25 (d, *J* = 7.2 Hz, 1H), 9.13 (d, *J* = 8.4 Hz, 1H), 8.81 (s, 1H), 8.54 (d, *J* = 8.0 Hz, 1H), 7.87 (t, *J* = 6.4 Hz, 1H), 7.75 (t, *J* = 7.2 Hz, 1H), 7.51–7.49 (m, 1H), 4.63 (q, *J* = 7.2 Hz, 2H), 1.57 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.8, 163.8, 146.1, 142.5, 135.0, 133.8, 131.1, 128.7, 126.8, 124.8, 124.4, 122.5, 100.0, 62.4, 14.5; **ESI-HRMS**: m/z Calcd for $C_{16}H_{12}NO_3^-$ $[M–H]^-$:266.0823, found 266.0820.

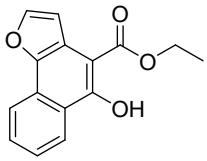
Ethyl 5-hydroxy-7-methyl-7*H*-benzo[*c*]carbazole-6-carboxylate



2ah

Pale yellow solid; yield 6.4 mg, 20%; $\mathbf{R_f}$ = 0.23 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 11.72 (s, 1H), 8.66 (d, *J* = 8.0 Hz, 1H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.43 (d, *J* = 7.6 Hz, 1H), 7.77 (t, *J* = 6.4 Hz, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.45 (q, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 7.2 Hz, 1H), 4.56 (q, *J* = 7.2 Hz, 2H), 3.85 (s, 3H), 1.47 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.4, 160.7, 141.8, 136.9, 133.1, 130.7, 125.4, 123.9, 123.4, 123.1, 123.0, 120.9, 120.9, 120.8, 110.1, 110.0, 97.0, 62.1, 35.8, 14.5; **ESI-HRMS**: m/z Calcd for $C_{20}H_{16}NO_3^-$ $[M–H]^-$:318.1136, found 318.1130.

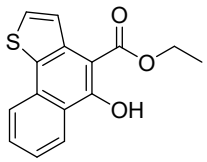
Ethyl 5-hydroxynaphtho[1,2-*b*]furan-4-carboxylate



2ai

Pale yellow solid; yield 4.4 mg, 17%; $\mathbf{R_f}$ = 0.39 (petroleum ether/ethyl acetate 50:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.36 (s, 1H), 8.47 (d, *J* = 8.4 Hz, 1H), 8.21 (d, *J* = 8.4 Hz, 1H), 7.73–7.69 (m, 2H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.23 (s, 1H), 4.54 (q, *J* = 6.8 Hz, 2H), 1.54 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.8, 159.5, 144.6, 144.4, 130.2, 125.2, 125.1, 125.0, 123.2, 120.0, 119.9, 109.5, 99.5, 61.7, 14.5; **ESI-HRMS**: m/z Calcd for $C_{15}H_{11}O_4^-$ $[M–H]^-$:255.0663, found 255.0660.

Ethyl 5-hydroxynaphtho[1,2-*b*]thiophene-4-carboxylate

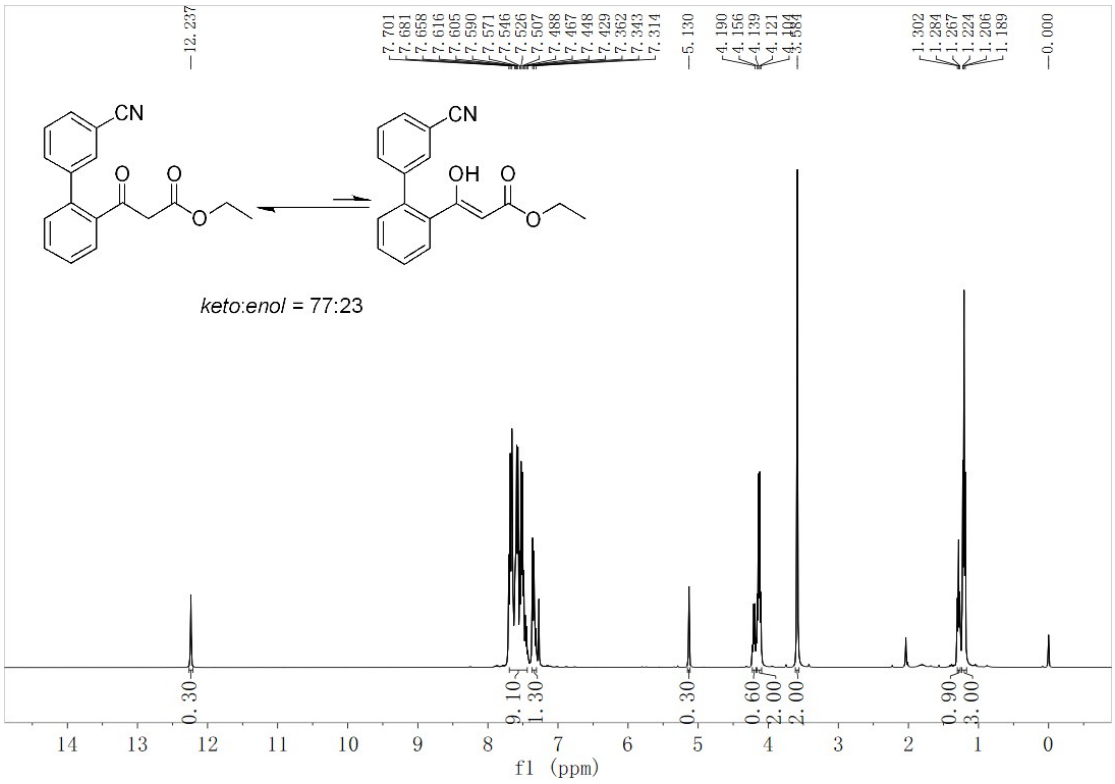


2aj

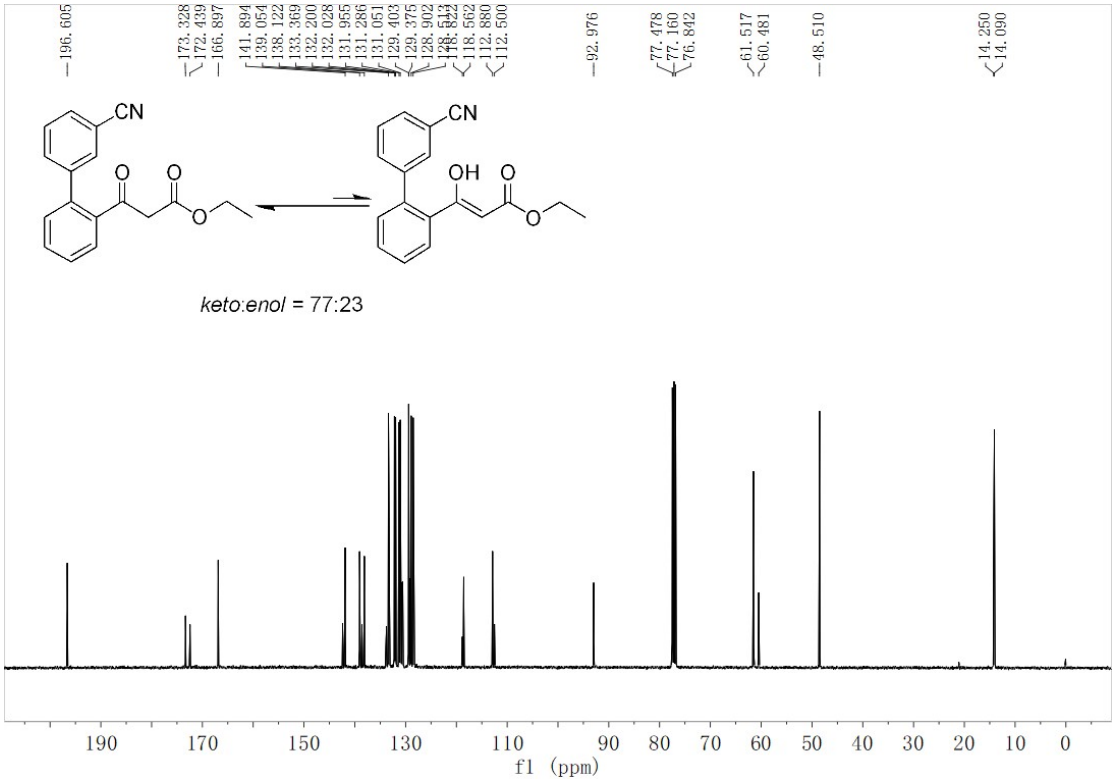
Pale yellow solid; yield 14.5 mg, 53%; $\mathbf{R_f}$ = 0.67 (petroleum ether/ethyl acetate 10:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.90 (s, 1H), 8.43 (d, *J* = 7.6 Hz, 1H), 7.97–7.95 (m, 2H), 7.62 (t, *J* = 6.4 Hz, 1H), 7.51–7.43 (m, 2H), 4.52 (q, *J* = 6.0 Hz, 2H), 1.51 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.0, 161.9, 133.7, 131.8, 130.4, 130.3, 126.6, 125.7, 125.4, 123.5, 123.4, 101.8, 61.9, 14.4; **ESI-HRMS**: m/z Calcd for $C_{15}H_{11}O_3S^-$ $[M–H]^-$: 271.0434, found 271.0421.

7. Copies of ^{1}H NMR, ^{13}C NMR and ^{19}F NMR

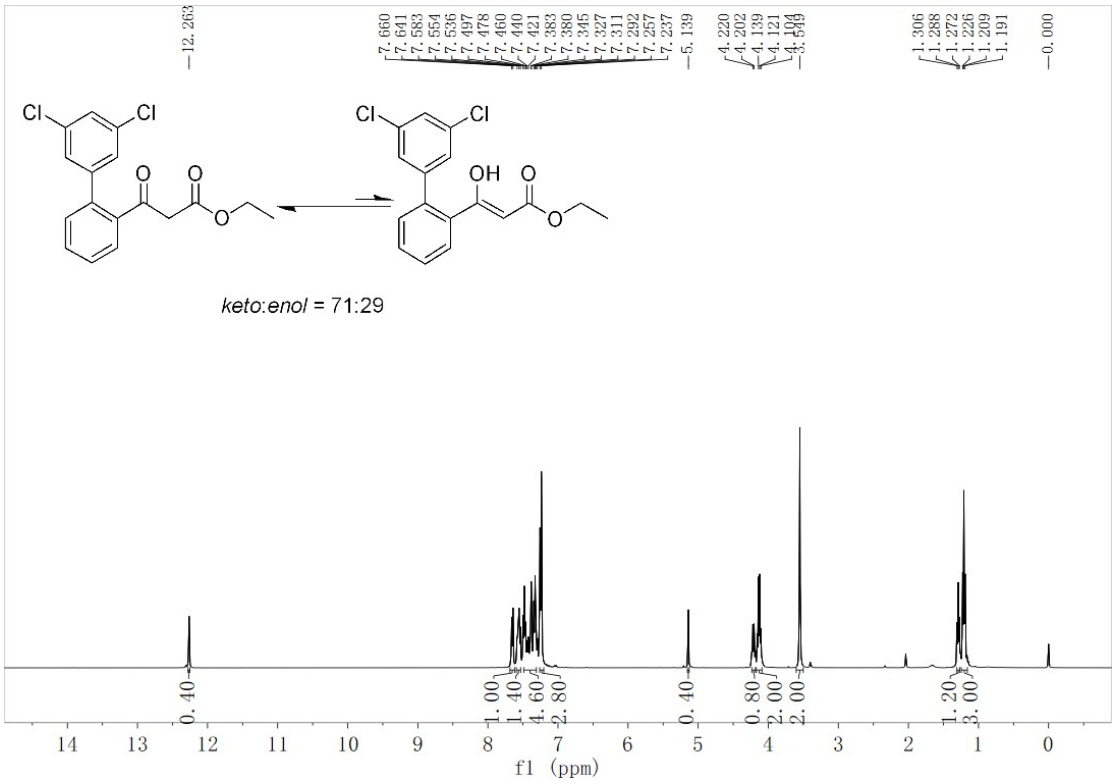
1t – ^{1}H NMR (400 MHz, $CDCl_3$)



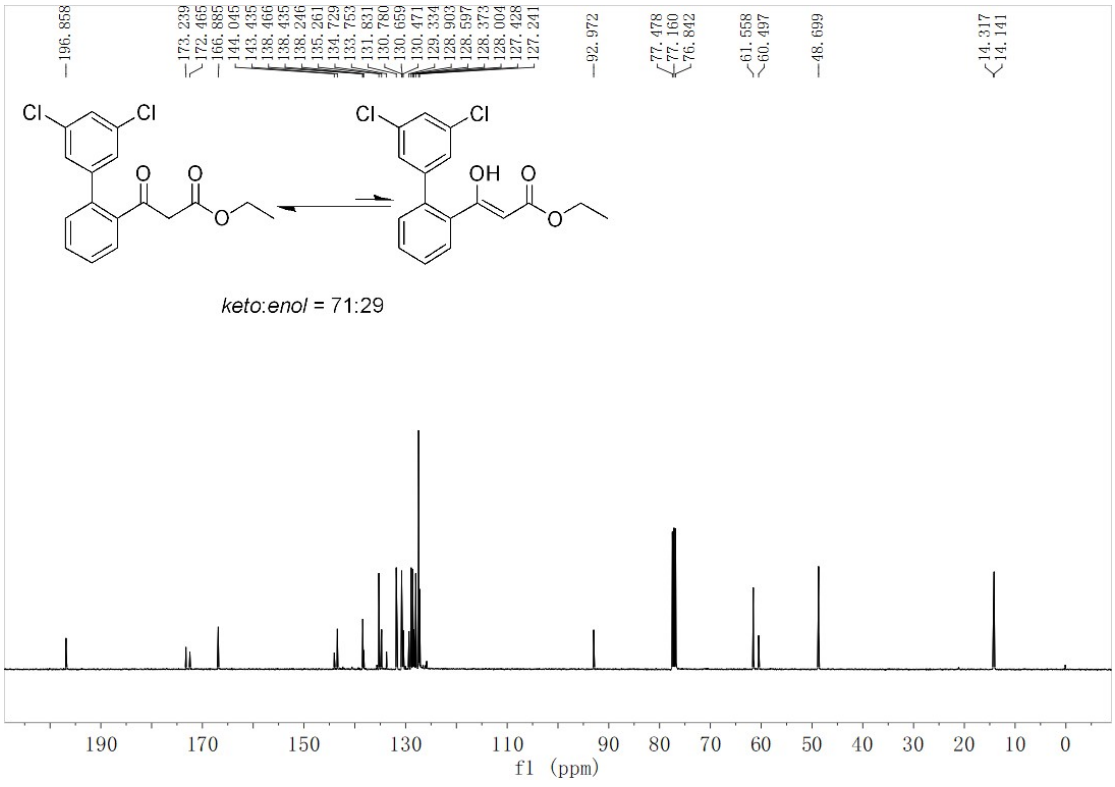
1t – ^{13}C NMR (101 MHz, $CDCl_3$)



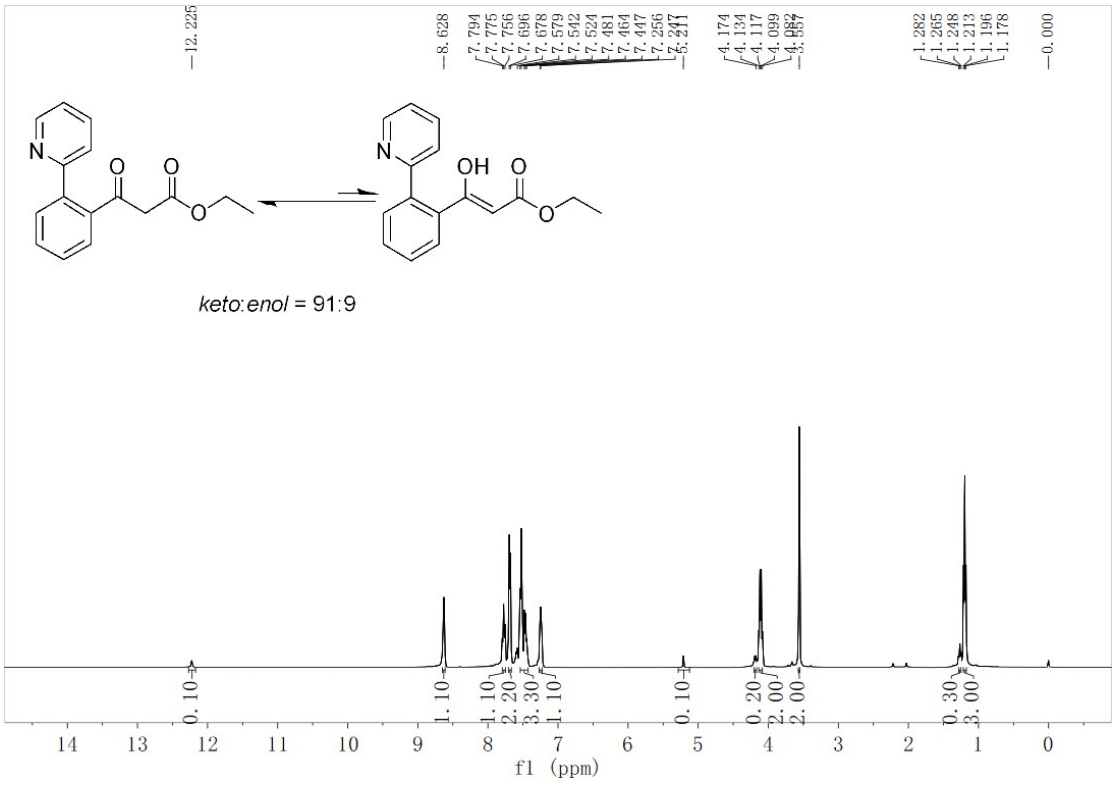
1u – ^{1}H NMR (400 MHz, $CDCl_3$)



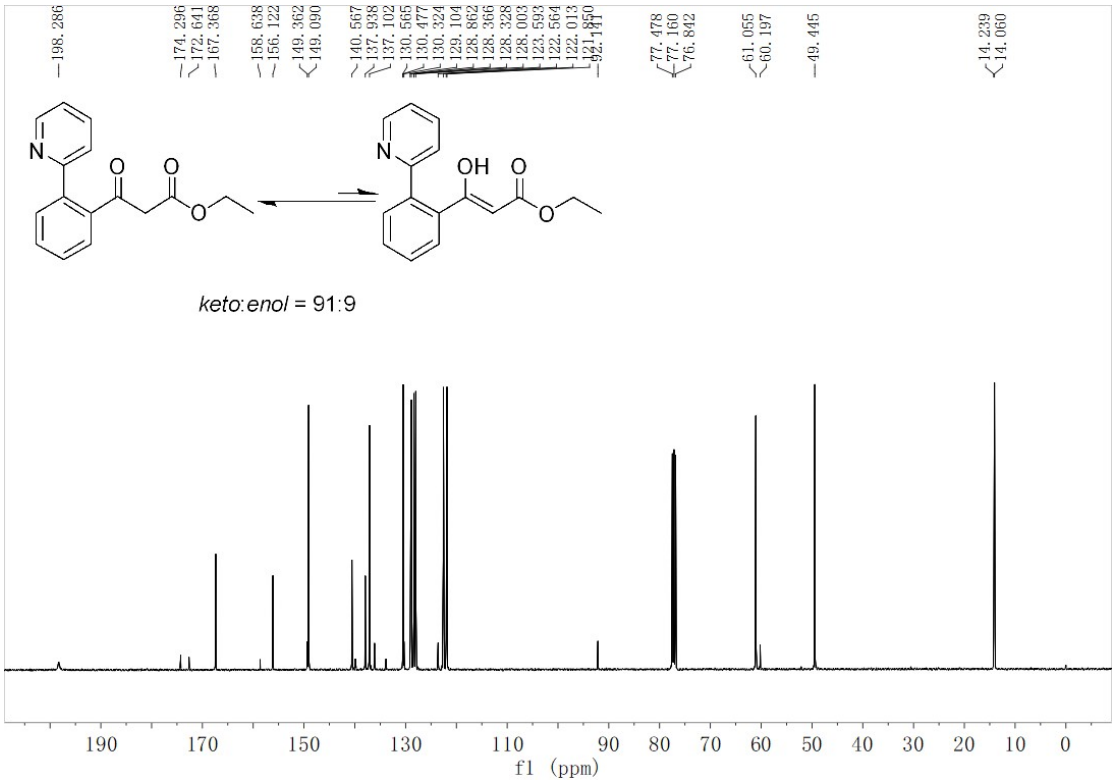
1u – ^{13}C NMR (101 MHz, $CDCl_3$)



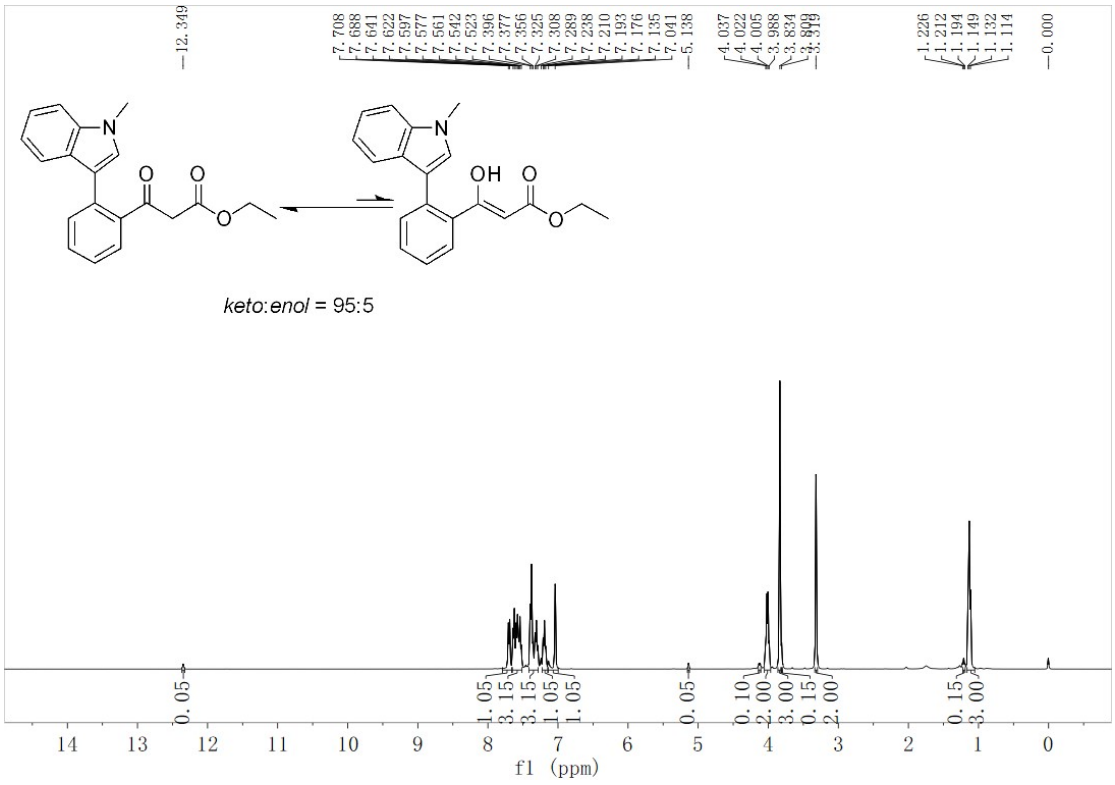
1ag – ^{1}H NMR (400 MHz, $CDCl_3$)



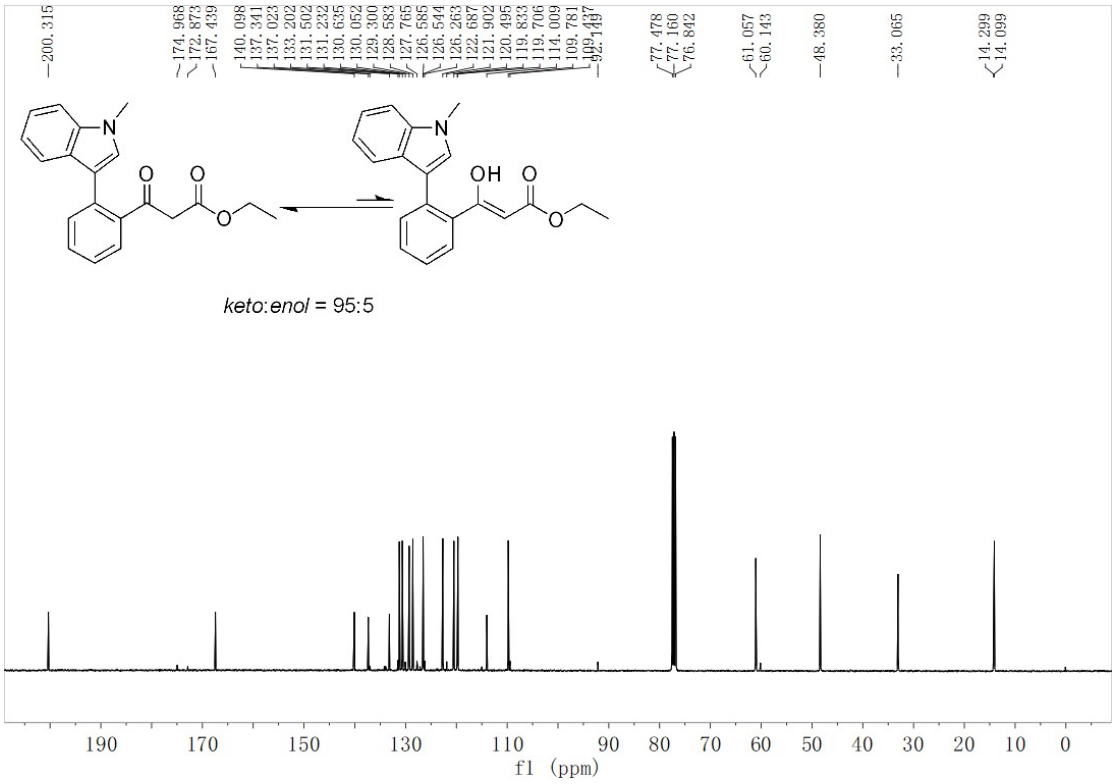
1ag – ^{13}C NMR (101 MHz, $CDCl_3$)



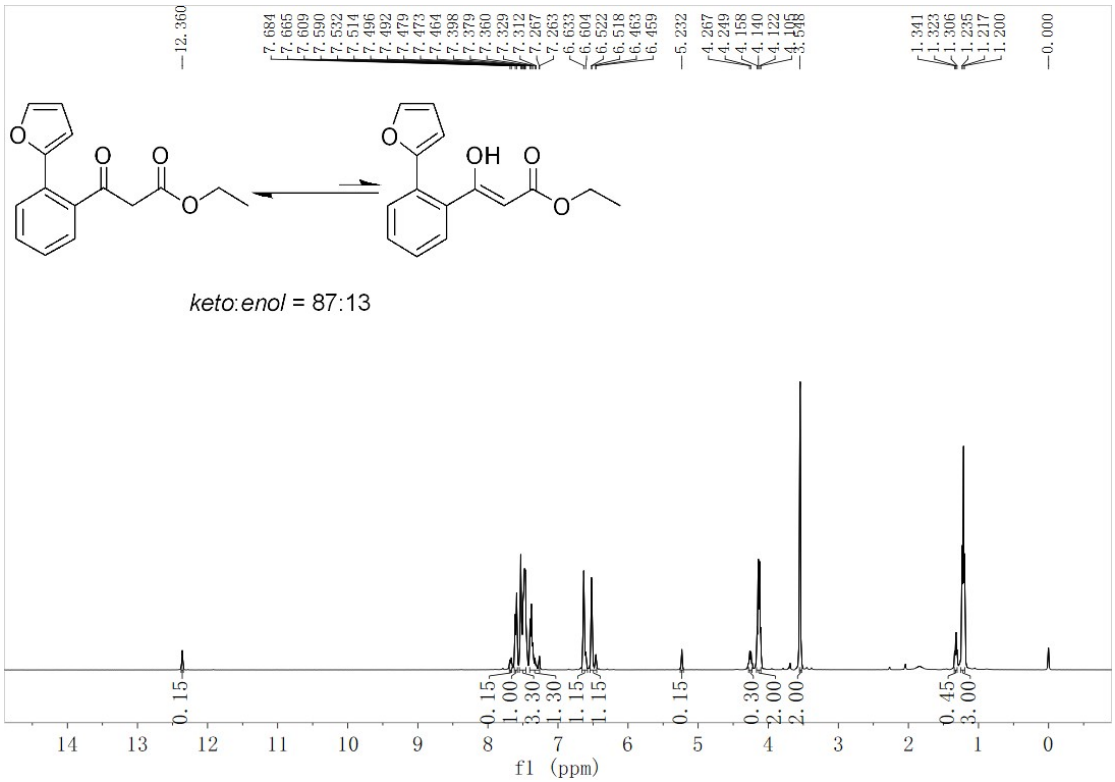
1ah – ^{1}H NMR (400 MHz, $CDCl_3$)



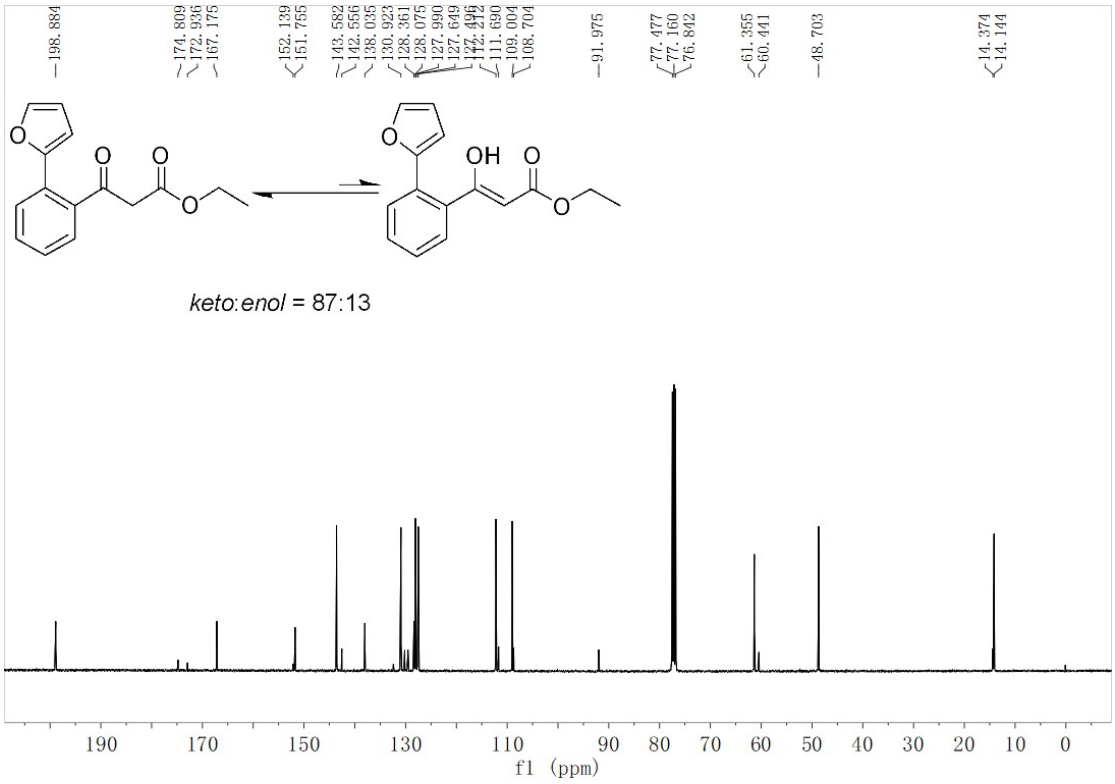
1ah – ^{13}C NMR (101 MHz, $CDCl_3$)



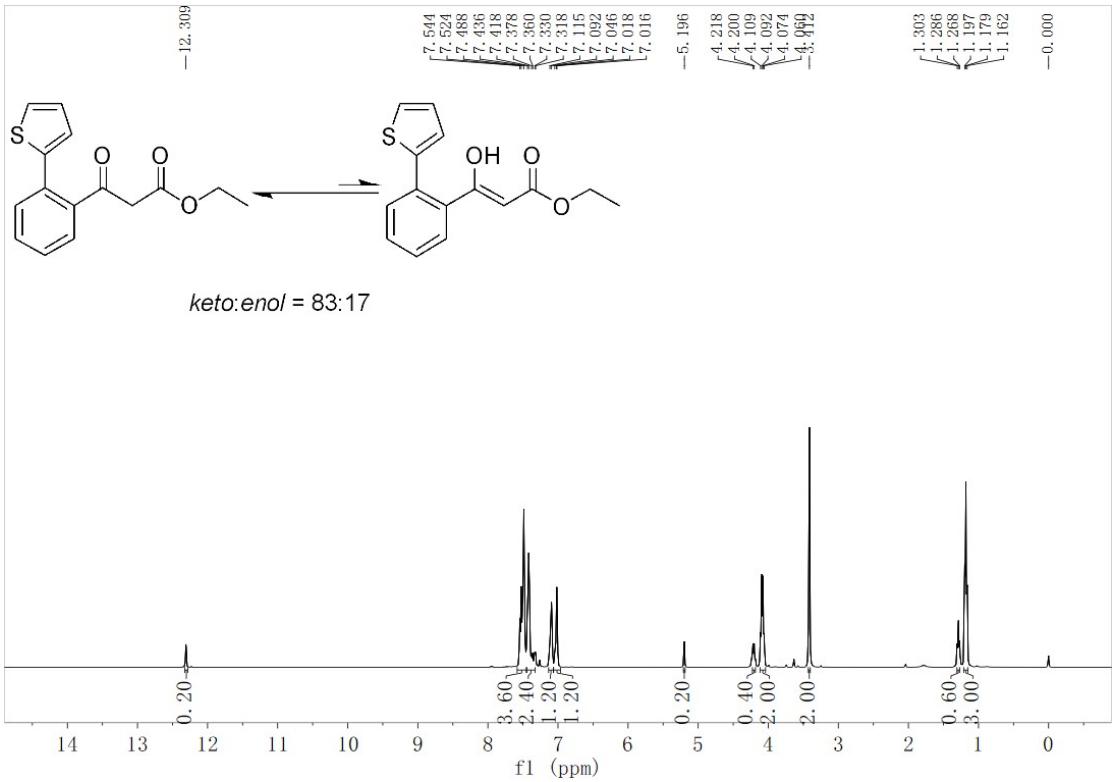
1ai – ^{1}H NMR (400 MHz, $CDCl_3$)



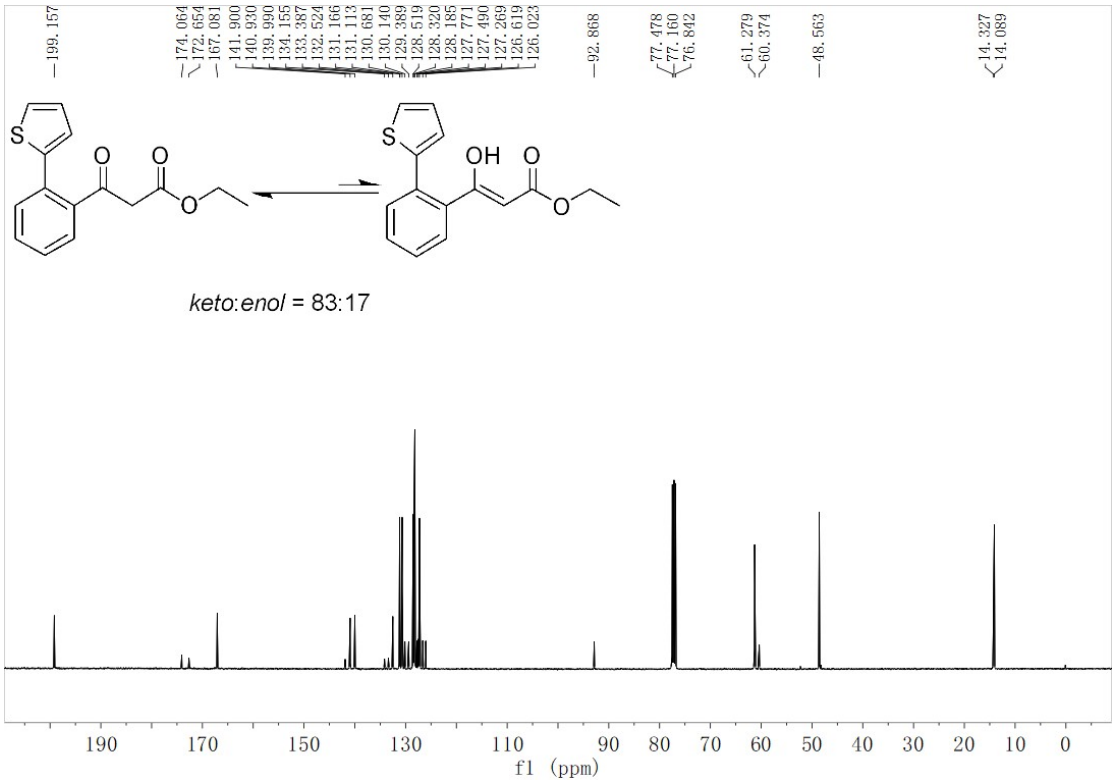
1ai – ^{13}C NMR (101 MHz, $CDCl_3$)



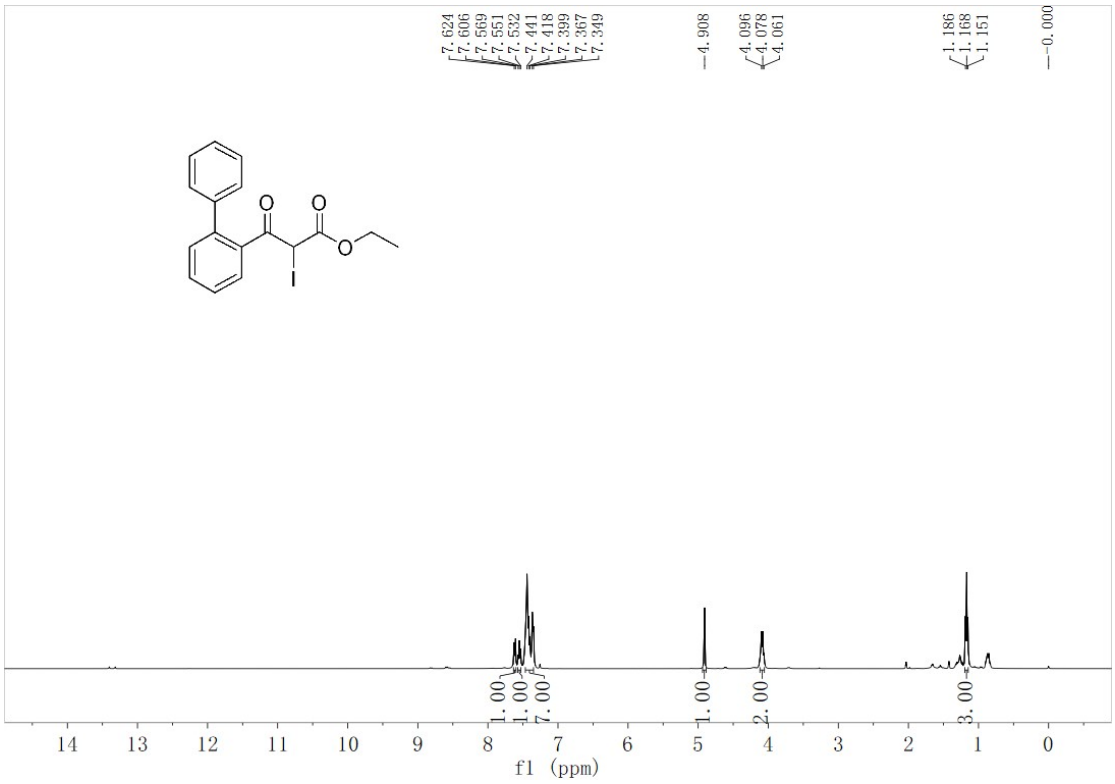
1aj – ^{1}H NMR (400 MHz, $CDCl_3$)



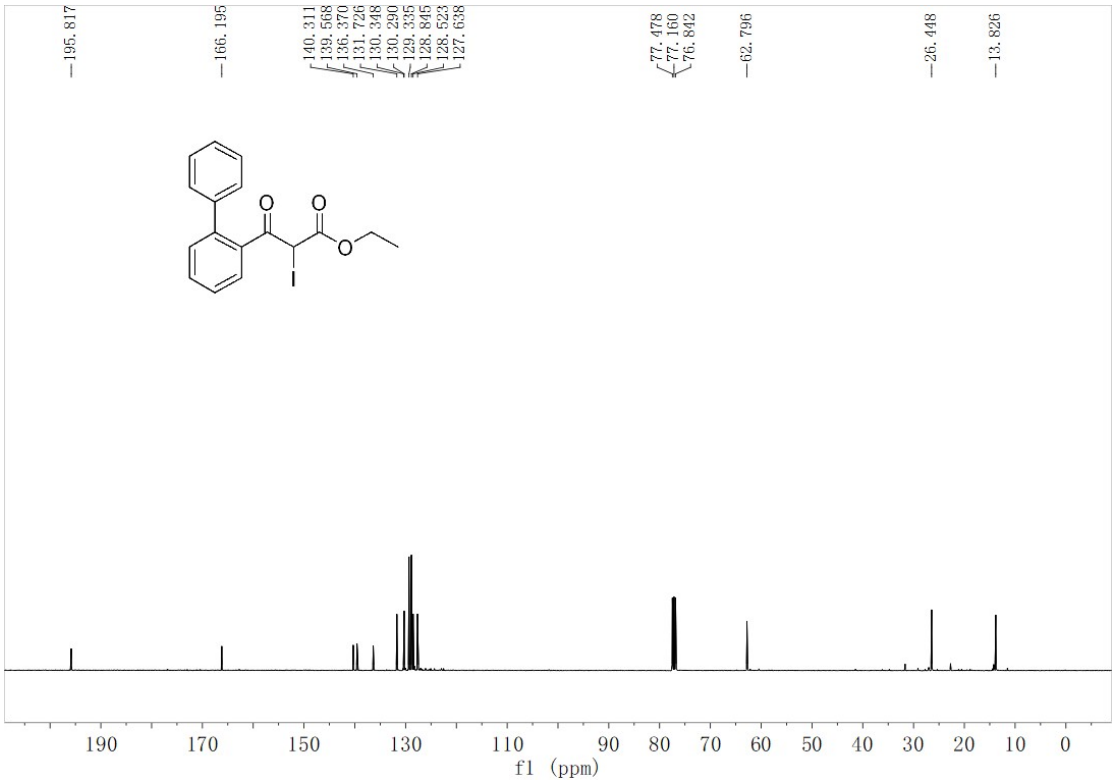
1aj – ^{13}C NMR (101 MHz, $CDCl_3$)



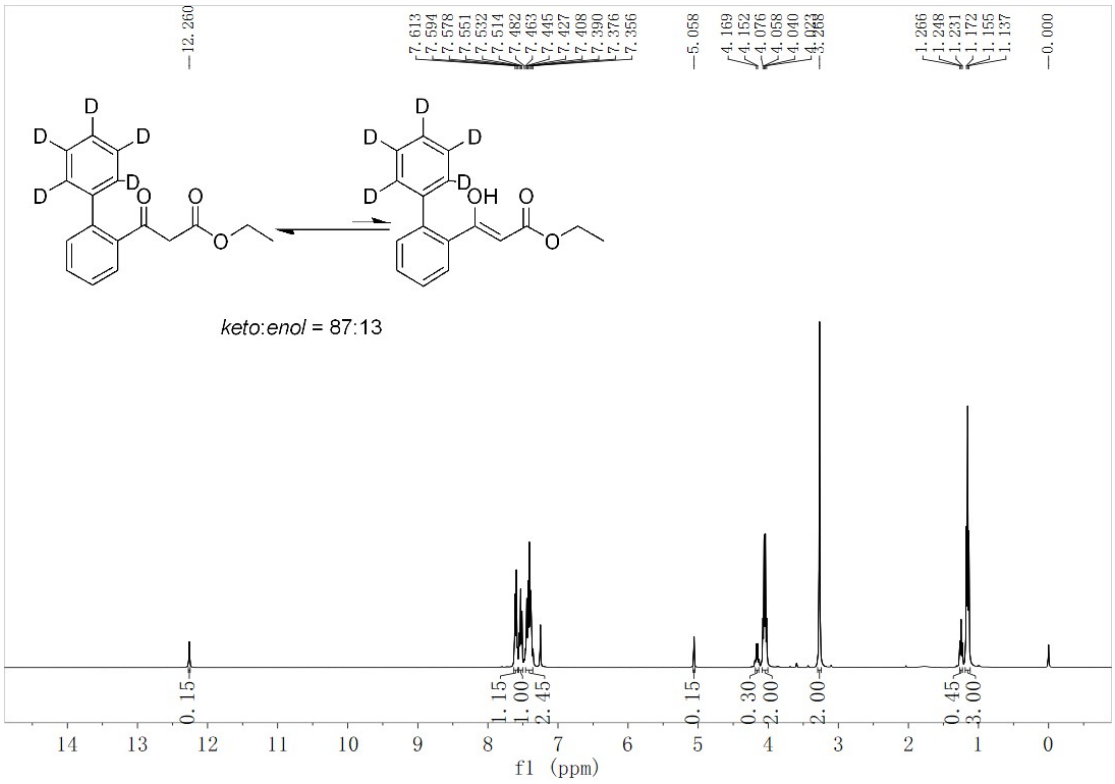
[I]-1a – ^{1}H NMR (400 MHz, $CDCl_3$)



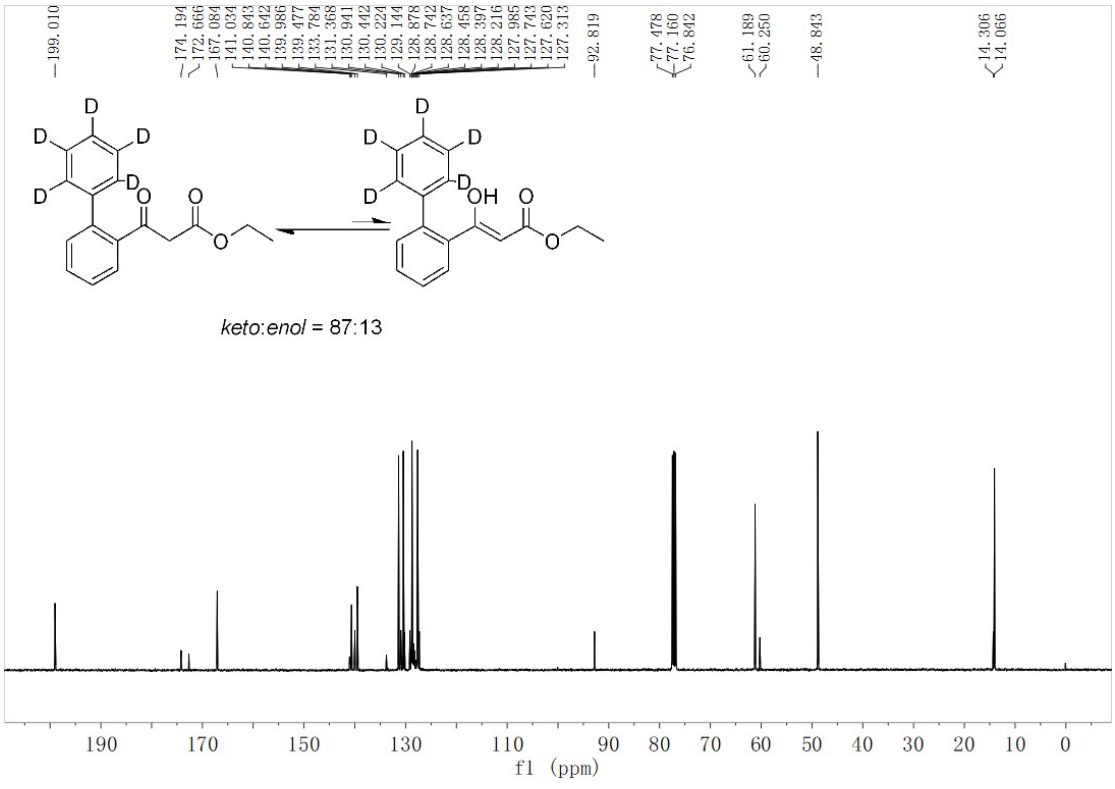
[I]-1a – ^{13}C NMR (101 MHz, $CDCl_3$)



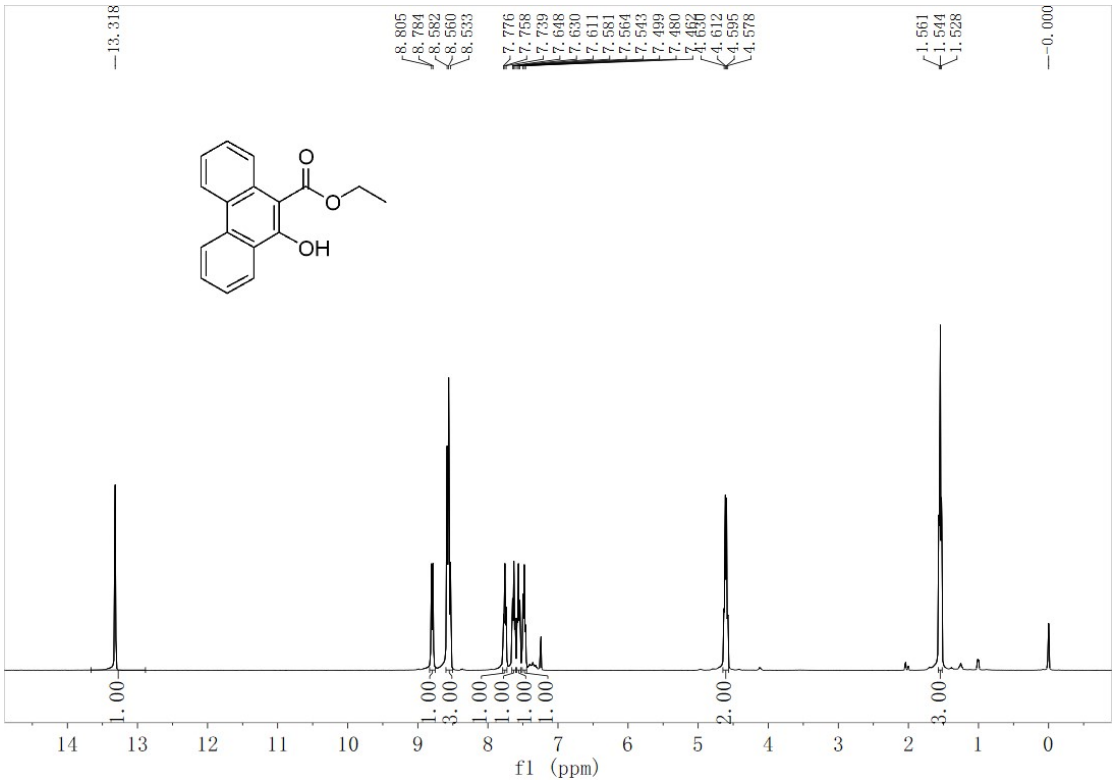
D_5-**1a** – ^{1}H NMR (400 MHz, $CDCl_3$)



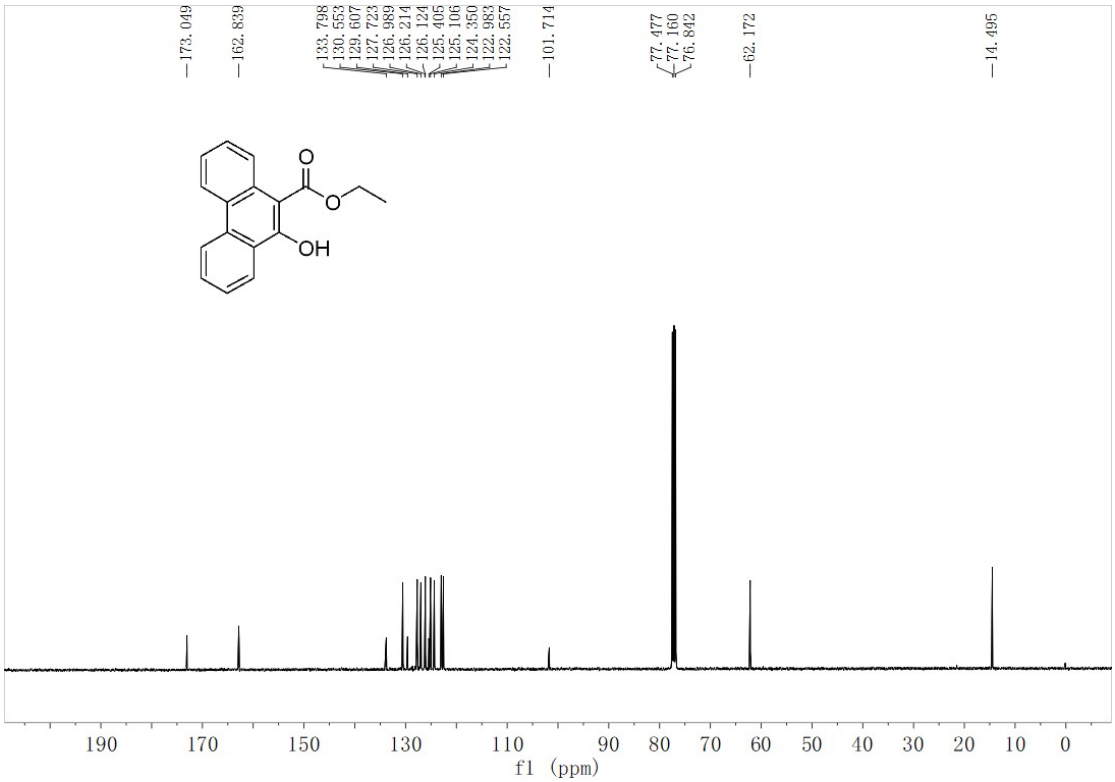
D_5-**1a** – ^{13}C NMR (101 MHz, $CDCl_3$)



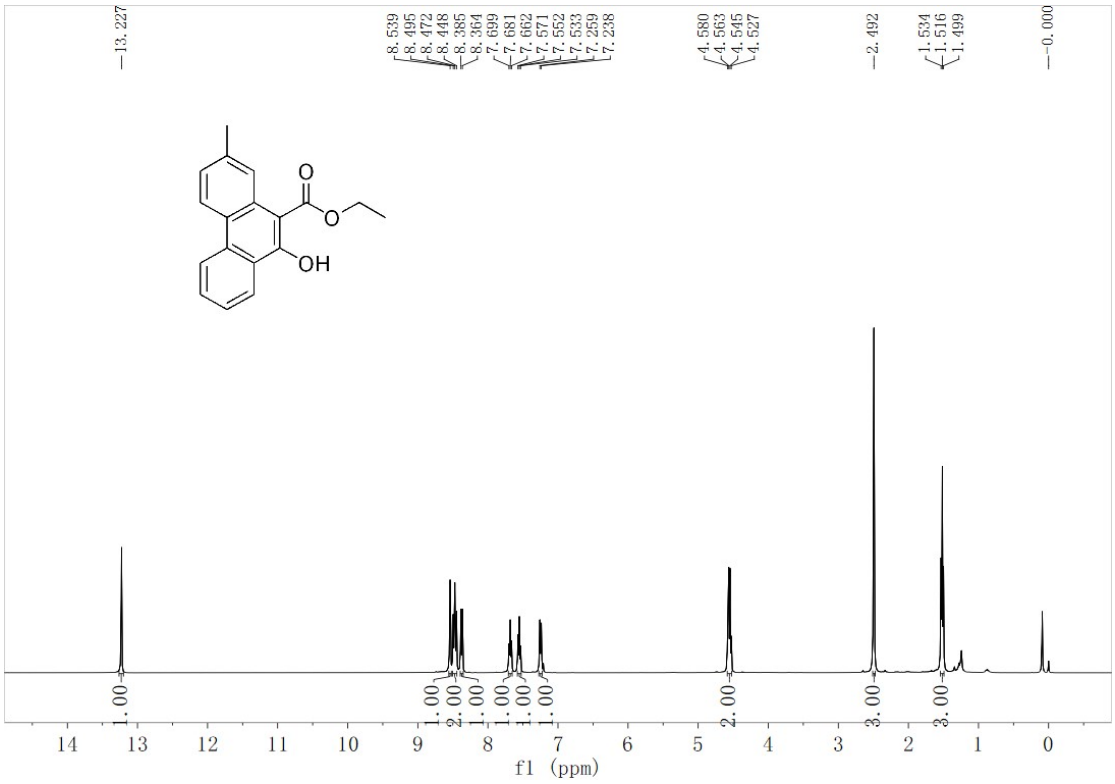
2a – ^{1}H NMR (400 MHz, $CDCl_3$)



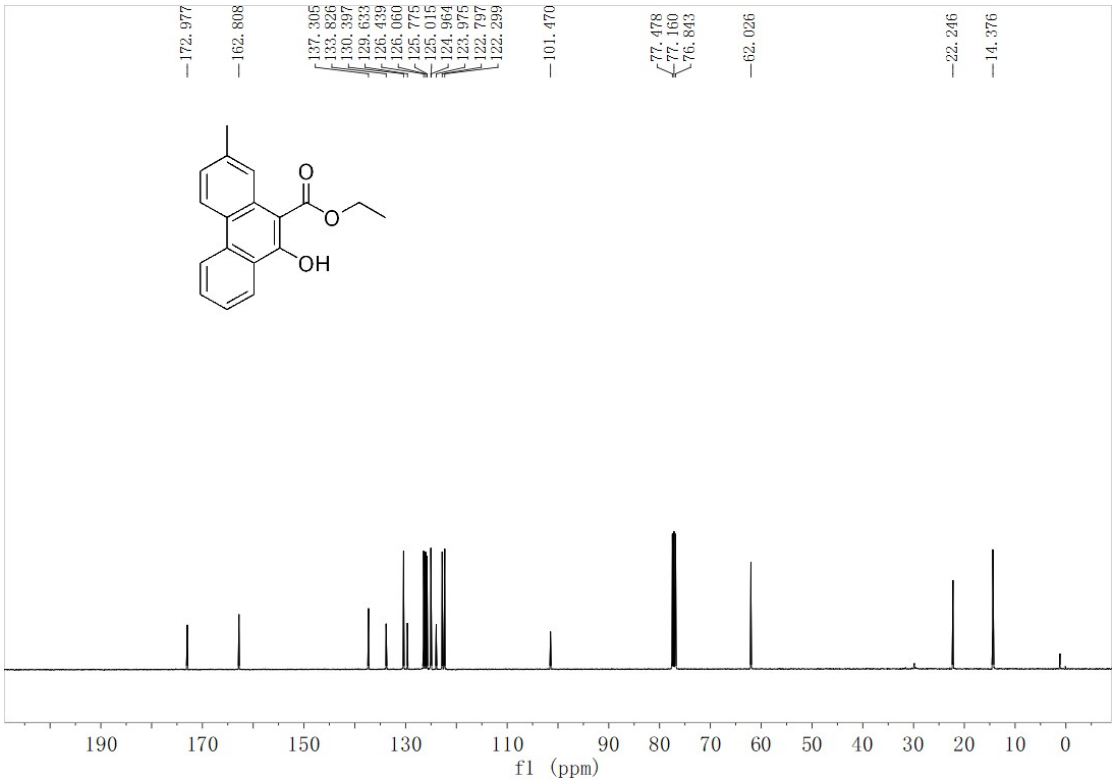
2a – ^{13}C NMR (101 MHz, $CDCl_3$)



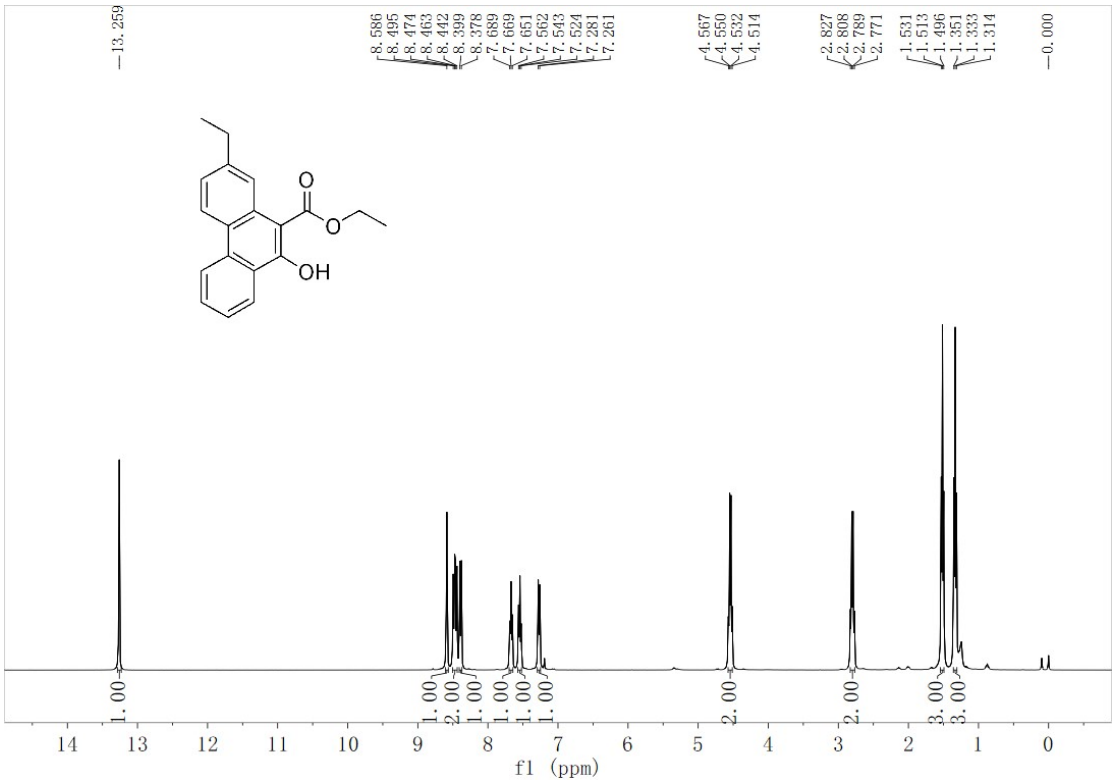
2b – ^{1}H NMR (400 MHz, $CDCl_3$)



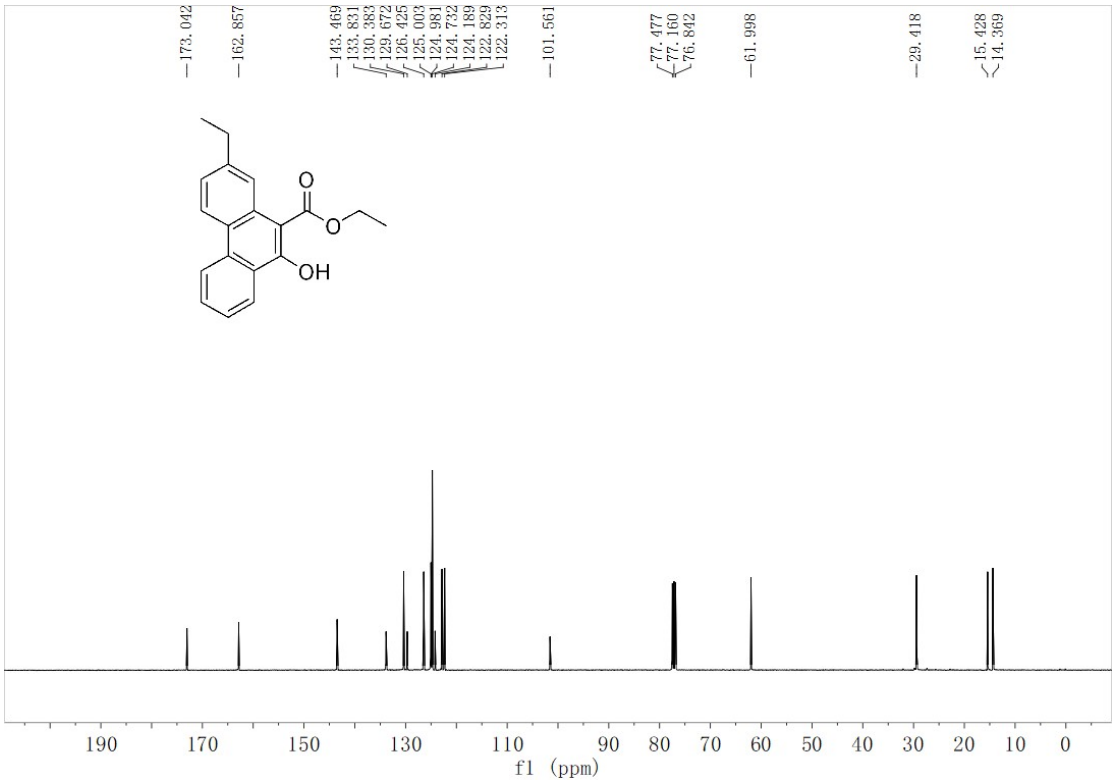
2b – ^{13}C NMR (101 MHz, $CDCl_3$)



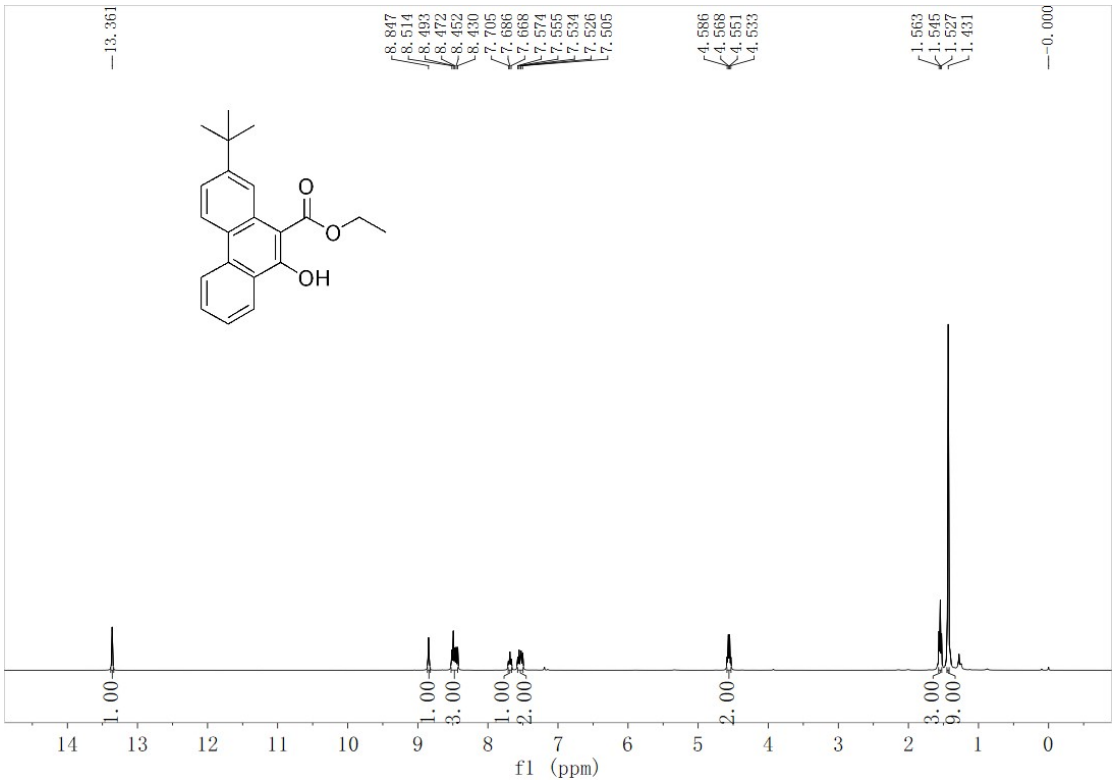
2c – ^{1}H NMR (400 MHz, $CDCl_3$)



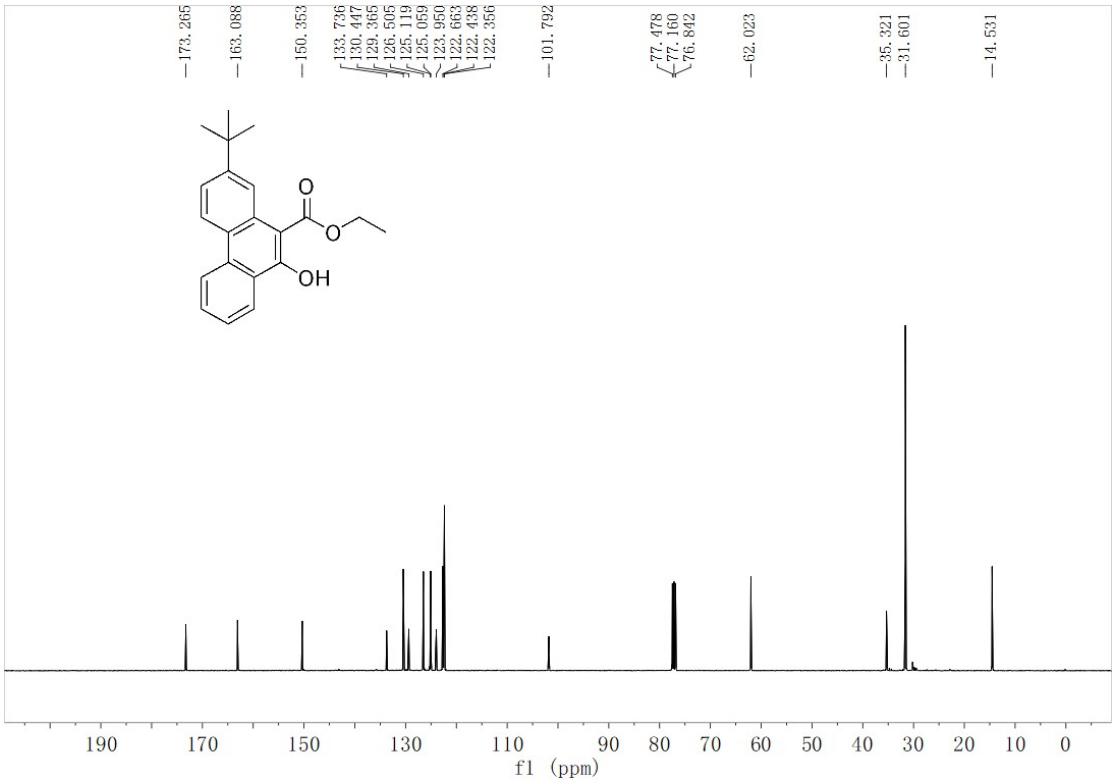
2c – ^{13}C NMR (101 MHz, $CDCl_3$)



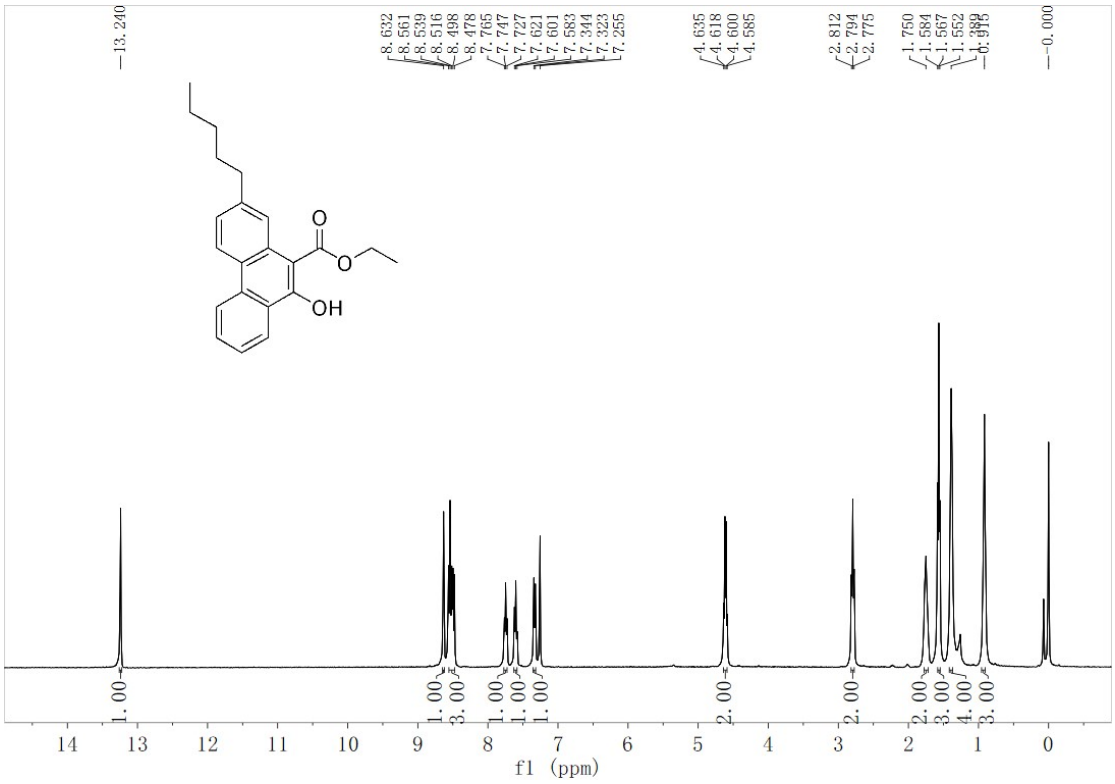
2d – ^{1}H NMR (400 MHz, $CDCl_3$)



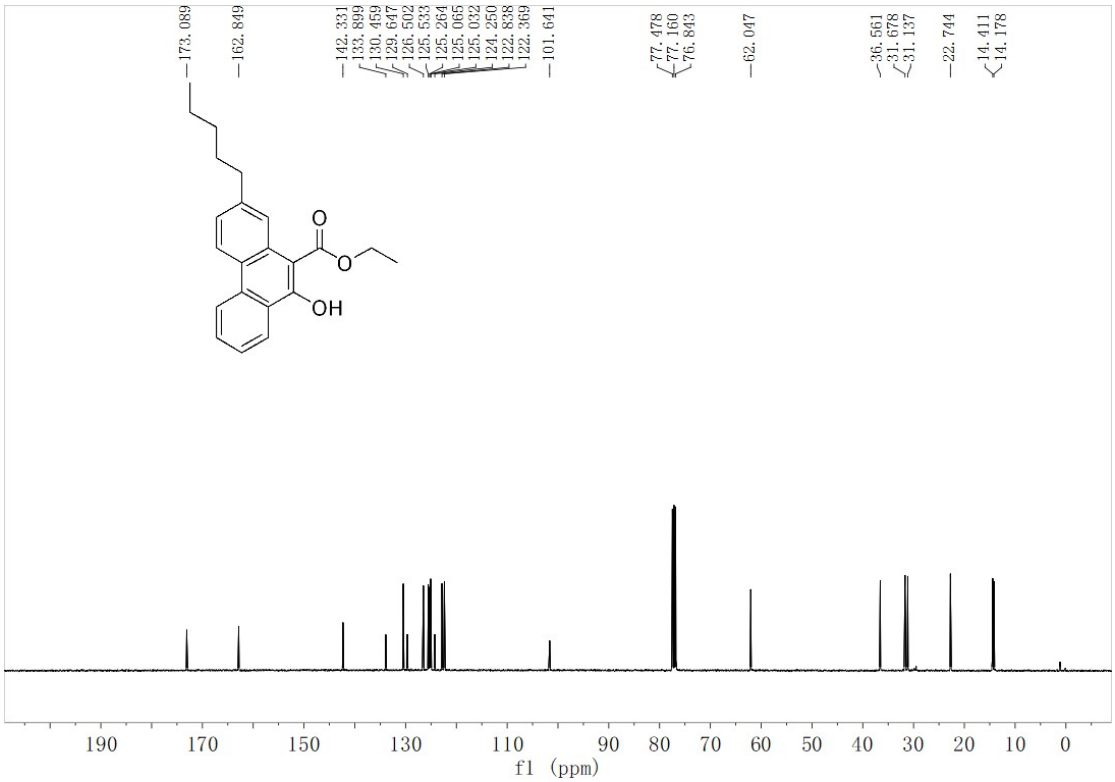
2d – ^{13}C NMR (101 MHz, $CDCl_3$)



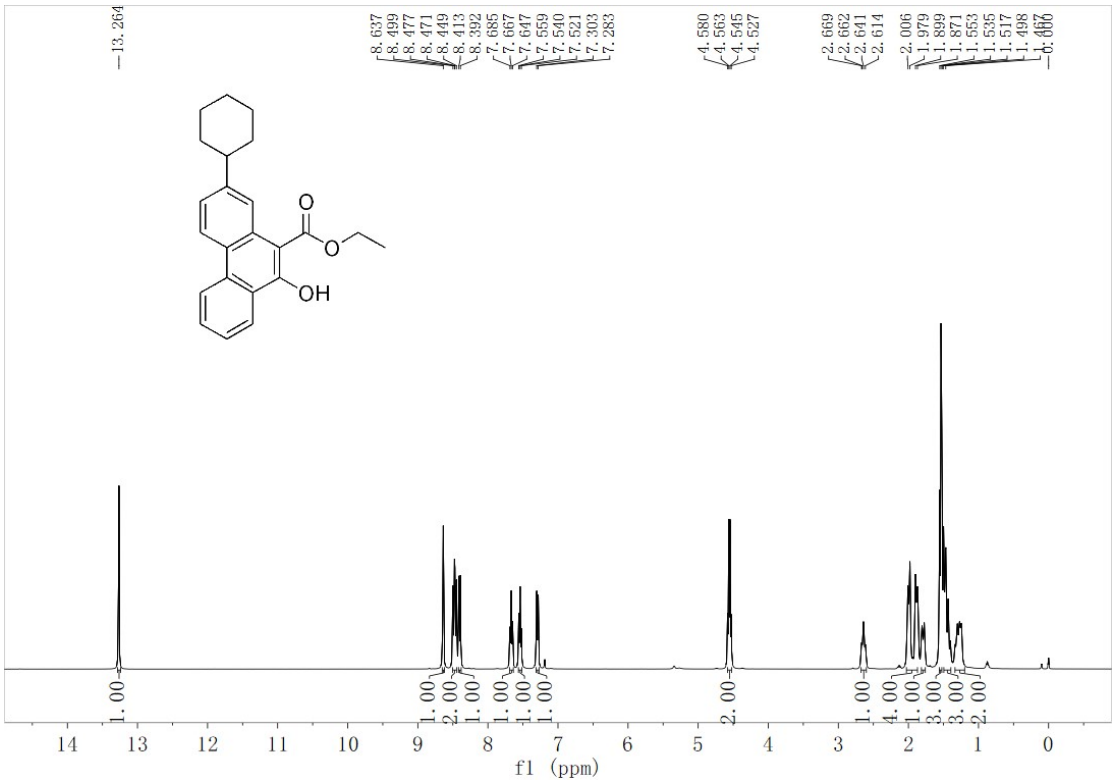
2e – ^{1}H NMR (400 MHz, $CDCl_3$)



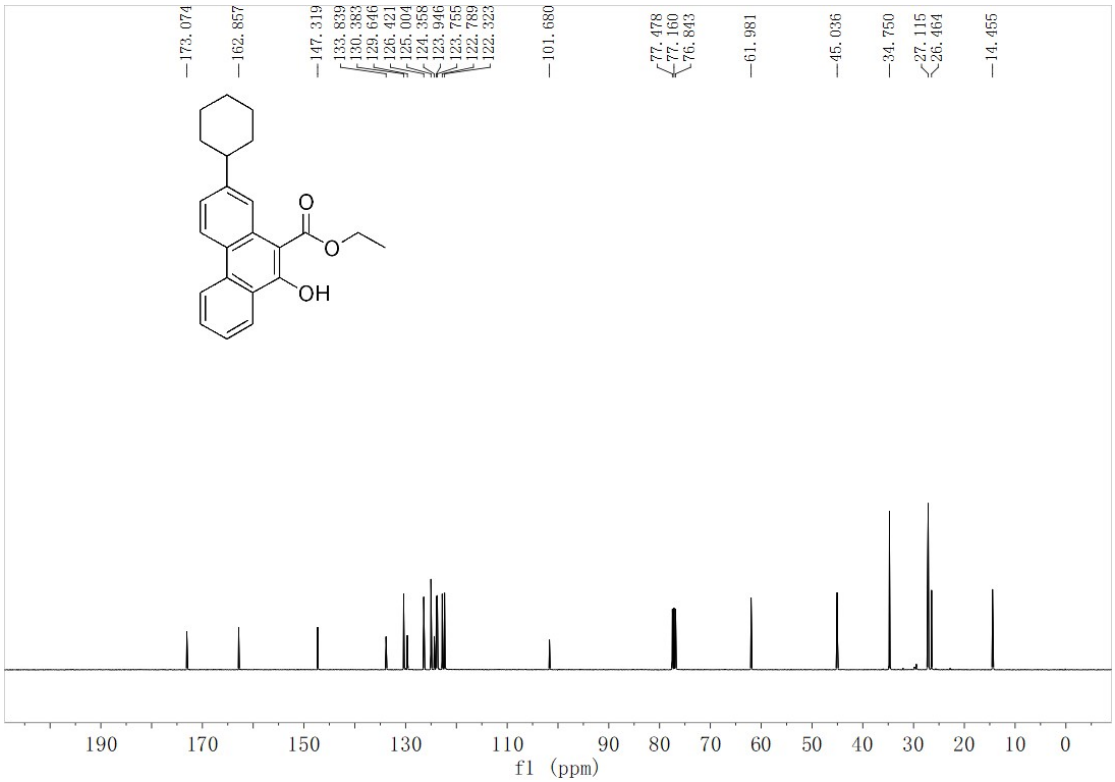
2e – ^{13}C NMR (101 MHz, $CDCl_3$)



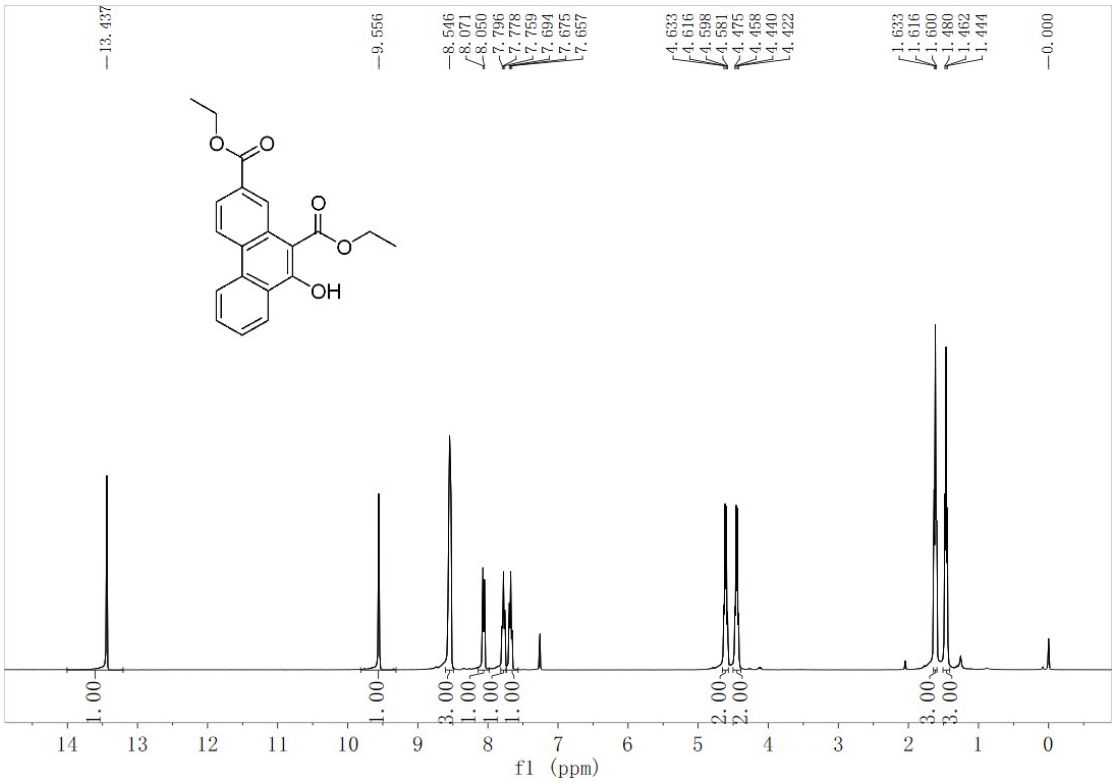
2f – ^{1}H NMR (400 MHz, $CDCl_3$)



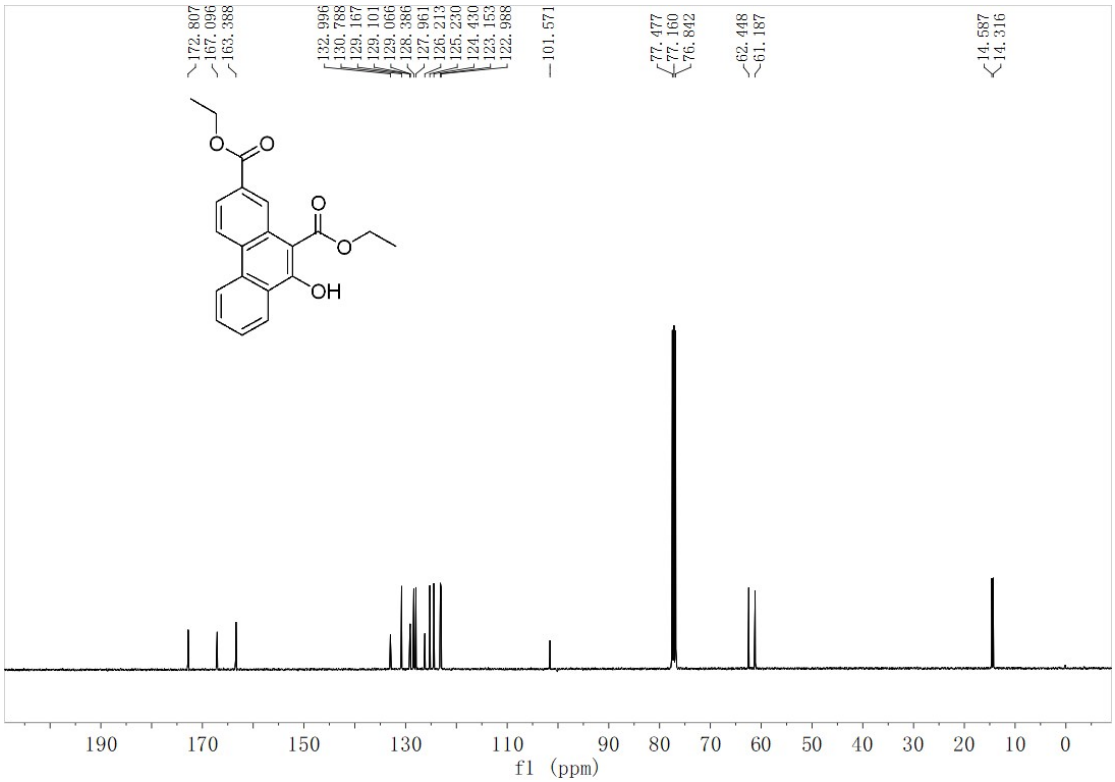
2f – ^{13}C NMR (101 MHz, $CDCl_3$)



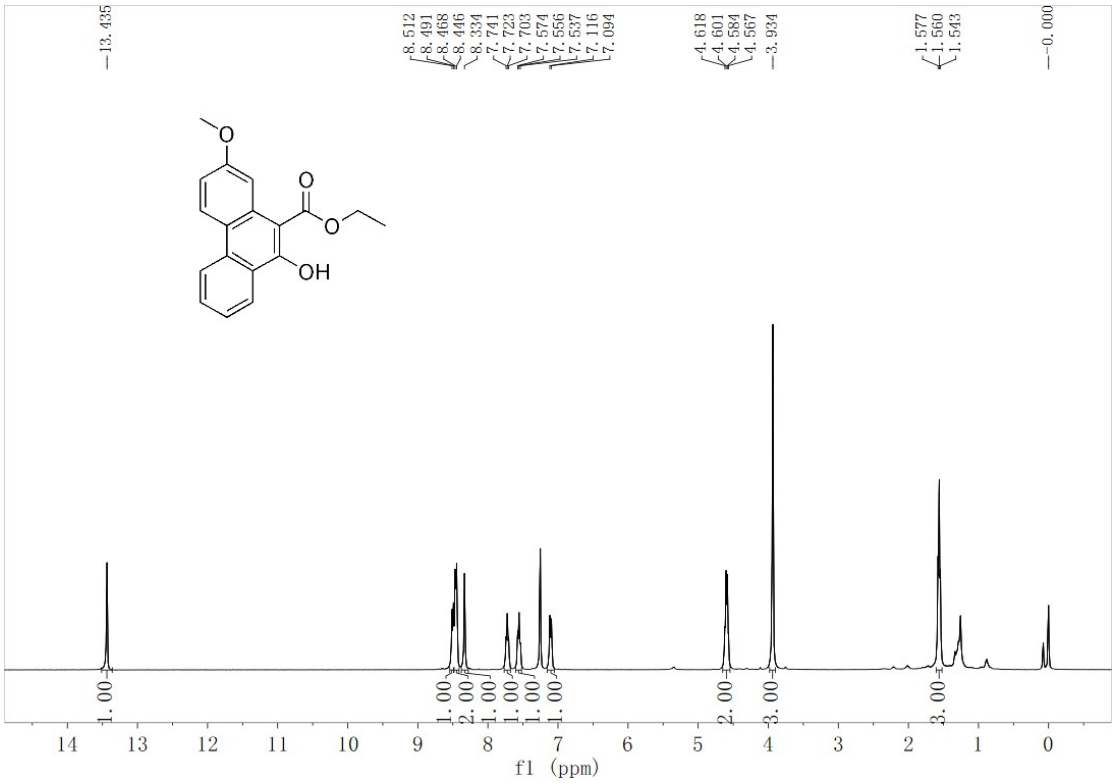
2g – ^{1}H NMR (400 MHz, $CDCl_3$)



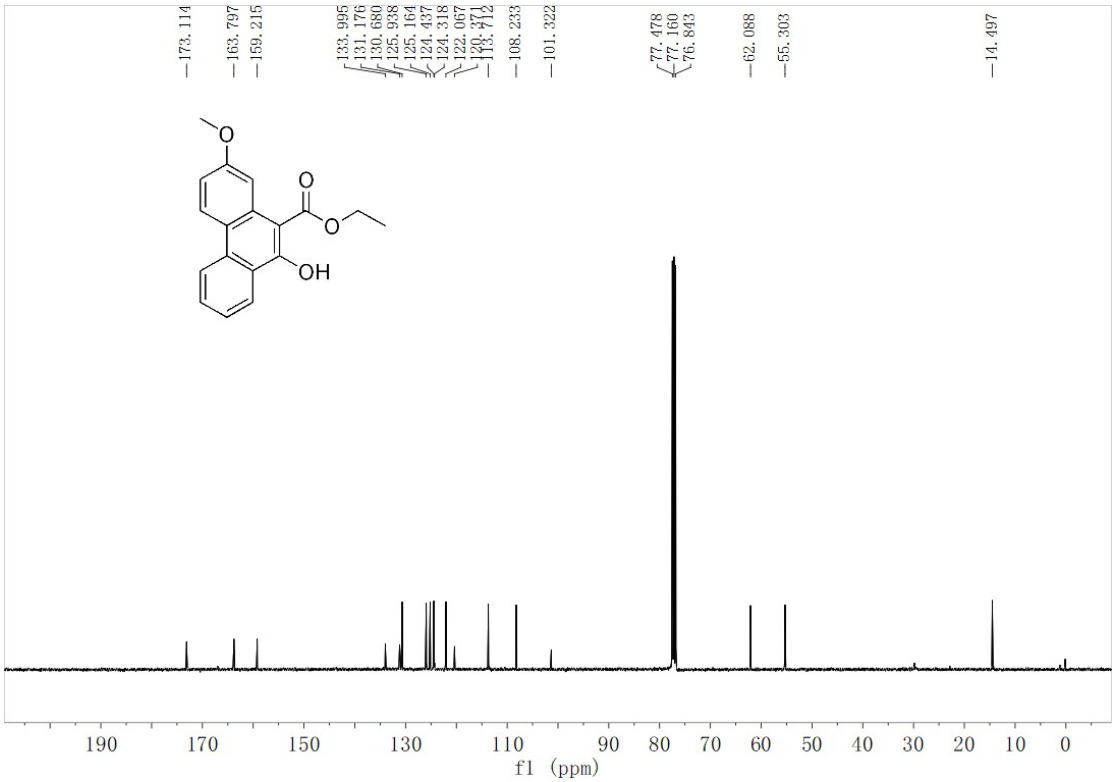
2g – ^{13}C NMR (101 MHz, $CDCl_3$)



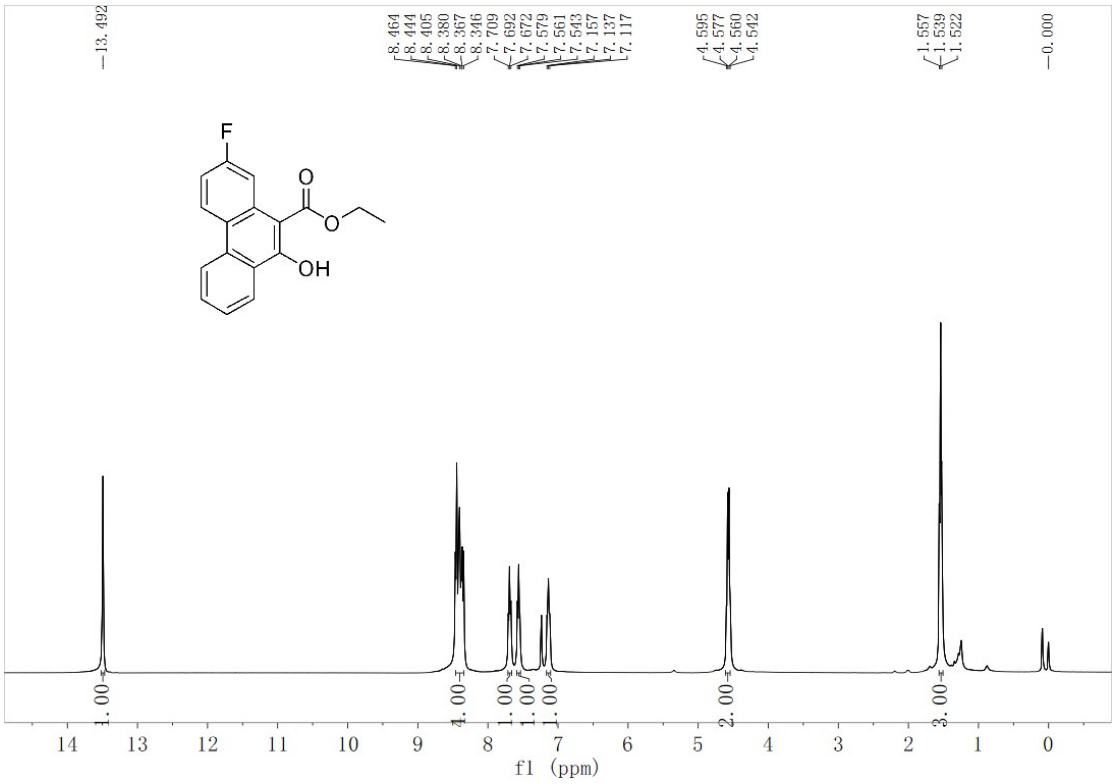
2h – ^{1}H NMR (400 MHz, $CDCl_3$)



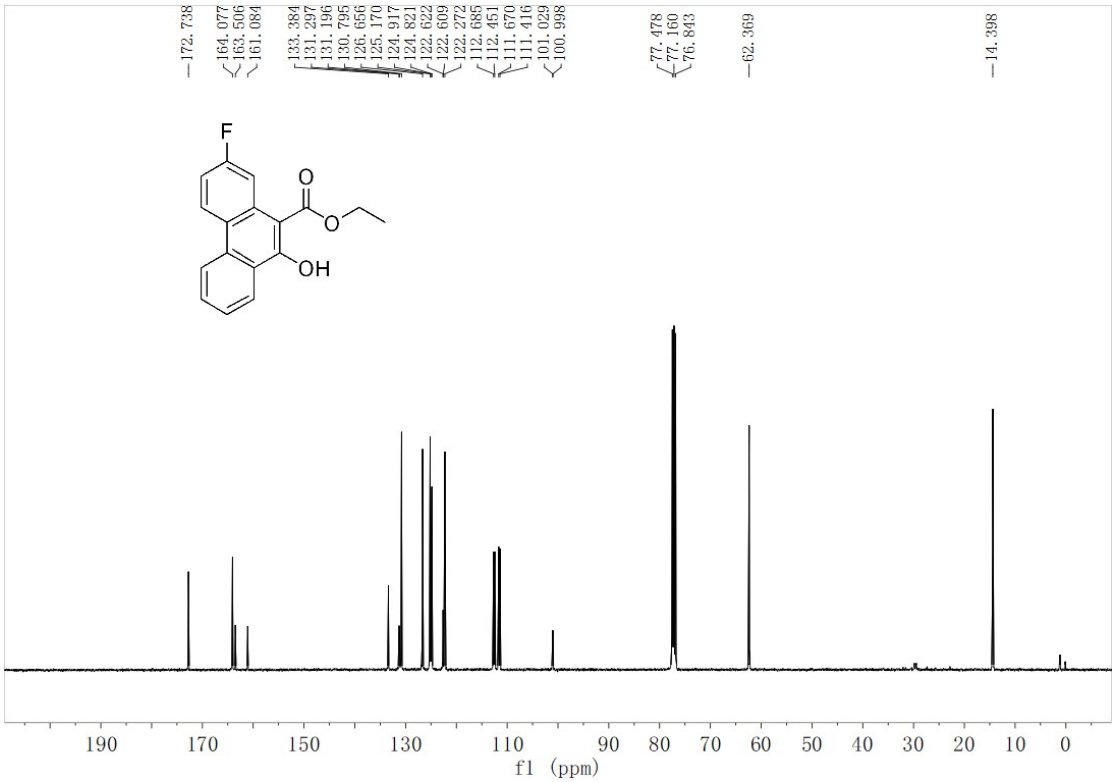
2h – ^{13}C NMR (101 MHz, $CDCl_3$)



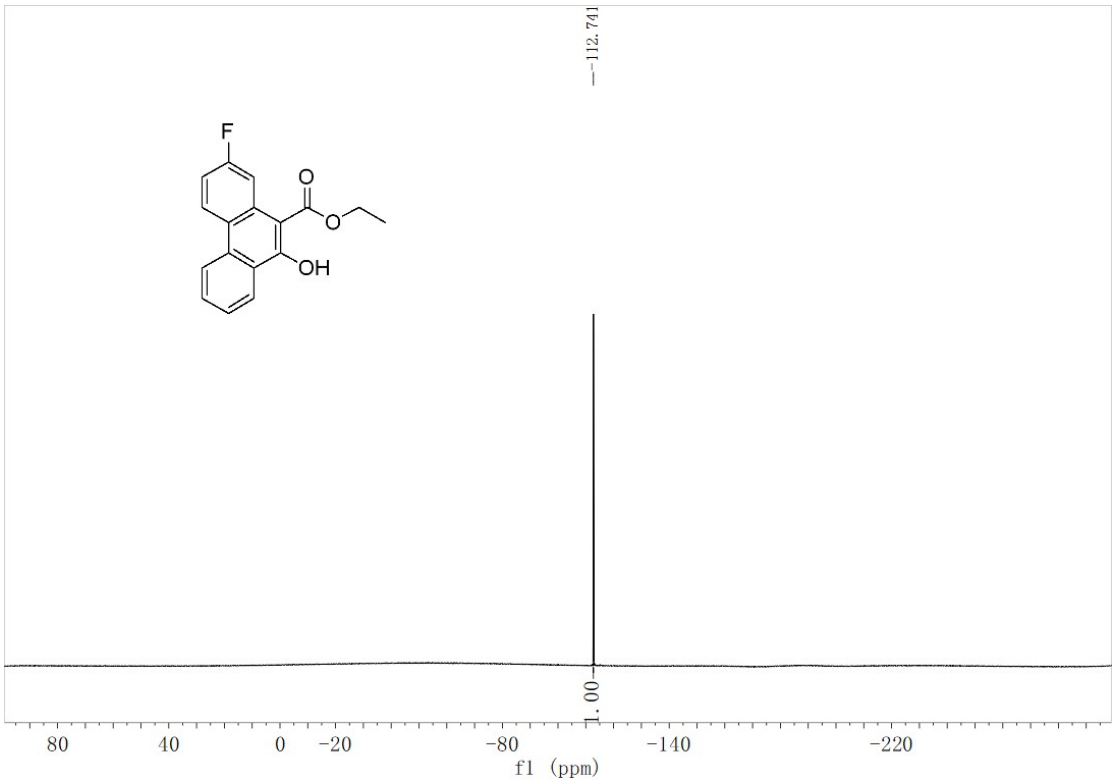
2i – ^{1}H NMR (400 MHz, $CDCl_3$)



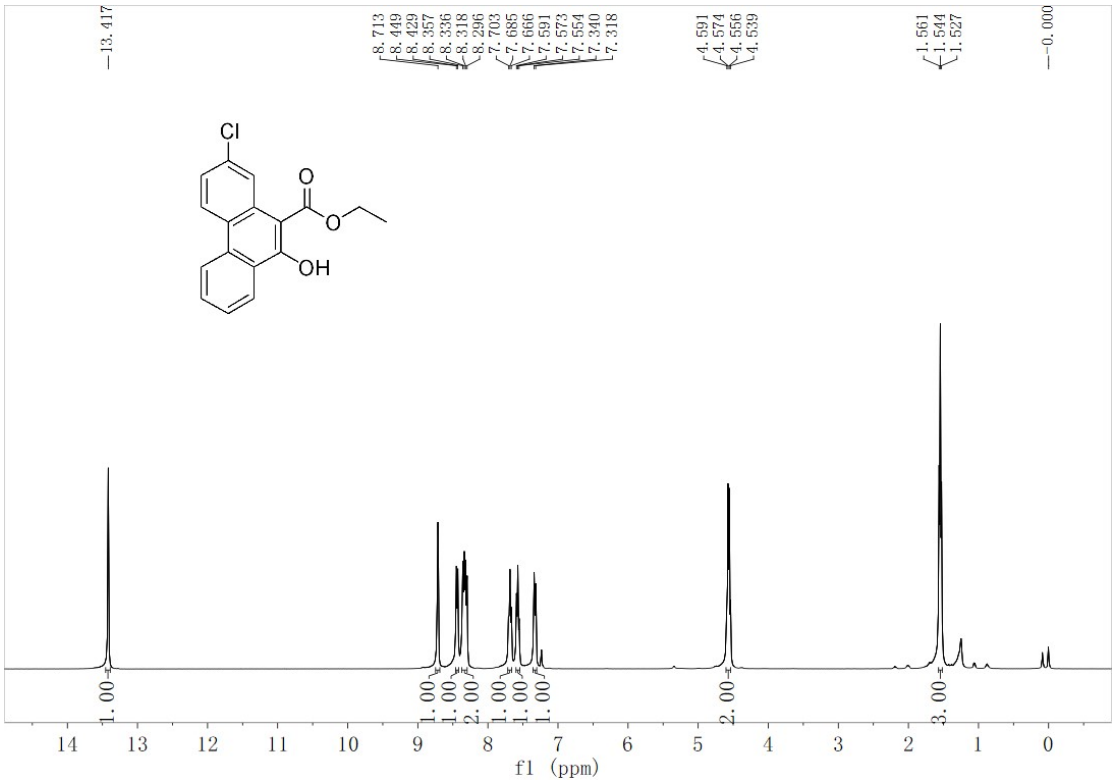
2i – ^{13}C NMR (101 MHz, $CDCl_3$)



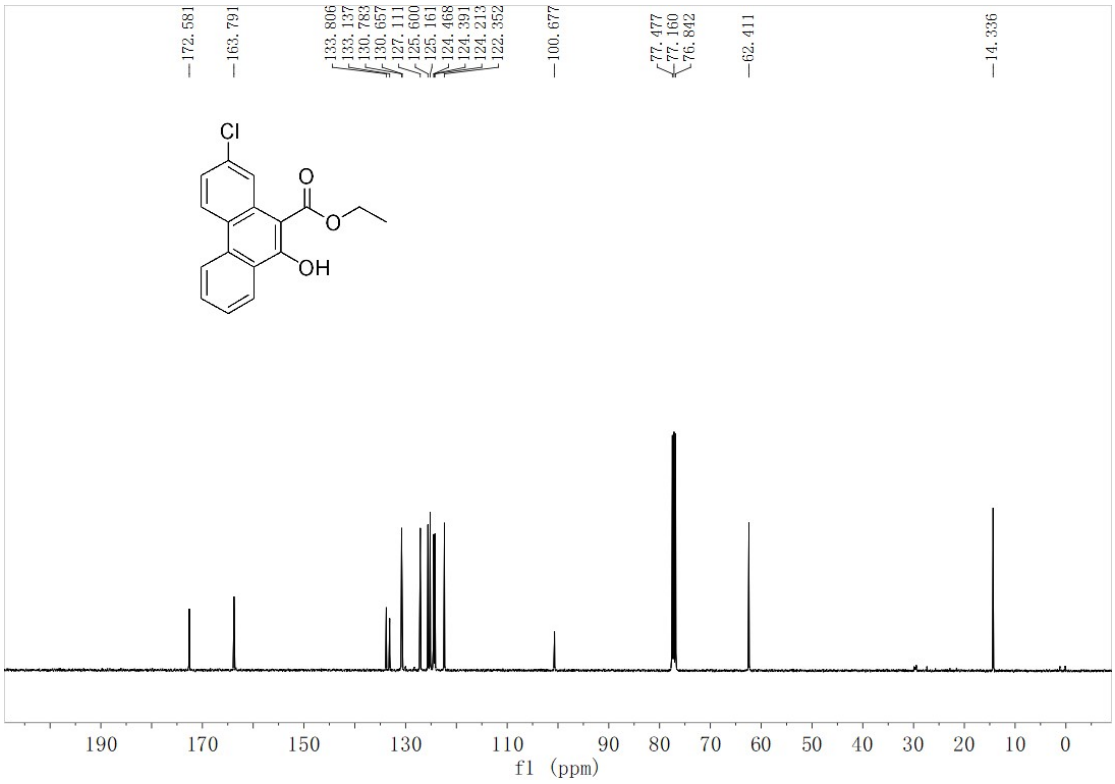
2i – ^{19}F NMR (377 MHz, $CDCl_3$)



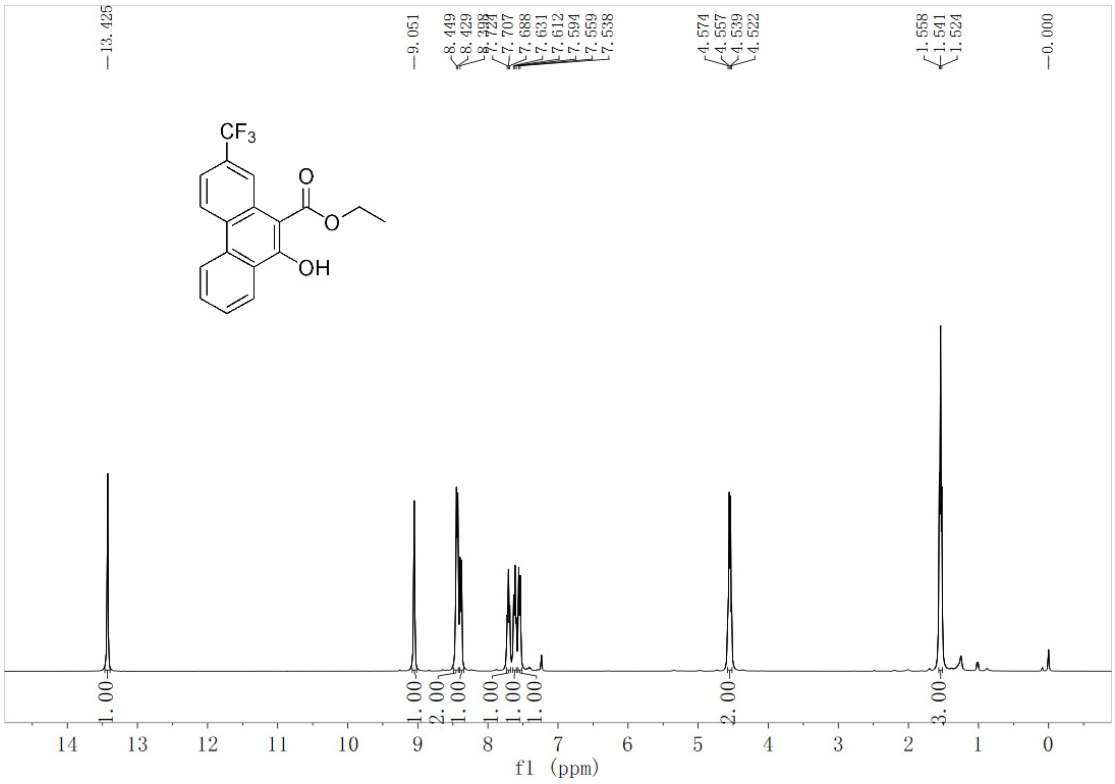
2j – ^{1}H NMR (400 MHz, $CDCl_3$)



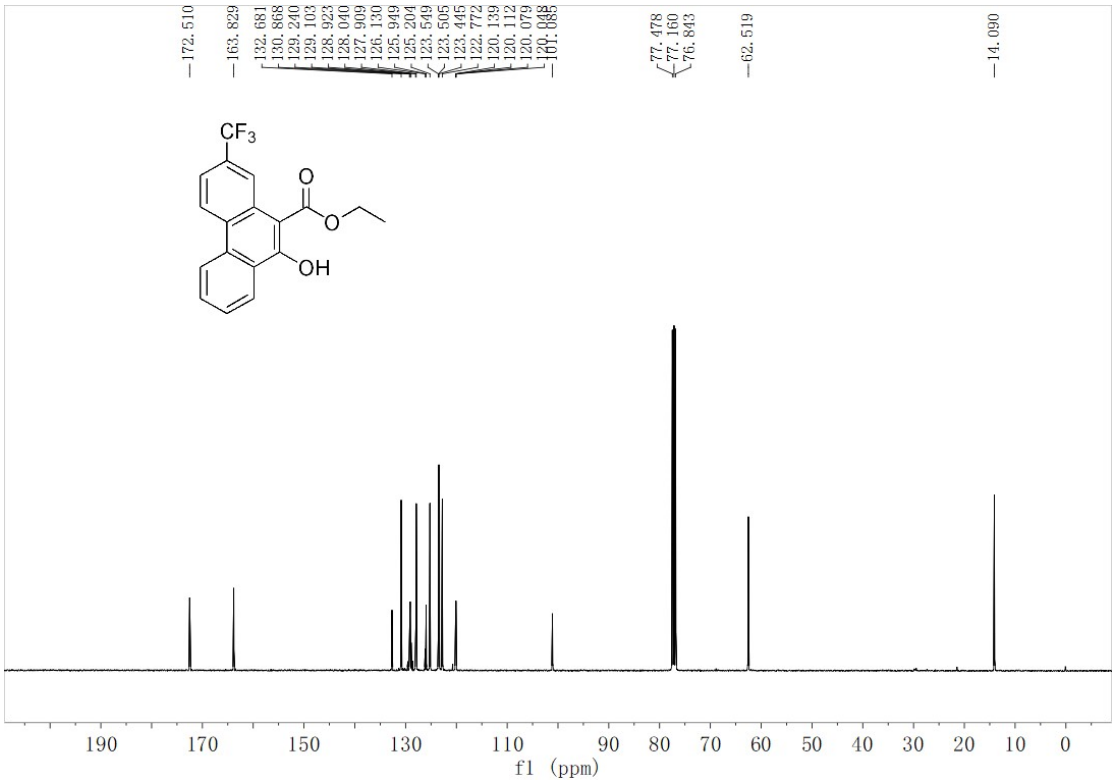
2j – ^{13}C NMR (101 MHz, $CDCl_3$)



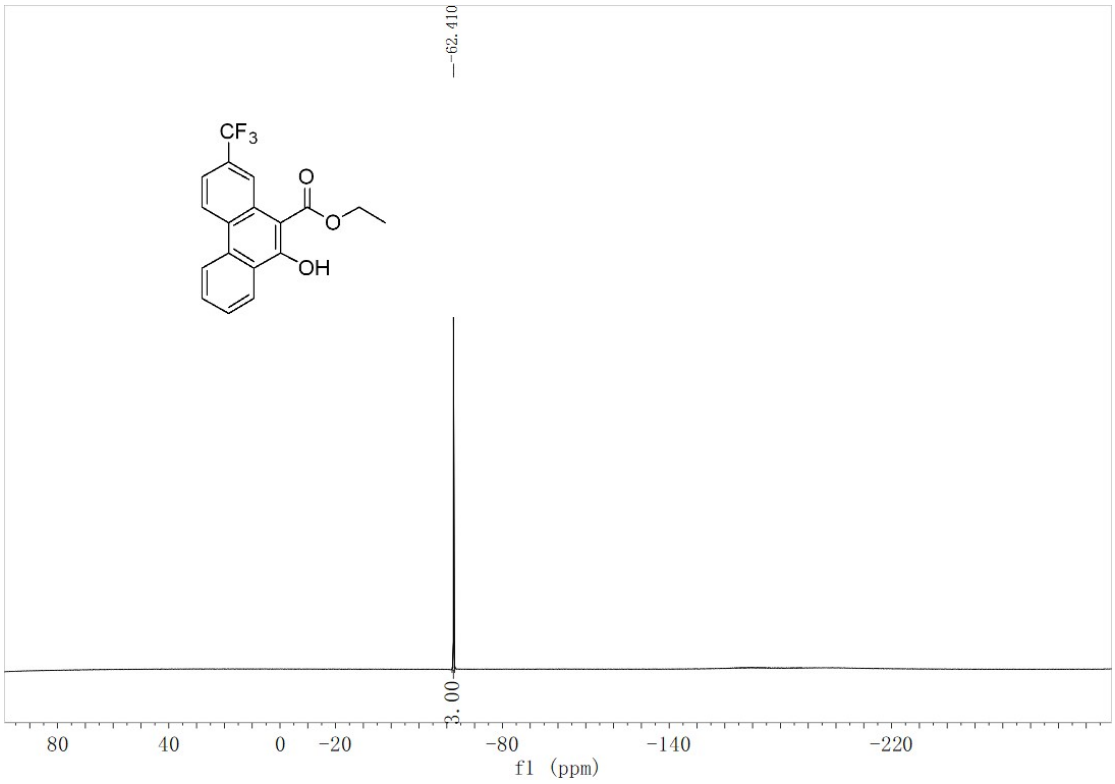
2k – ^{1}H NMR (400 MHz, $CDCl_3$)



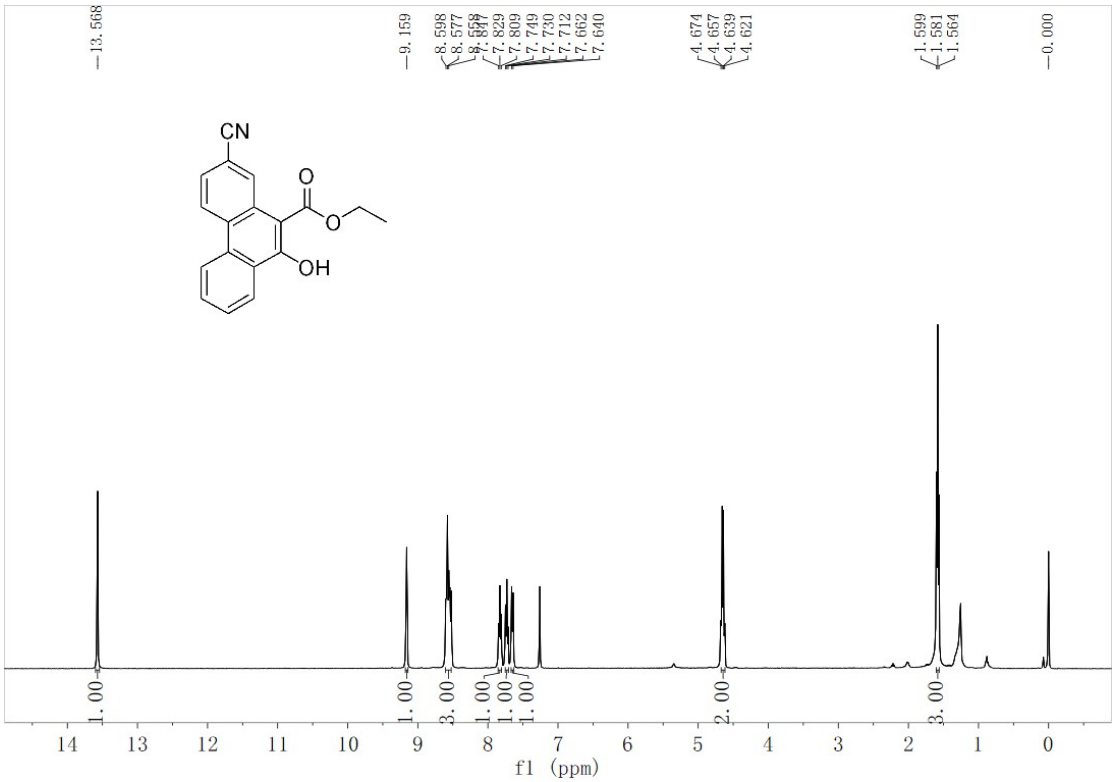
2k – ^{13}C NMR (101 MHz, $CDCl_3$)



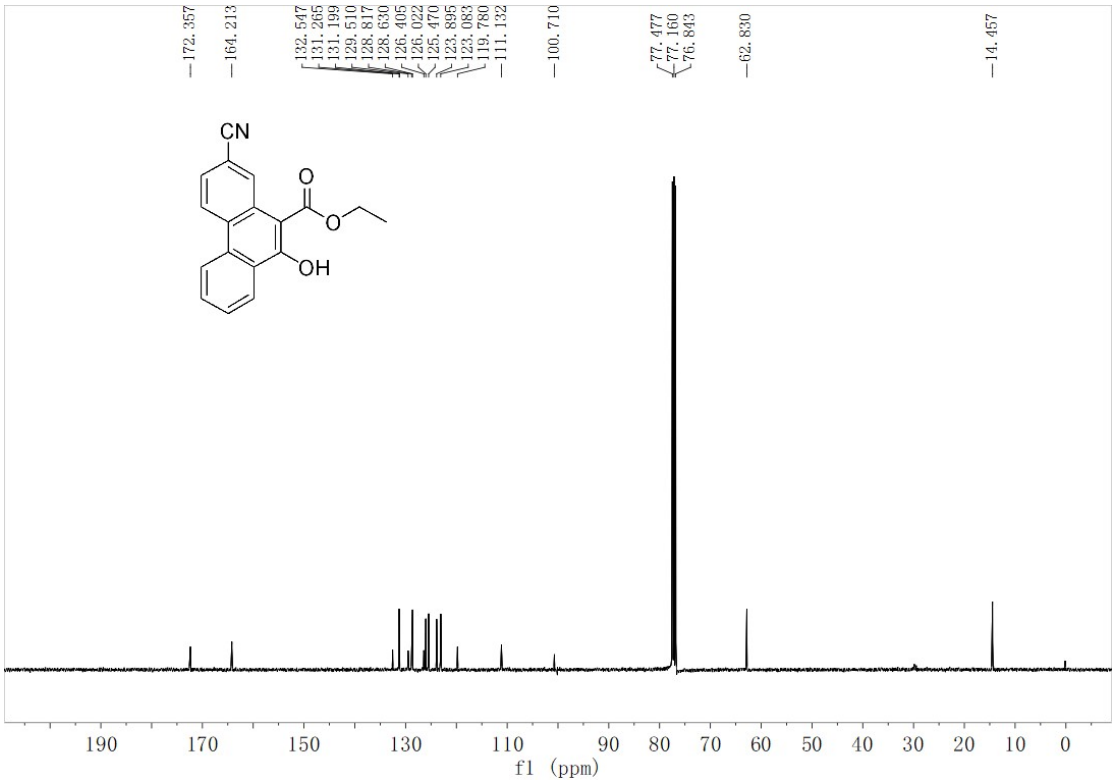
2k – ^{19}F NMR (377 MHz, $CDCl_3$)



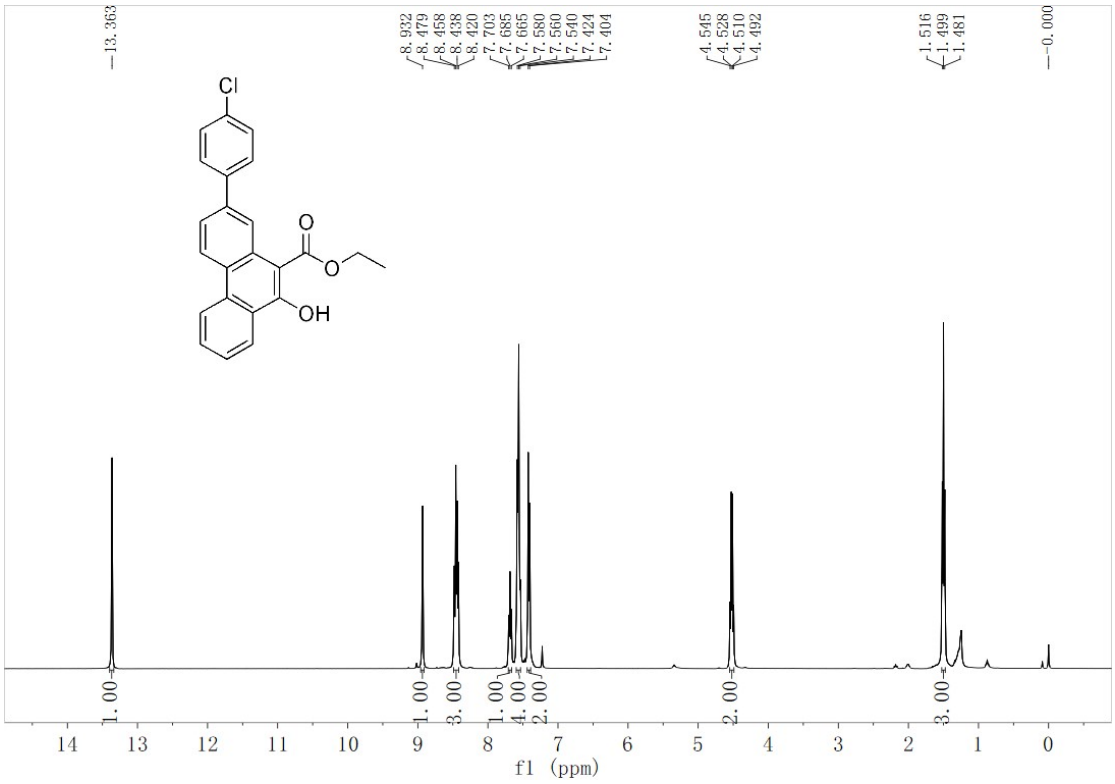
2l – ^{1}H NMR (400 MHz, $CDCl_3$)



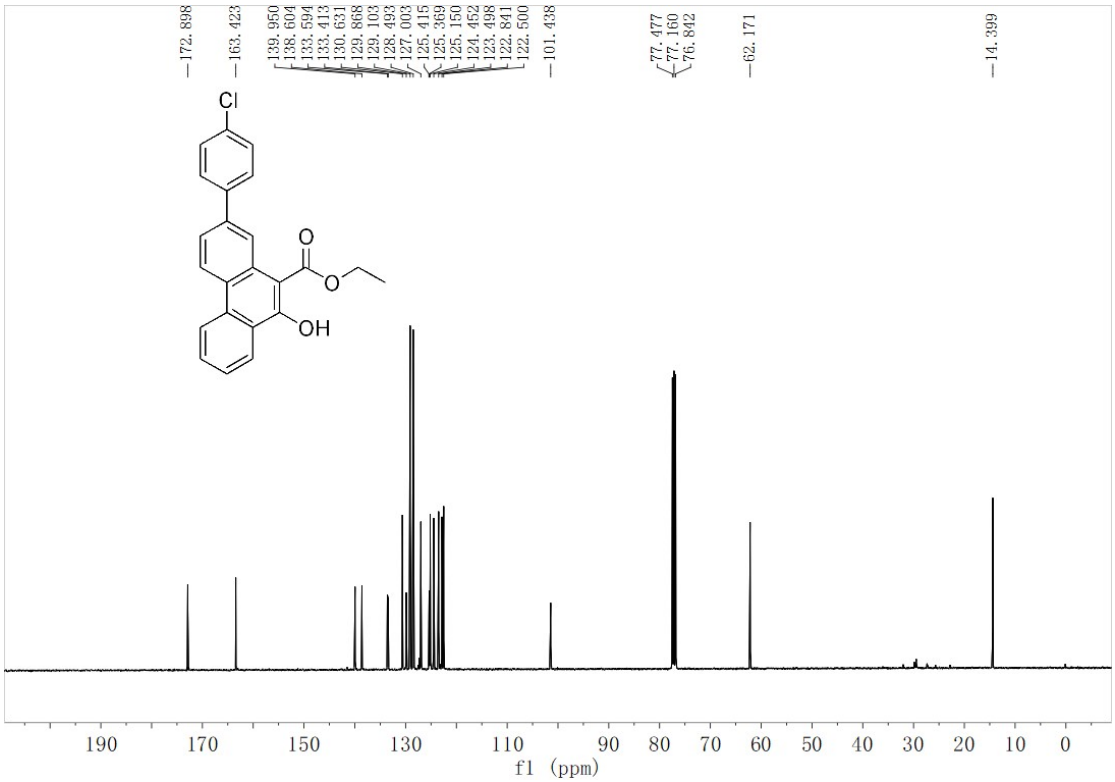
2l – ^{13}C NMR (101 MHz, $CDCl_3$)



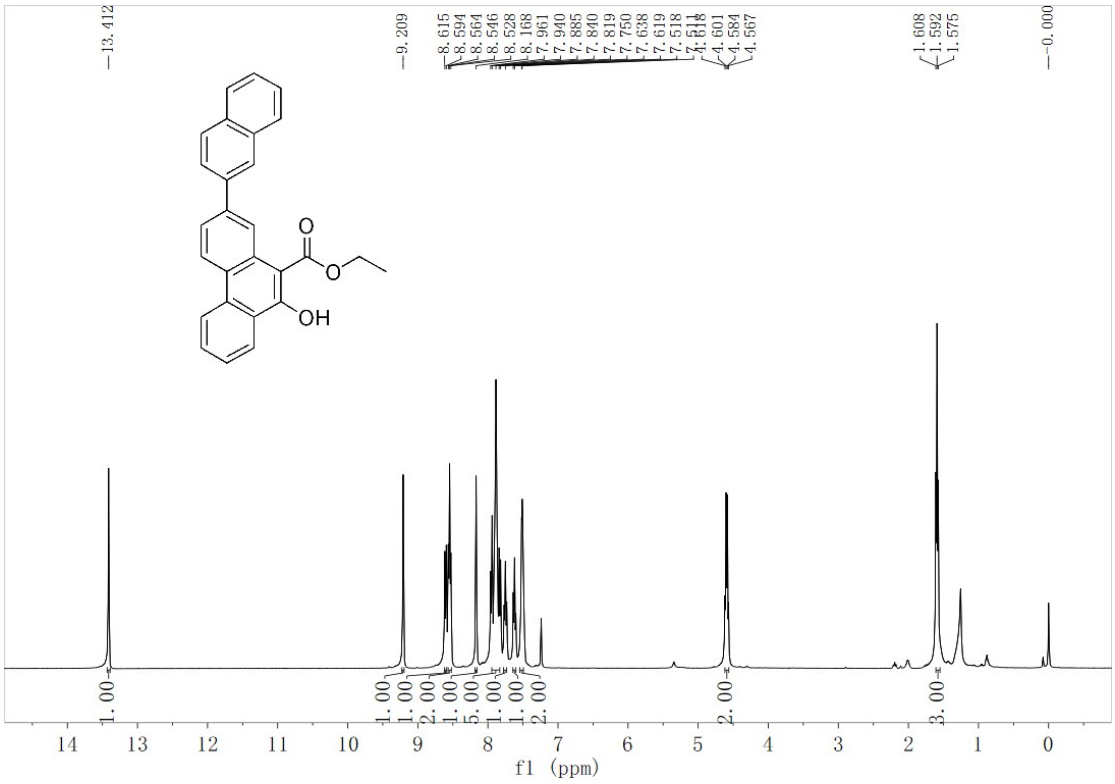
2m – ^{1}H NMR (400 MHz, $CDCl_3$)



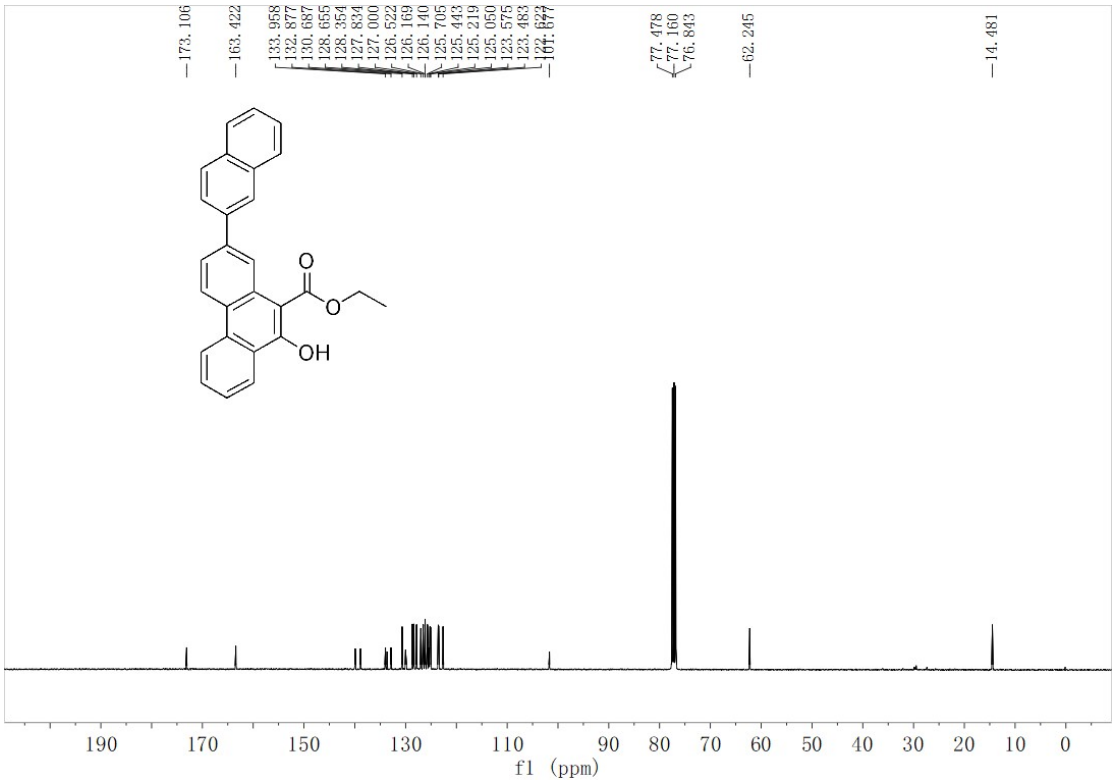
2m – ^{13}C NMR (101 MHz, $CDCl_3$)



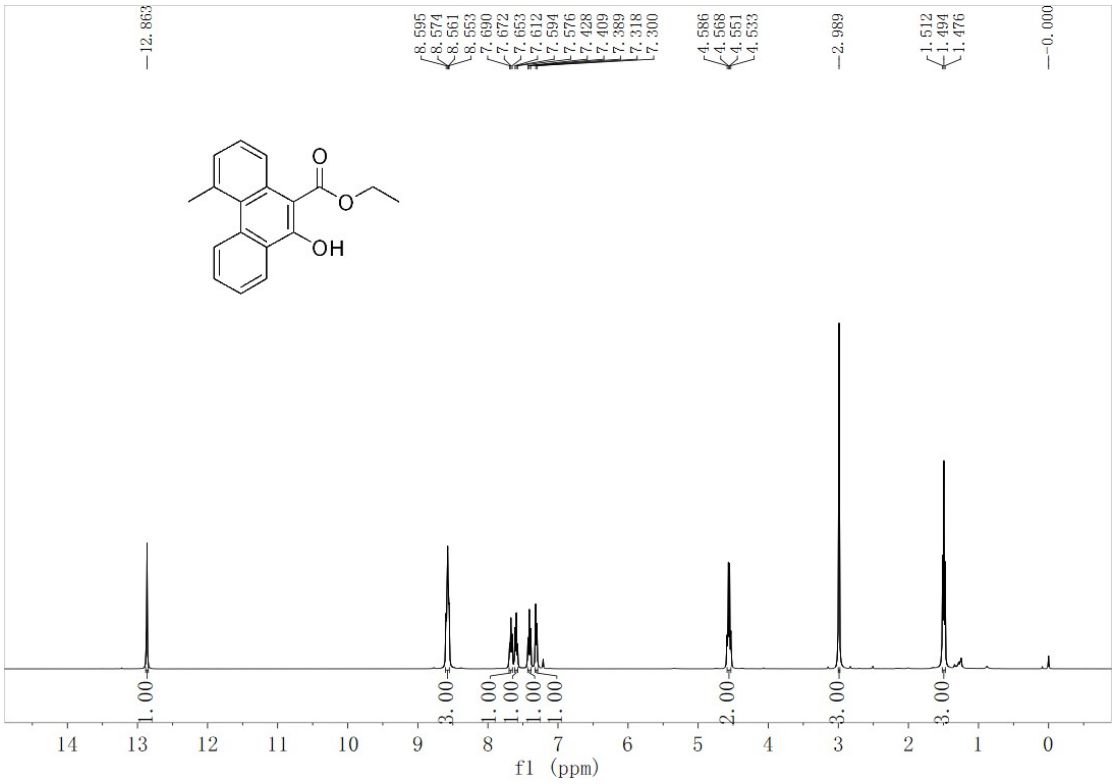
2n – ^{1}H NMR (400 MHz, $CDCl_3$)



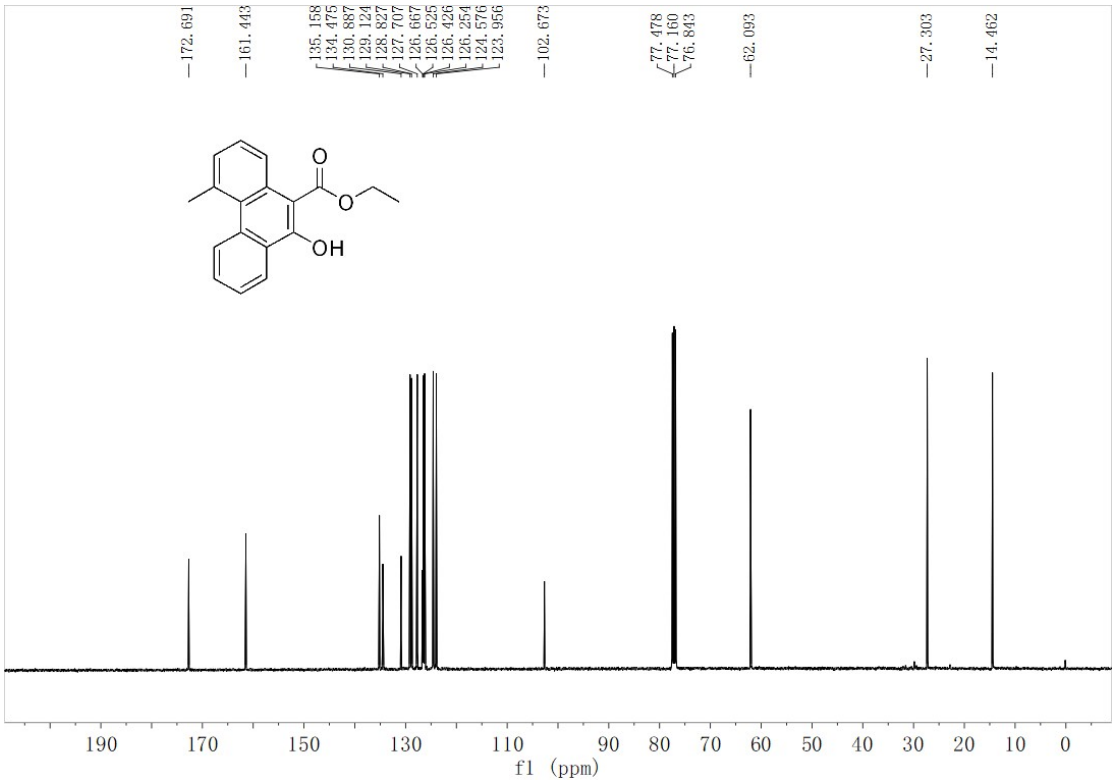
2n – ^{13}C NMR (101 MHz, $CDCl_3$)



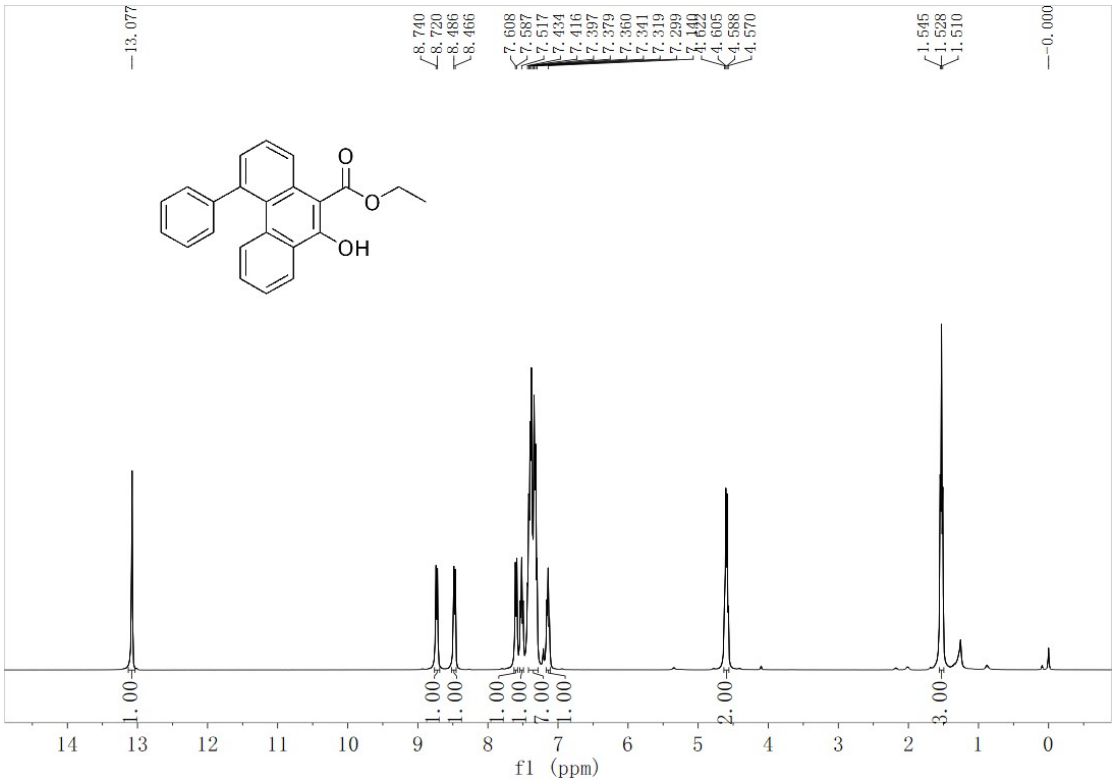
2o – ^{1}H NMR (400 MHz, $CDCl_3$)



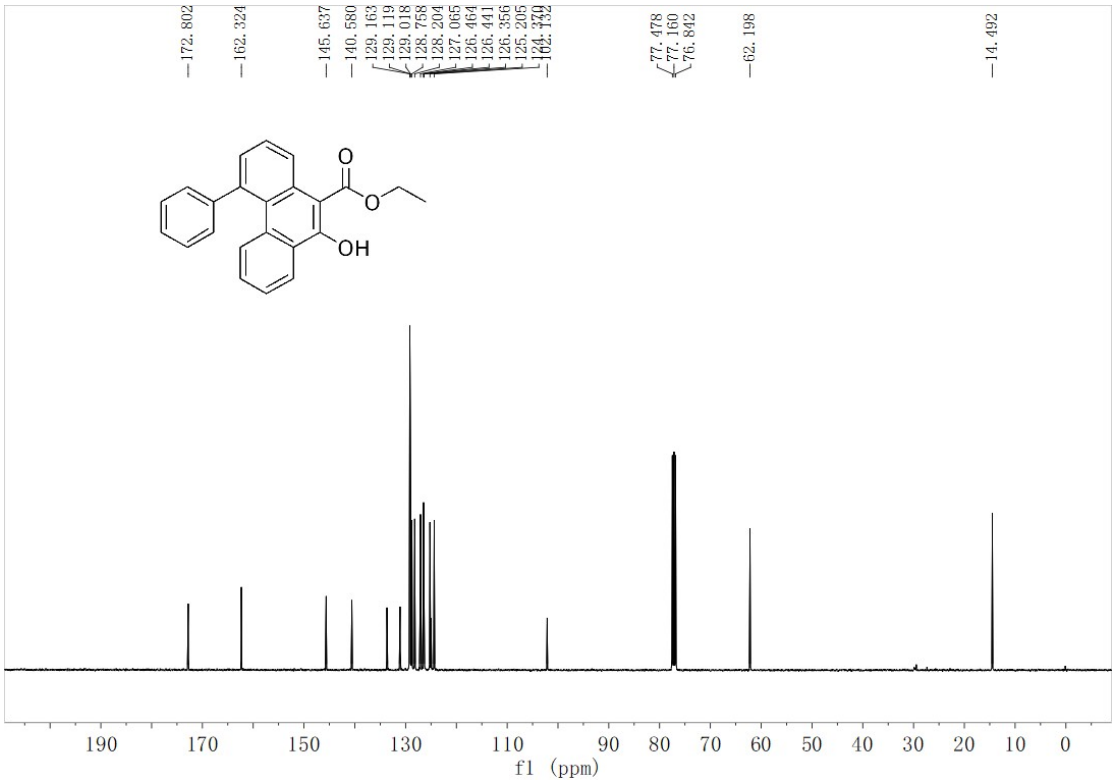
2o – ^{13}C NMR (101 MHz, $CDCl_3$)



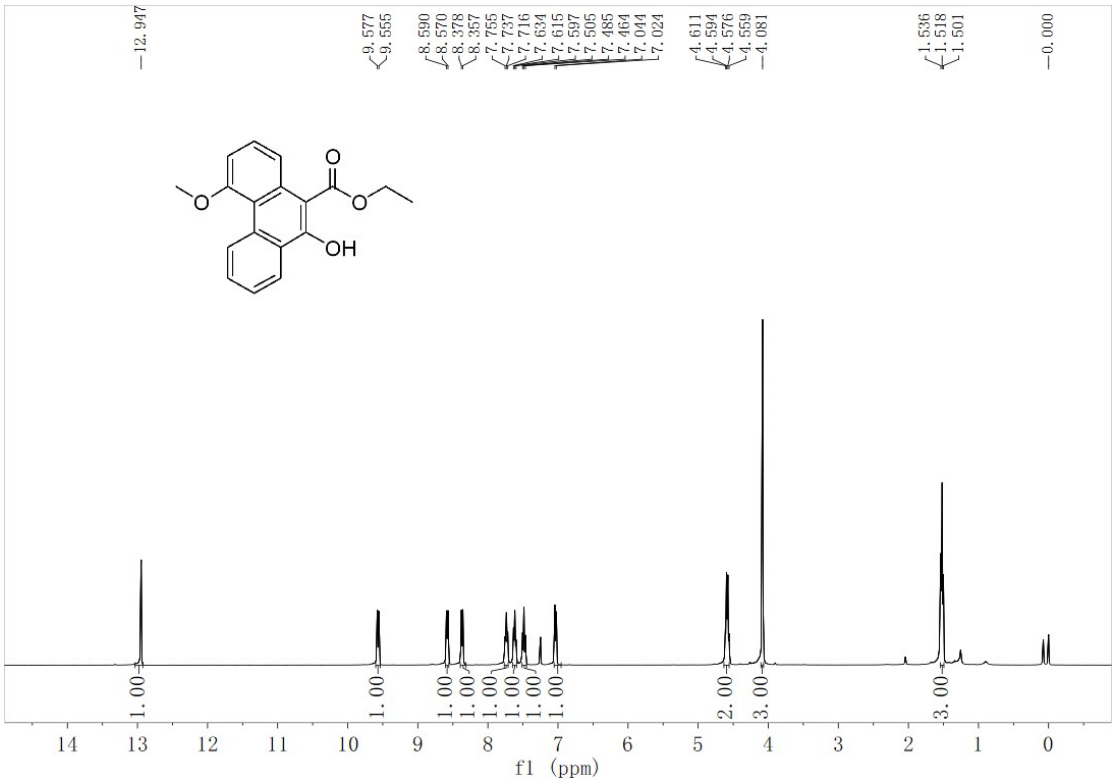
2p – ^{1}H NMR (400 MHz, $CDCl_3$)



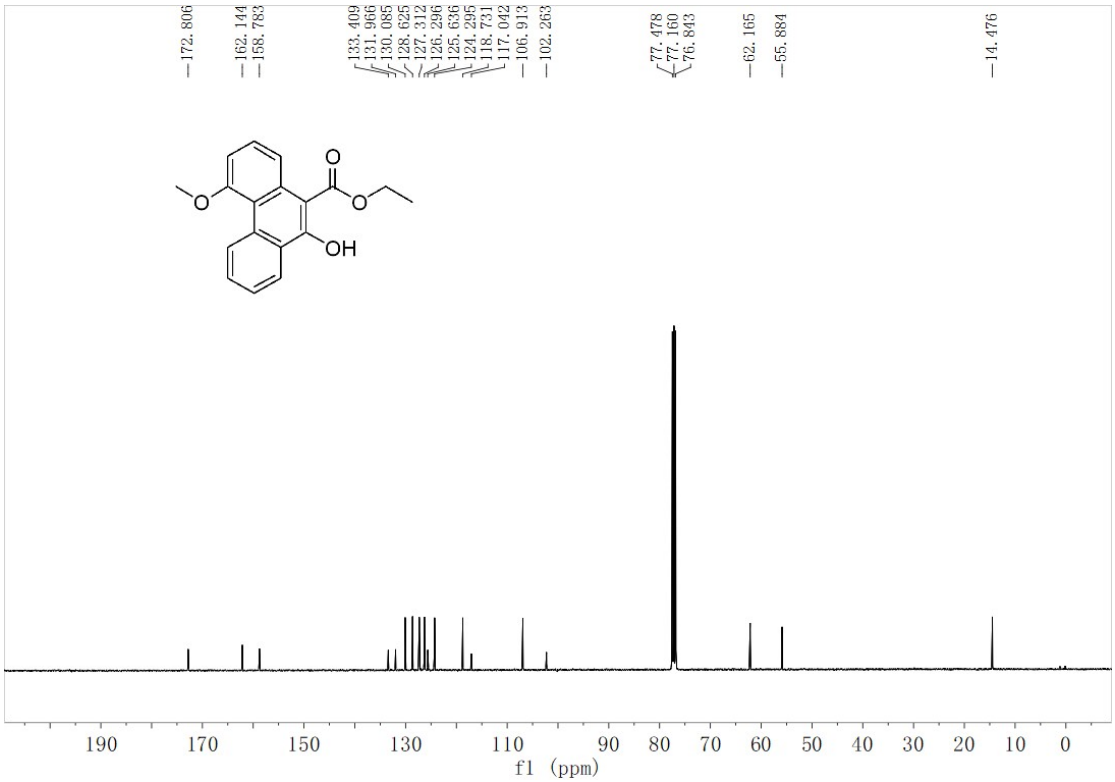
2p – ^{13}C NMR (101 MHz, $CDCl_3$)



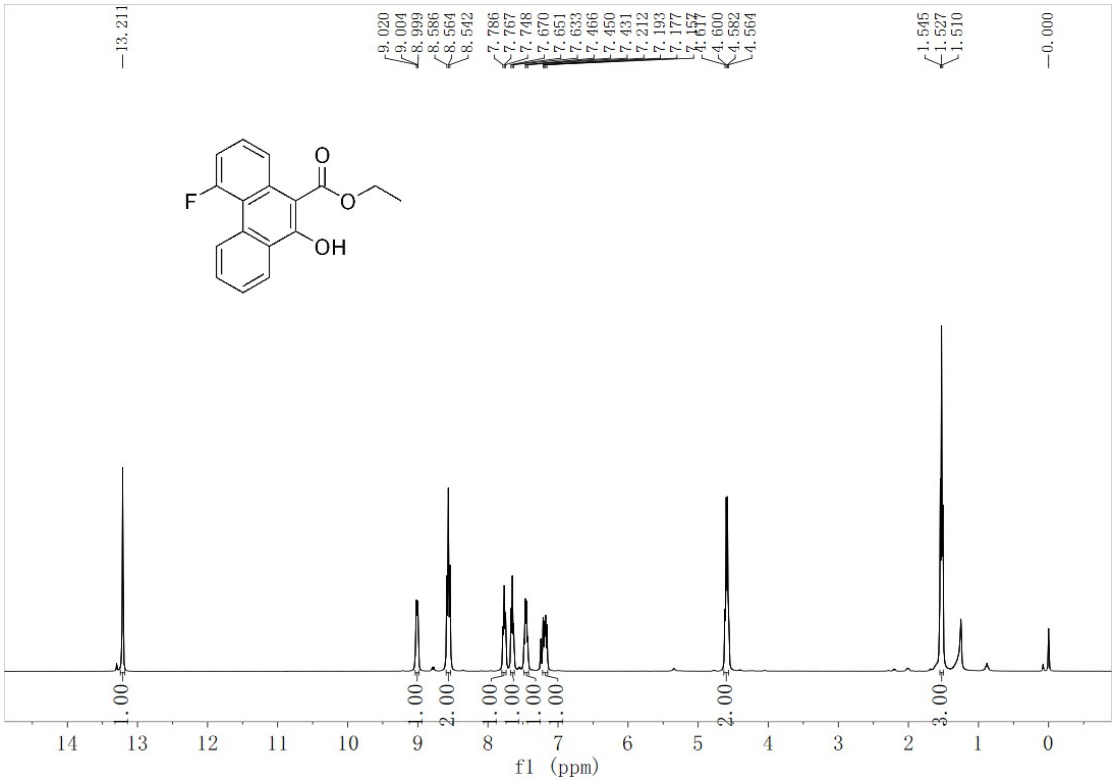
2q – ^{1}H NMR (400 MHz, $CDCl_3$)



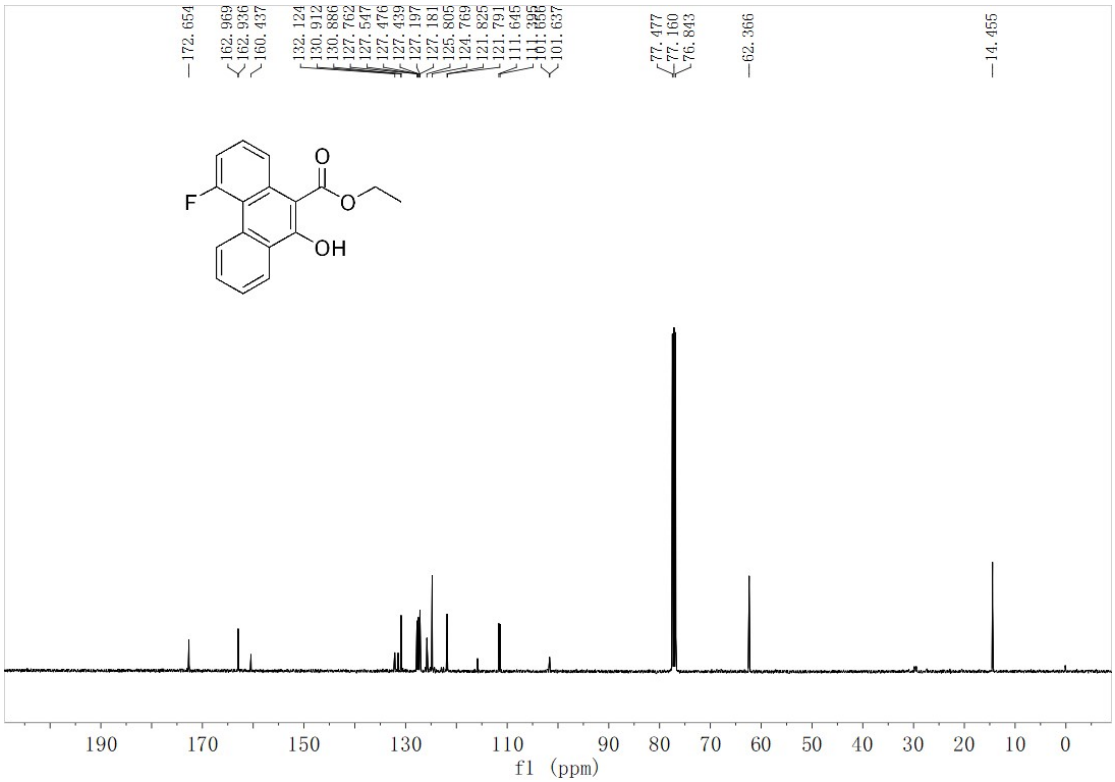
2q – ^{13}C NMR (101 MHz, $CDCl_3$)



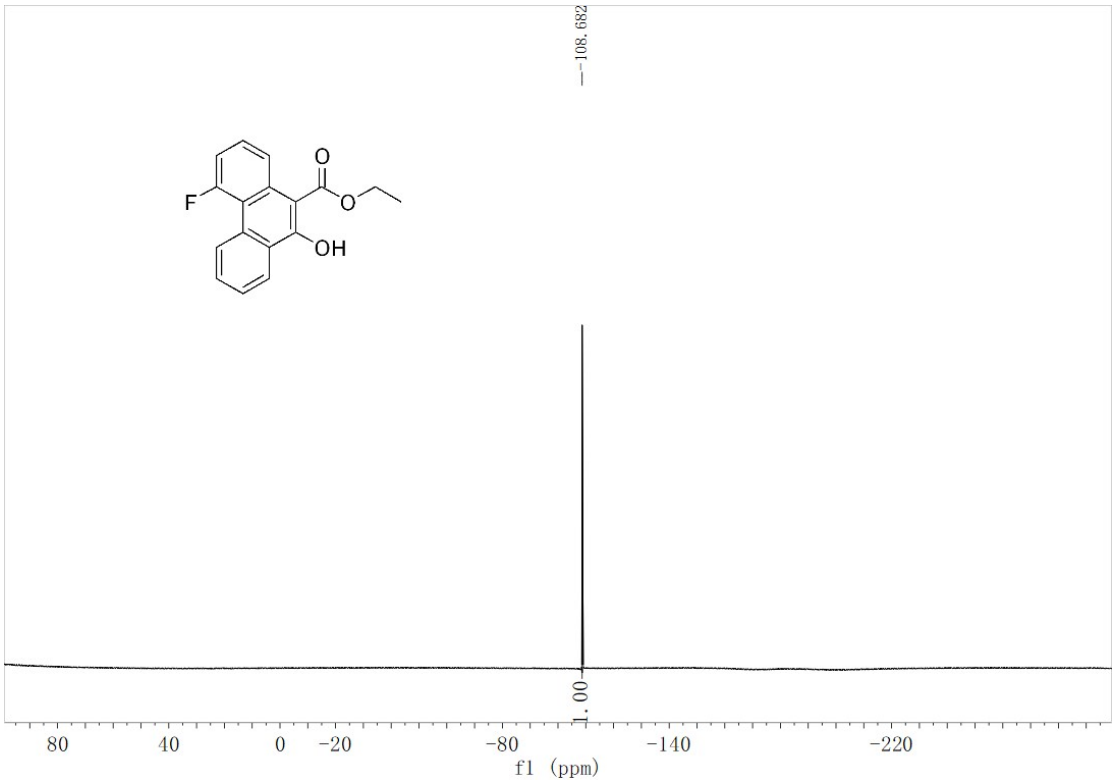
2r – ^{1}H NMR (400 MHz, $CDCl_3$)



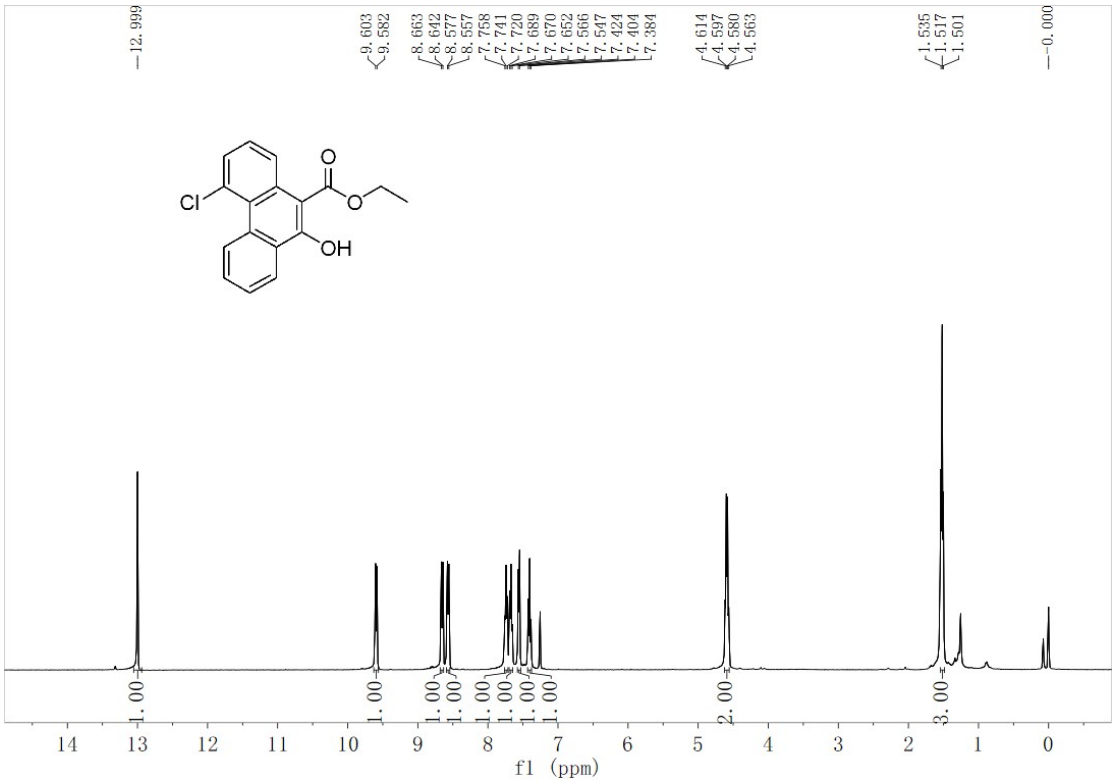
2r – ^{13}C NMR (101 MHz, $CDCl_3$)



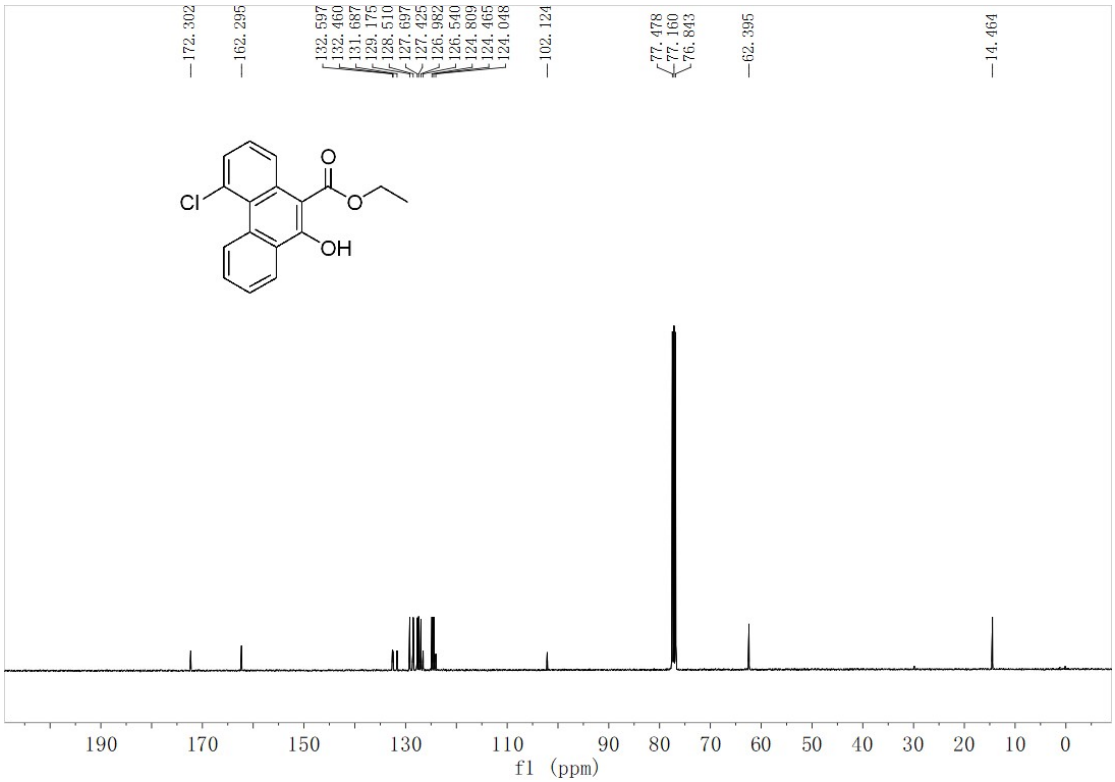
2r – ^{19}F NMR (377 MHz, $CDCl_3$)



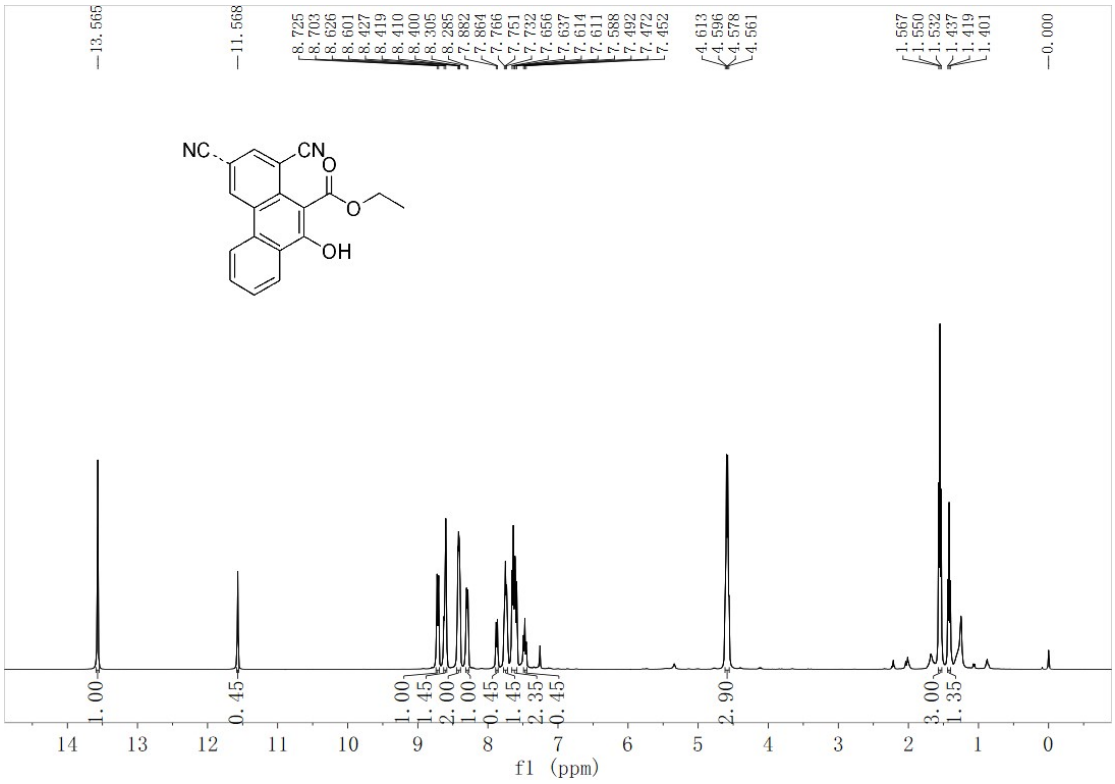
2s – ^{1}H NMR (400 MHz, $CDCl_3$)



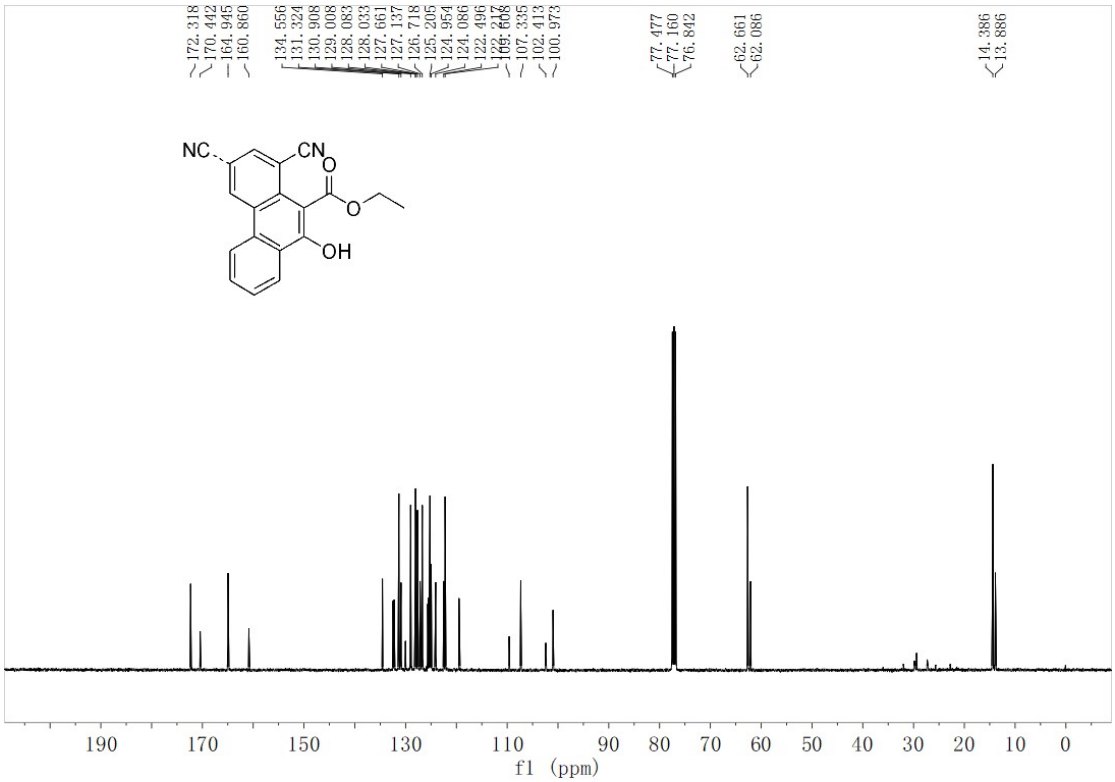
2s – ^{13}C NMR (101 MHz, $CDCl_3$)



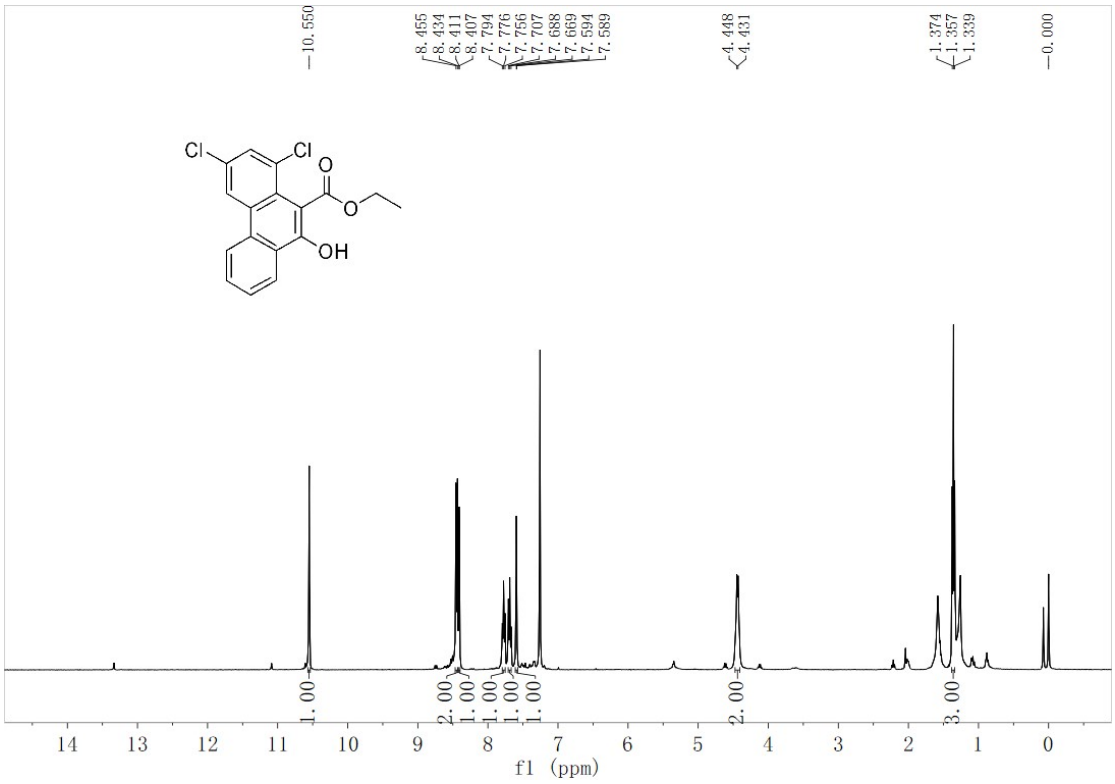
2t:(2t') – ^{1}H NMR (400 MHz, $CDCl_3$)



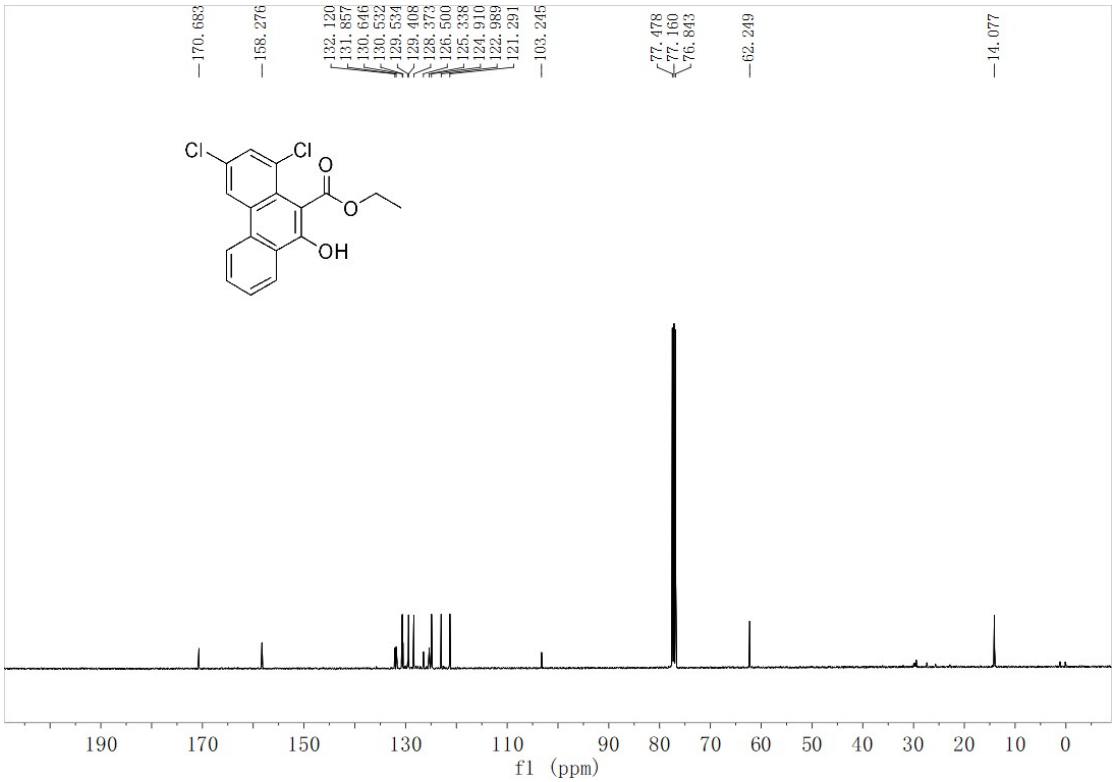
2t:(2t') – ^{13}C NMR (101 MHz, $CDCl_3$)



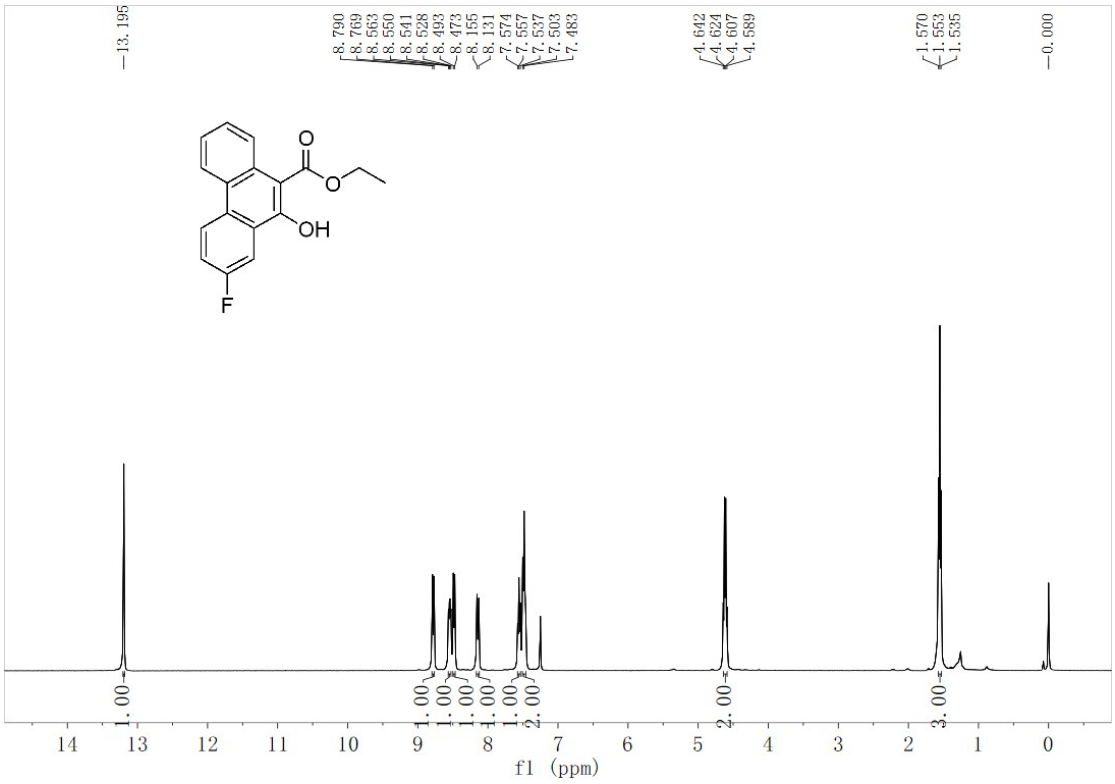
2u – ^{1}H NMR (400 MHz, $CDCl_3$)



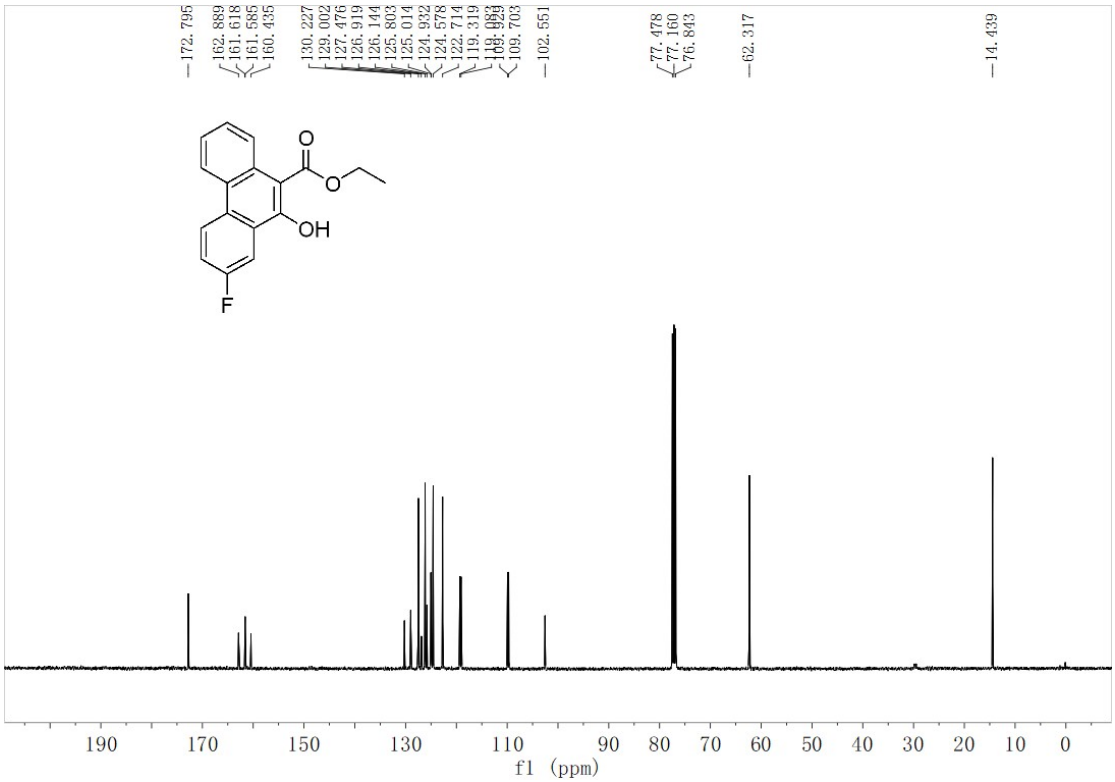
2u – ^{13}C NMR (101 MHz, $CDCl_3$)



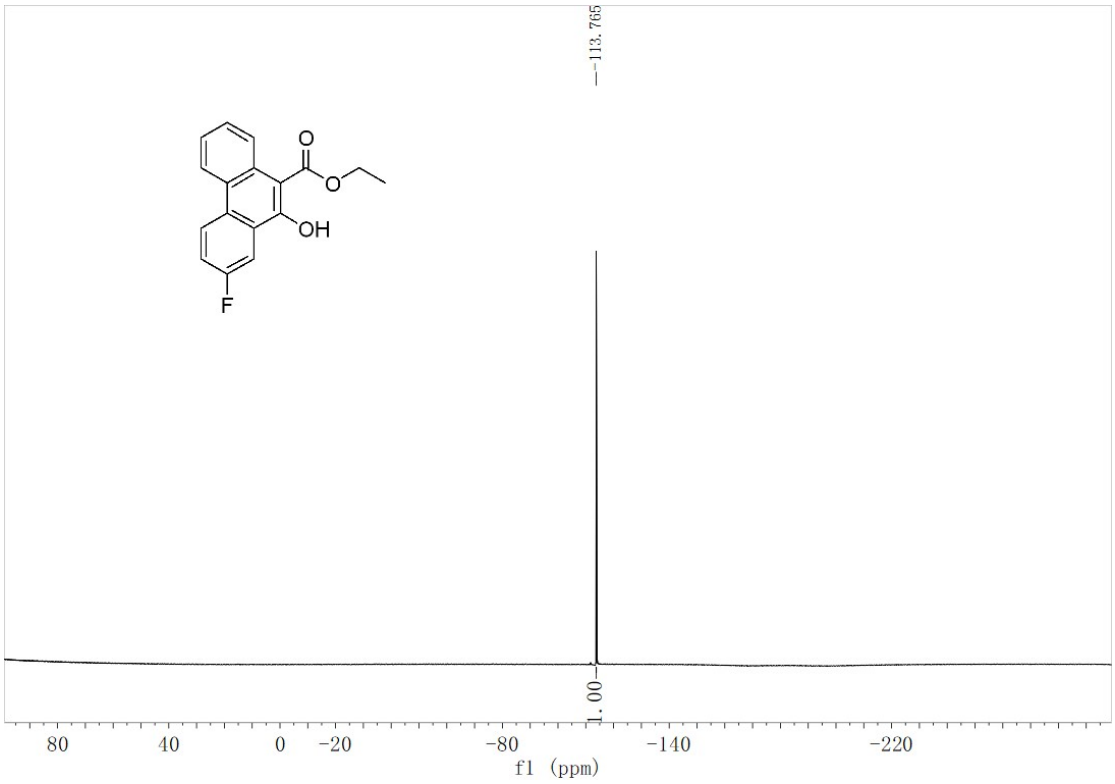
2v – ^{1}H NMR (400 MHz, $CDCl_3$)



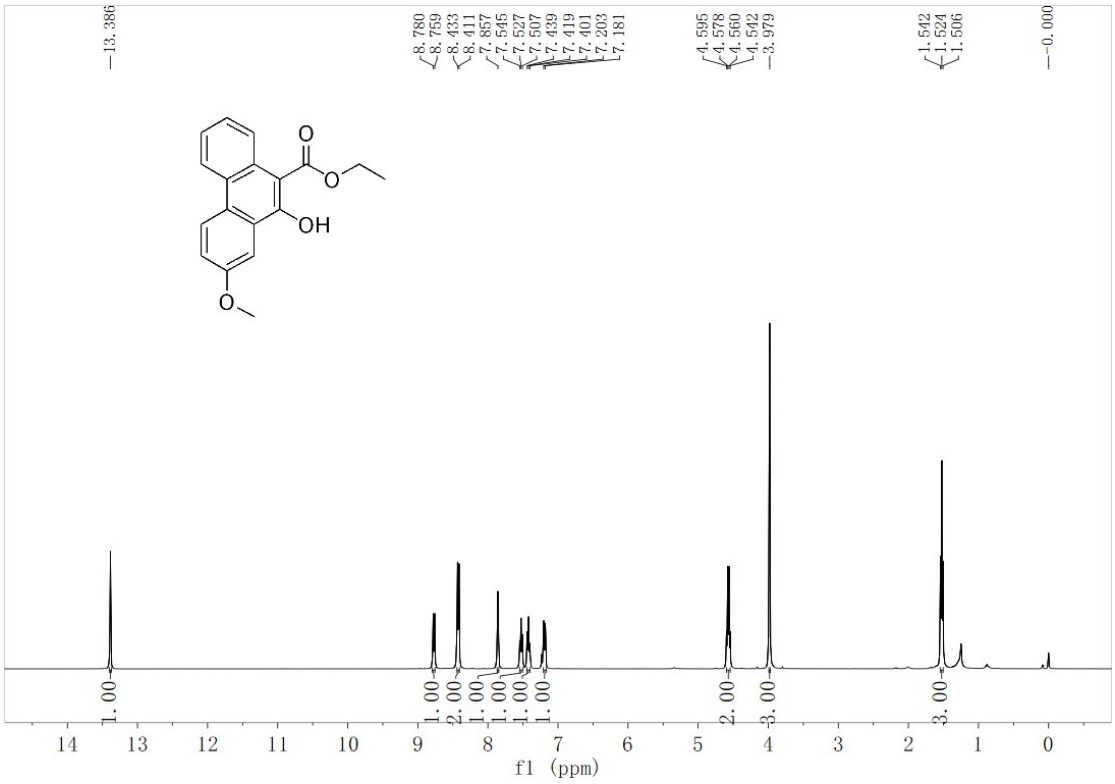
2v – ^{13}C NMR (101 MHz, $CDCl_3$)



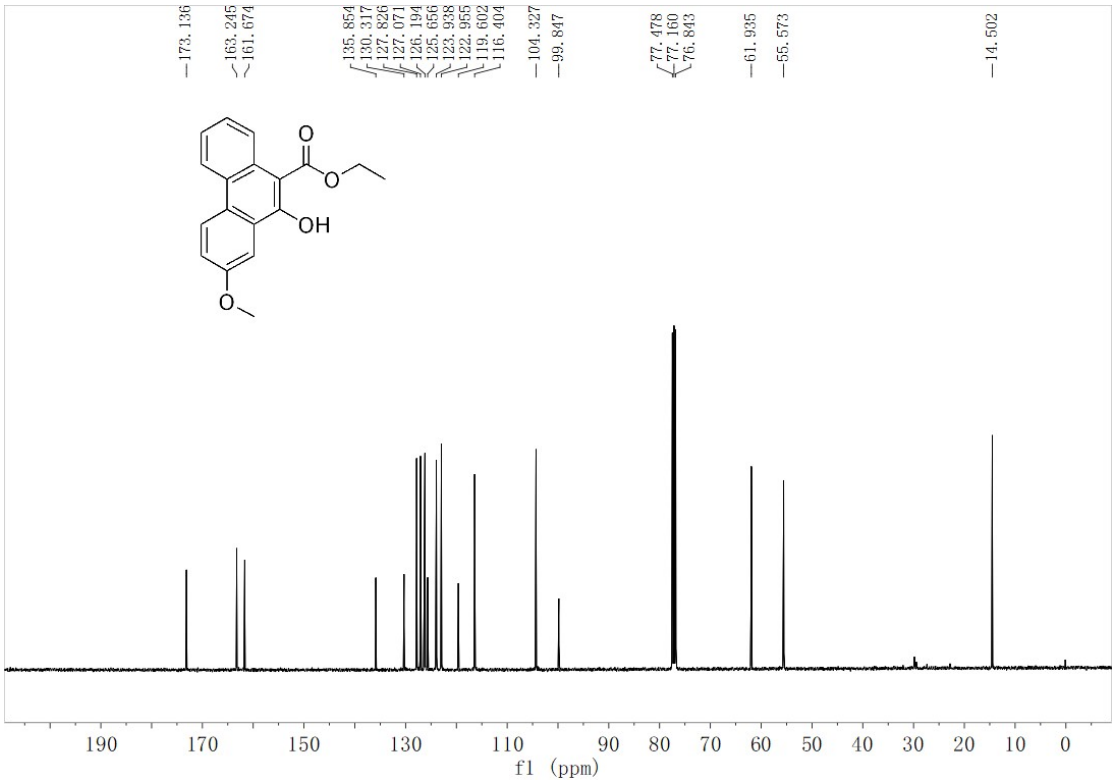
2v – ^{19}F NMR (377 MHz, $CDCl_3$)



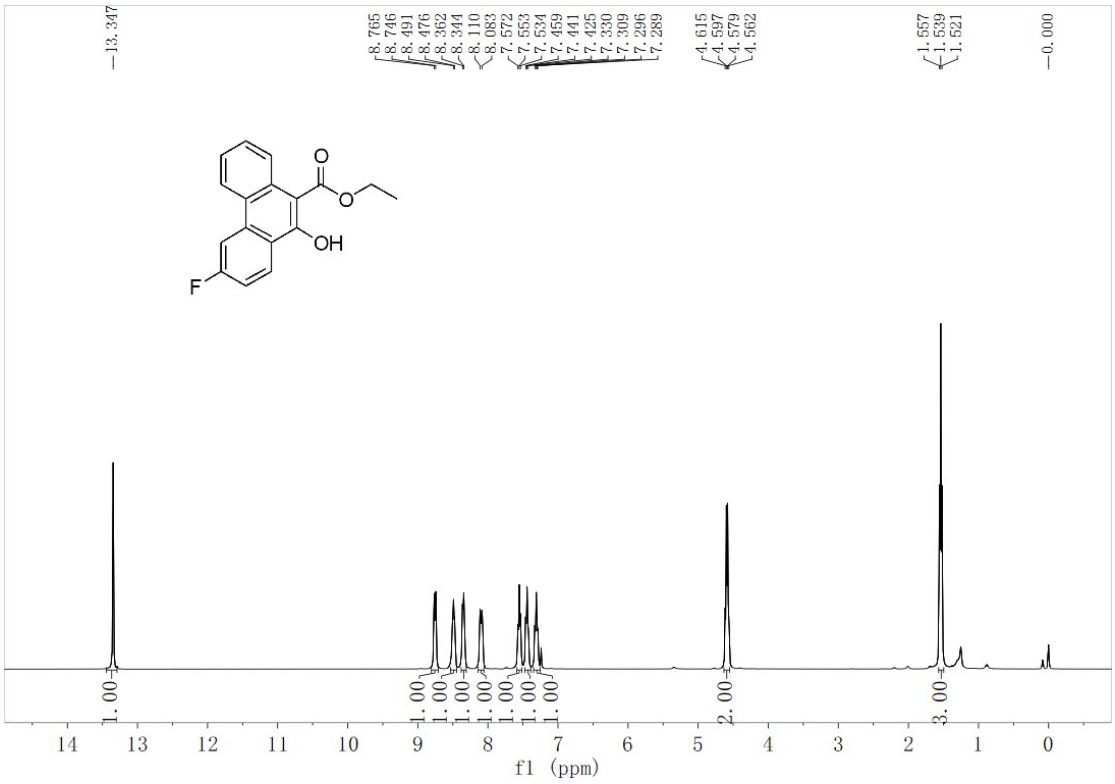
2w – ^{1}H NMR (400 MHz, $CDCl_3$)



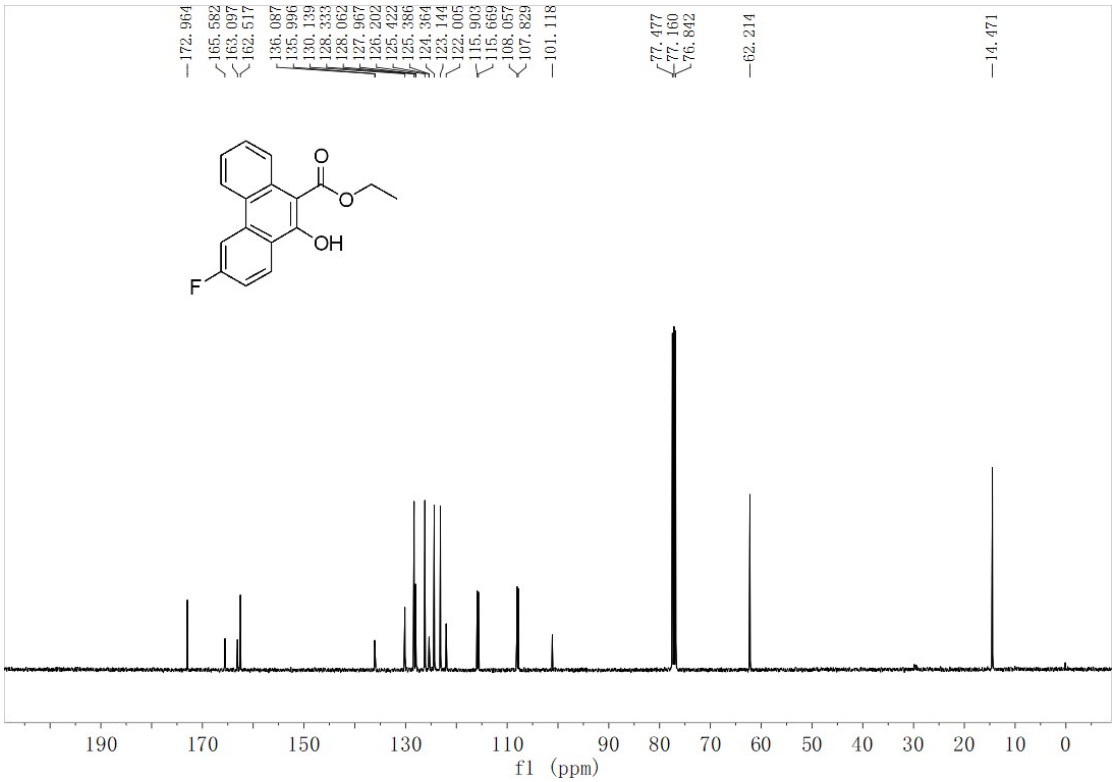
2w – ^{13}C NMR (101 MHz, $CDCl_3$)



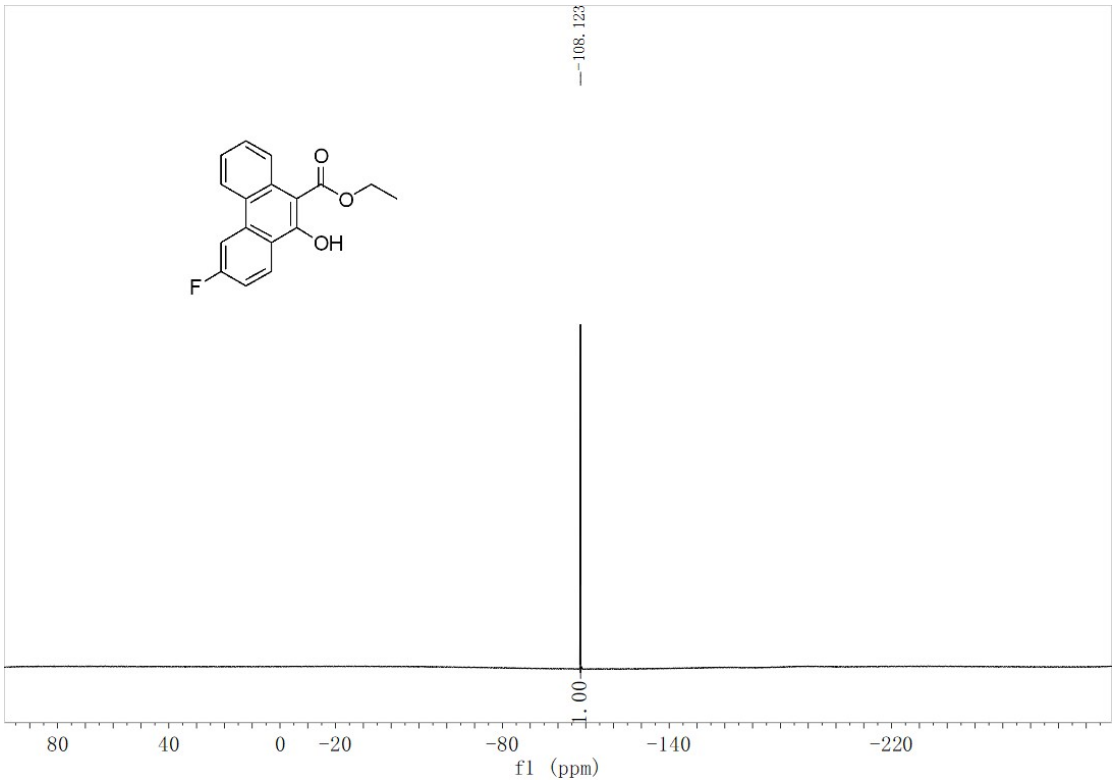
2x – ^{1}H NMR (400 MHz, $CDCl_3$)



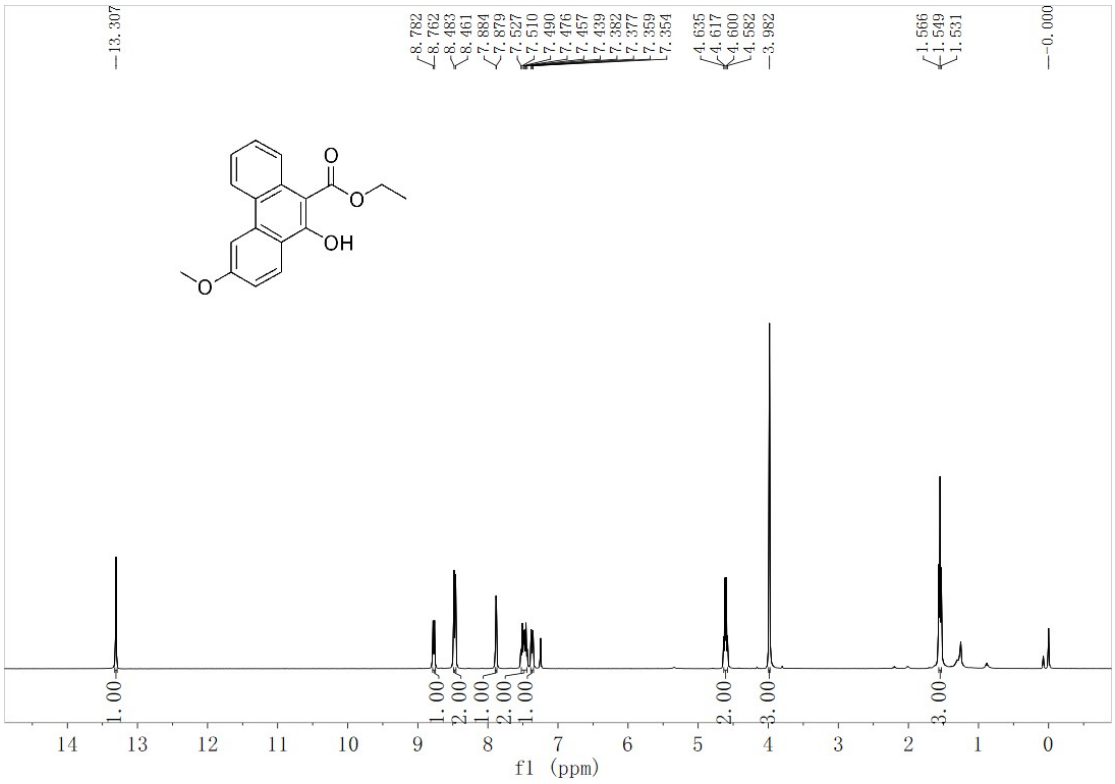
2x – ^{13}C NMR (101 MHz, $CDCl_3$)



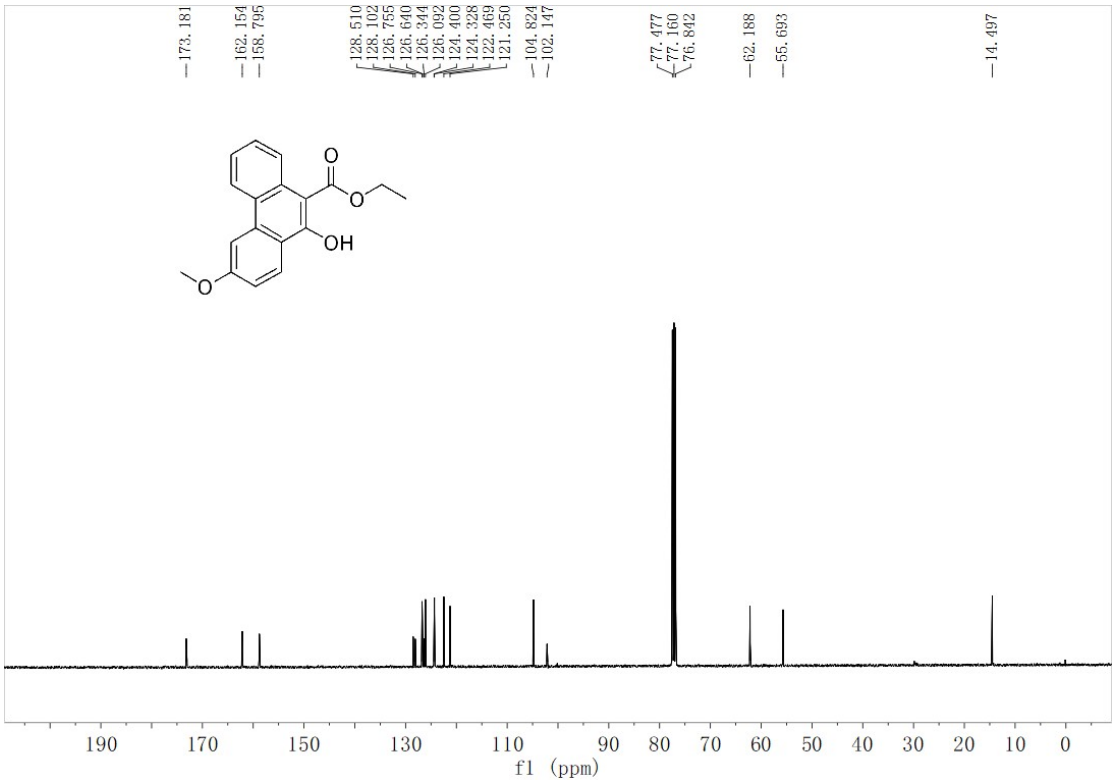
2x – ^{19}F NMR (377 MHz, $CDCl_3$)



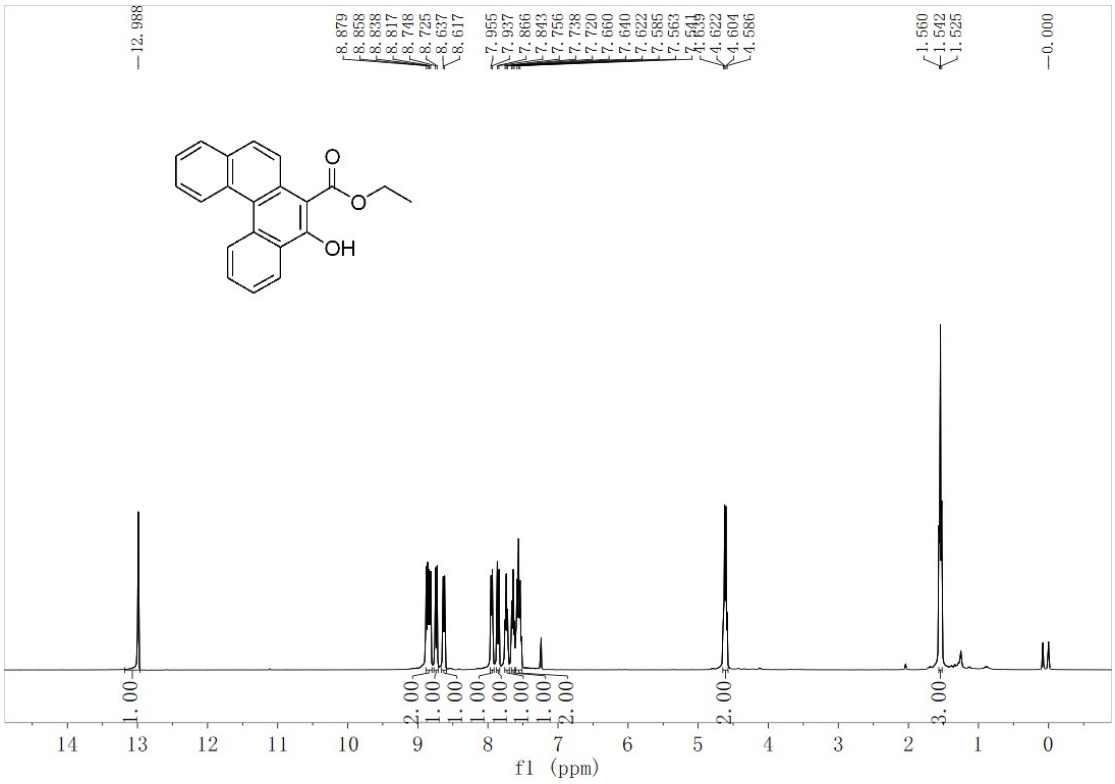
2y – ^{1}H NMR (400 MHz, $CDCl_3$)



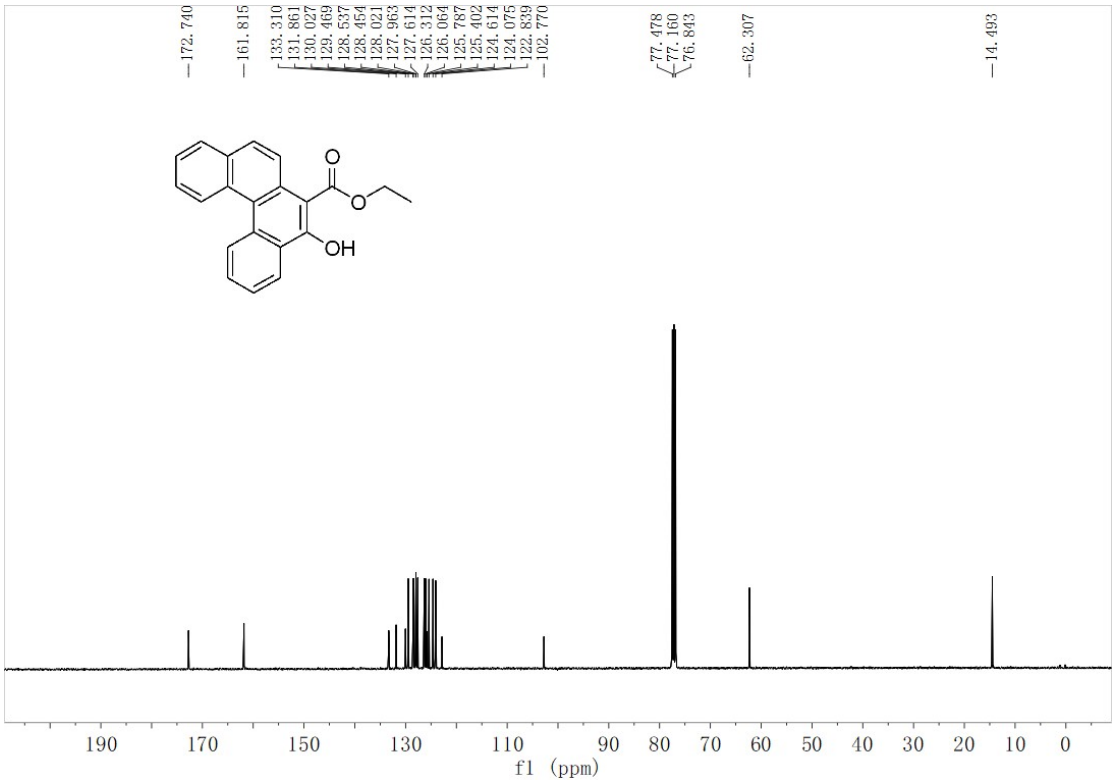
2y – ^{13}C NMR (101 MHz, $CDCl_3$)



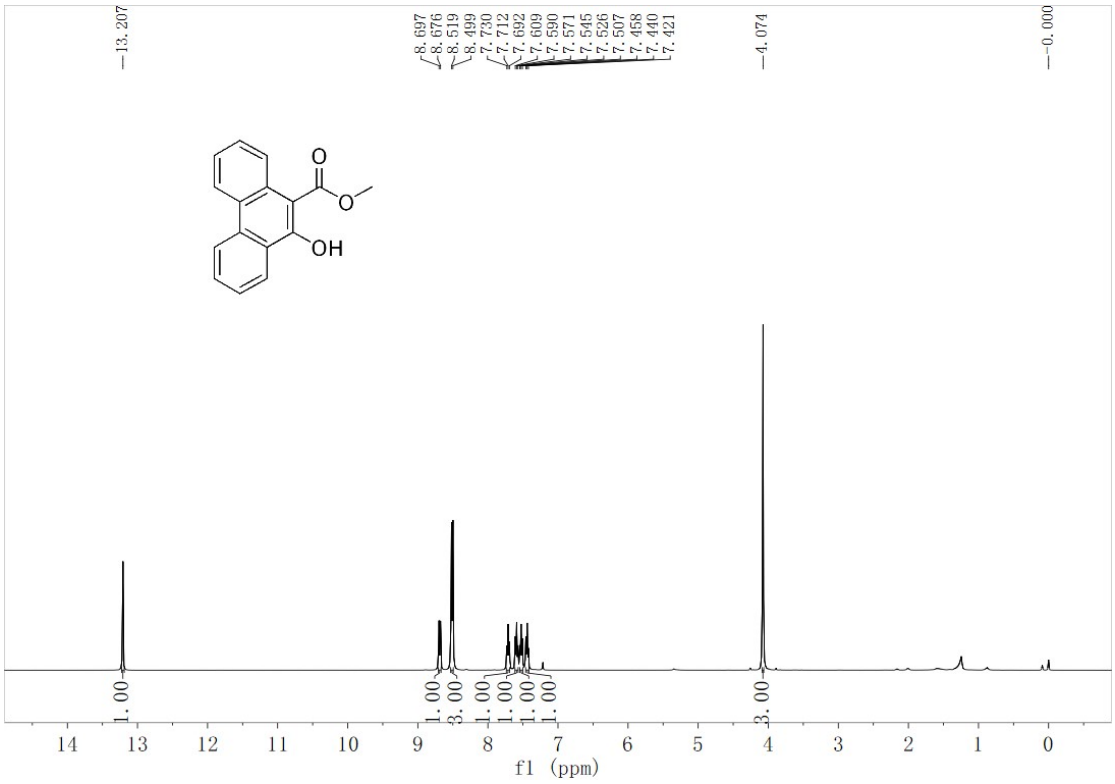
2z – ^{1}H NMR (400 MHz, $CDCl_3$)



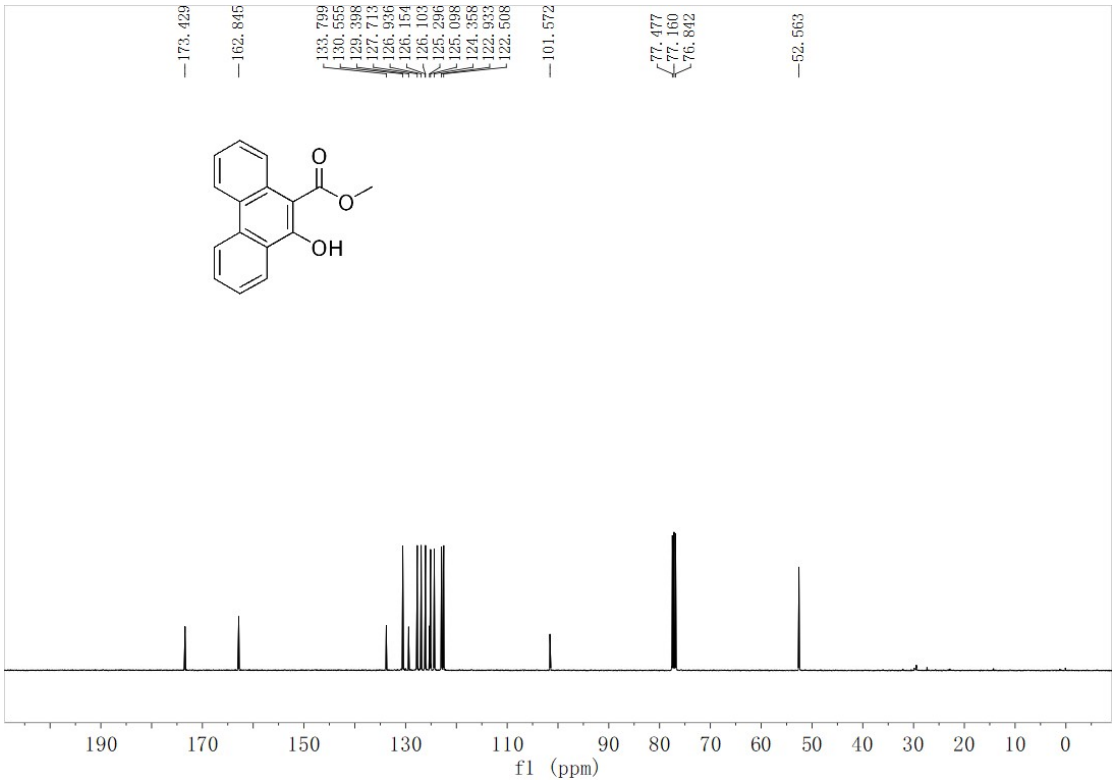
2z – ^{13}C NMR (101 MHz, $CDCl_3$)



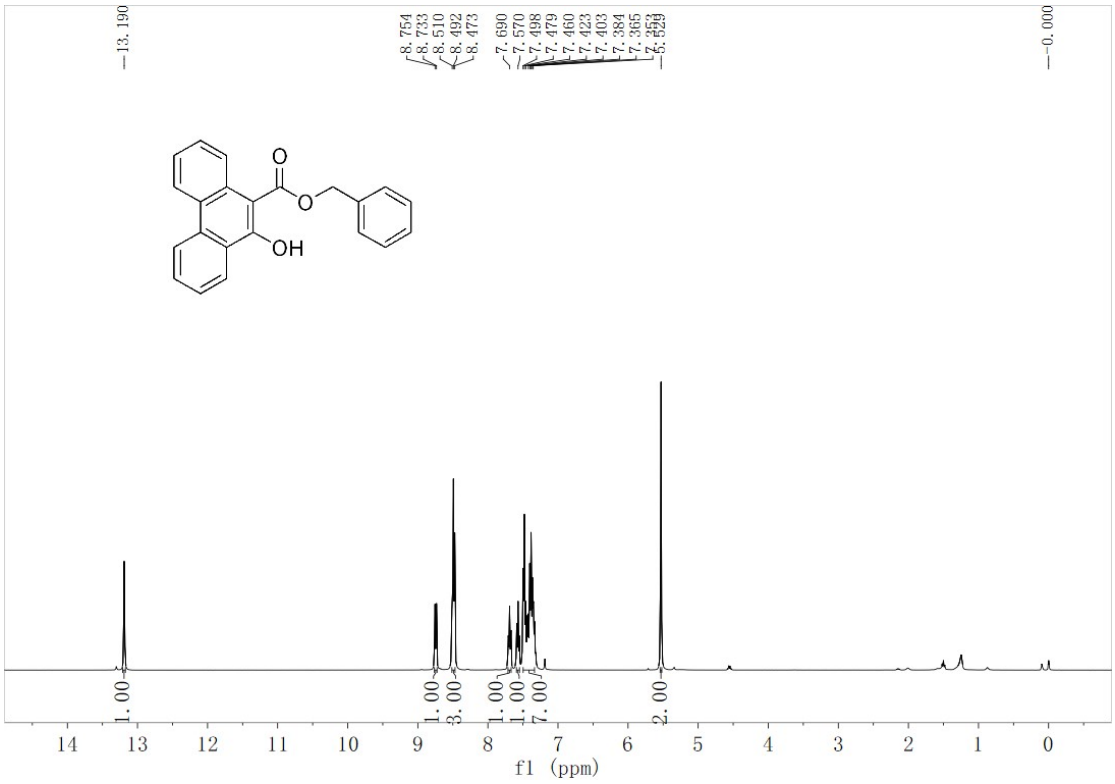
2aa – ^{1}H NMR (400 MHz, $CDCl_3$)



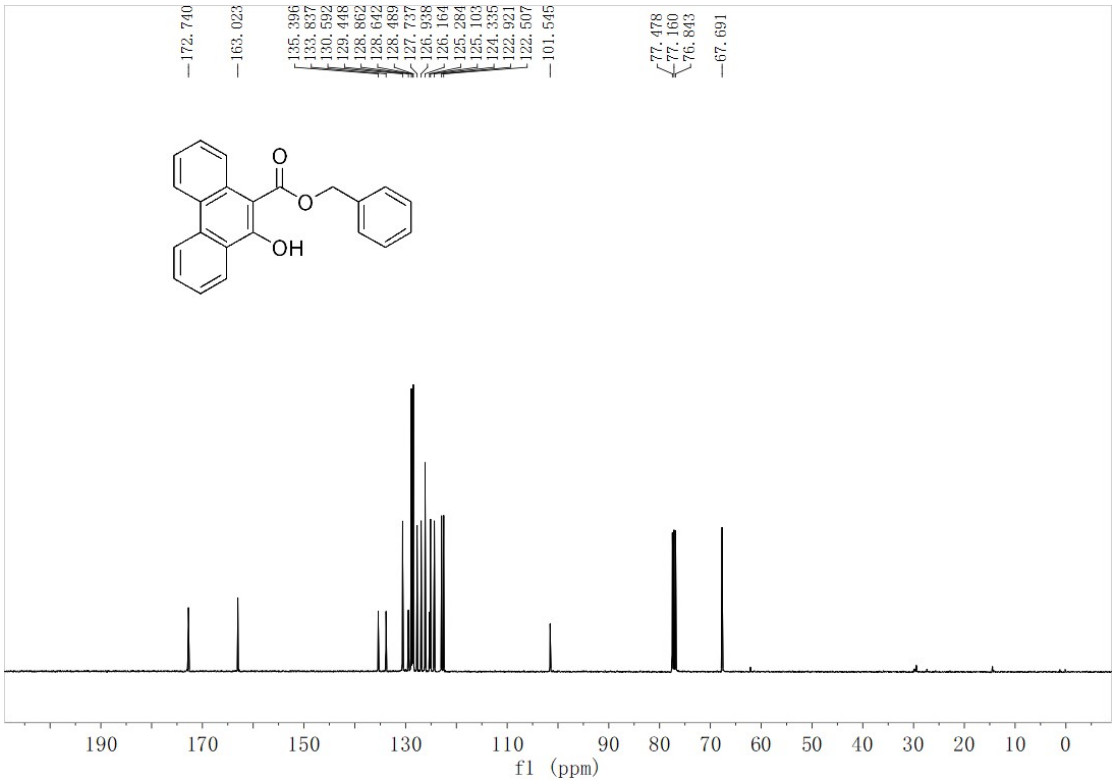
2aa – ^{13}C NMR (101 MHz, $CDCl_3$)



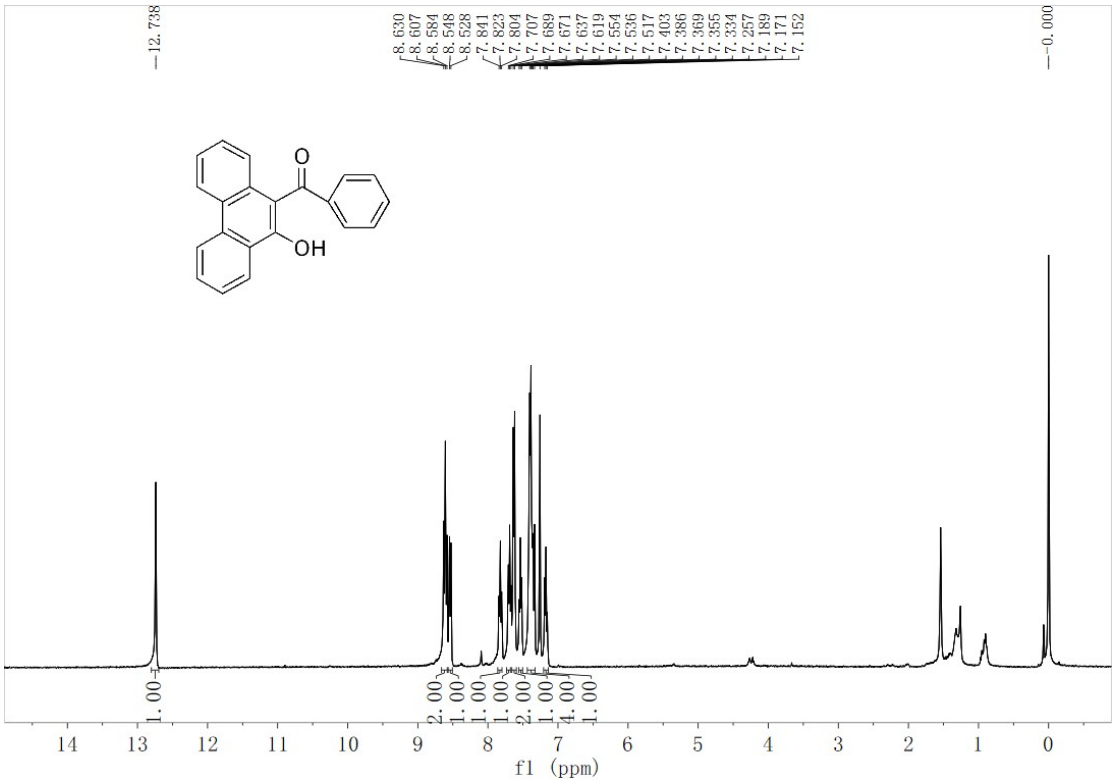
2ab – ^{1}H NMR (400 MHz, $CDCl_3$)



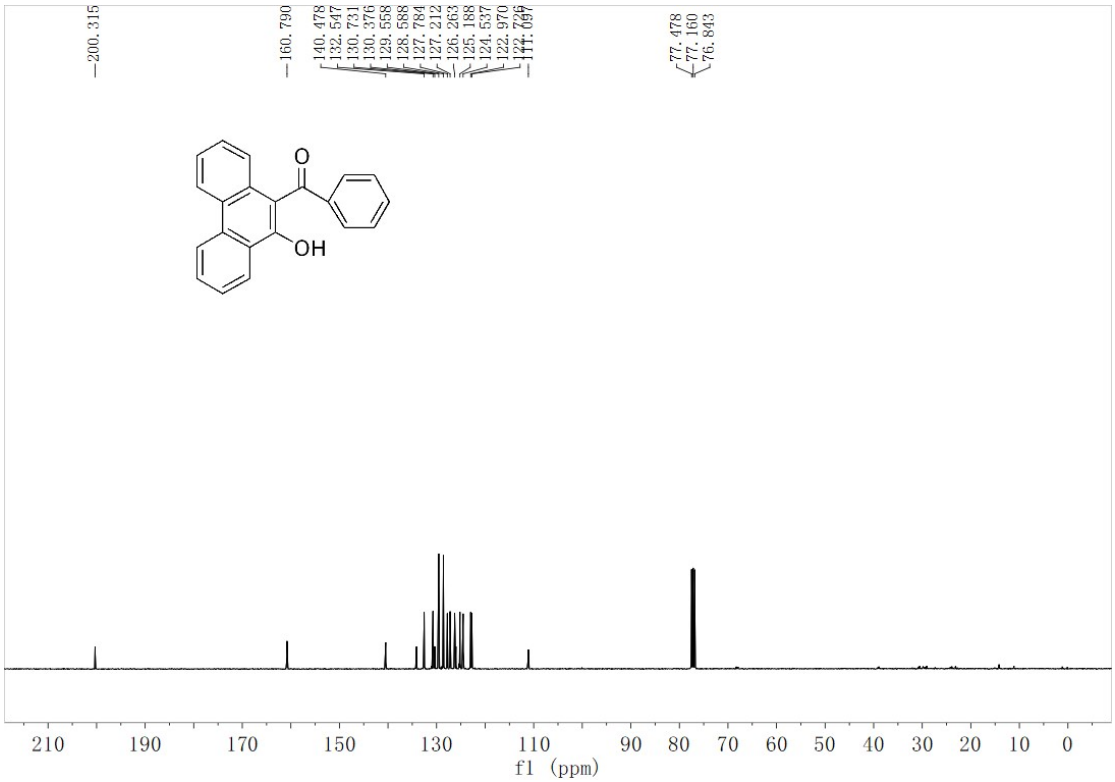
2ab – ^{13}C NMR (101 MHz, $CDCl_3$)



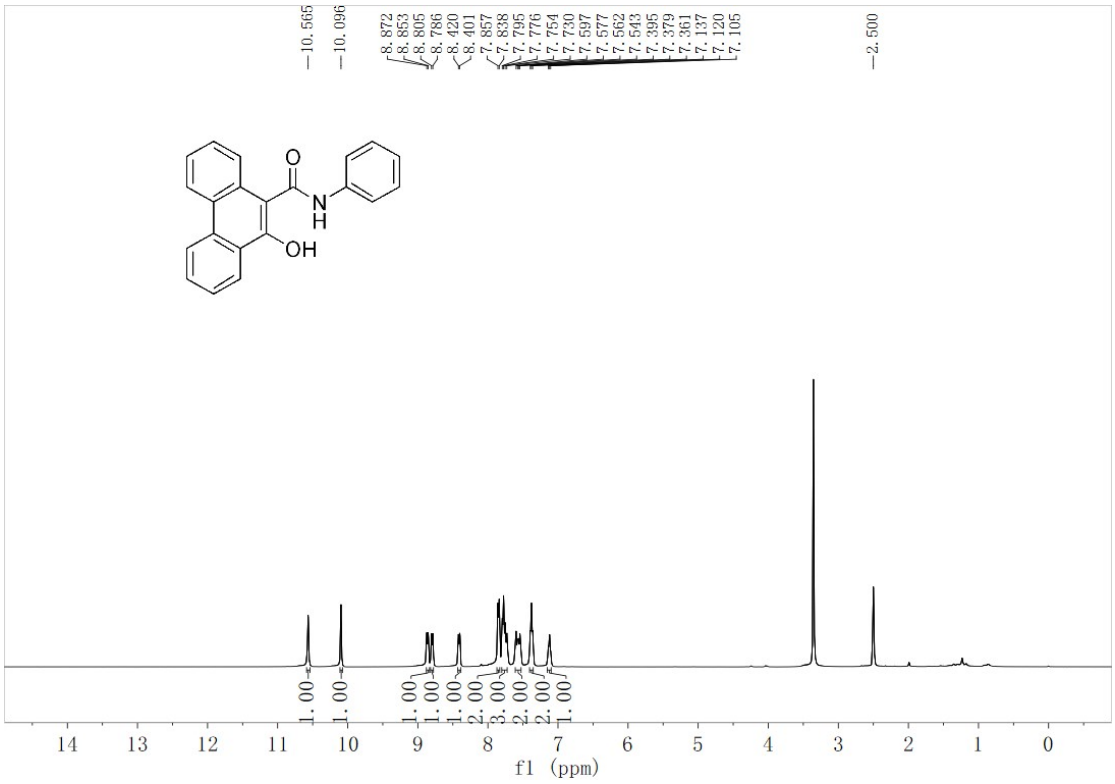
2ac – ^{1}H NMR (400 MHz, $CDCl_3$)



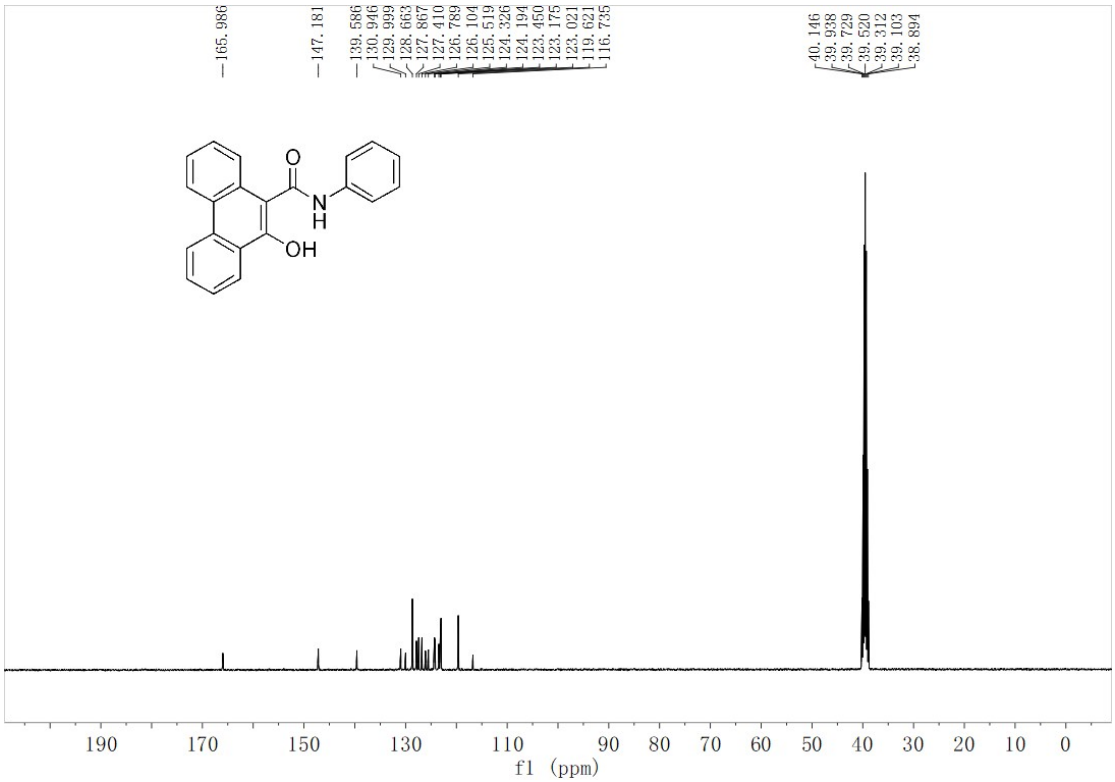
2ac – ^{13}C NMR (101 MHz, $CDCl_3$)



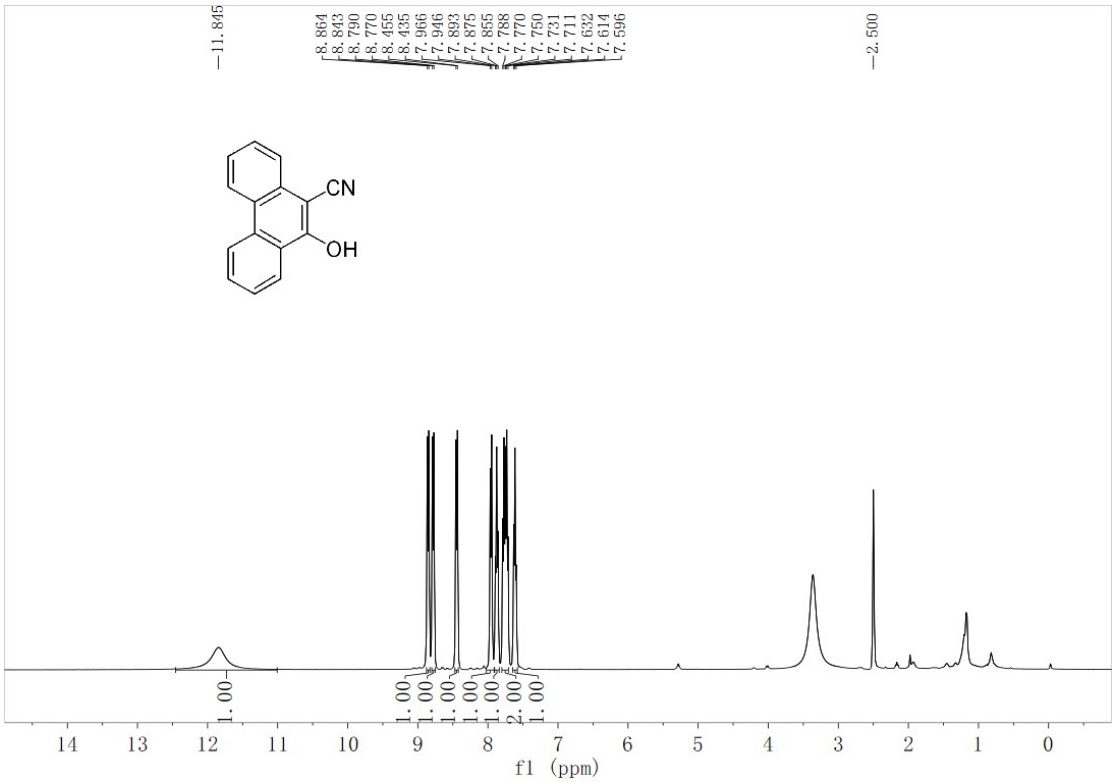
2ad – ^{1}H NMR (400 MHz, DMSO-d_6)



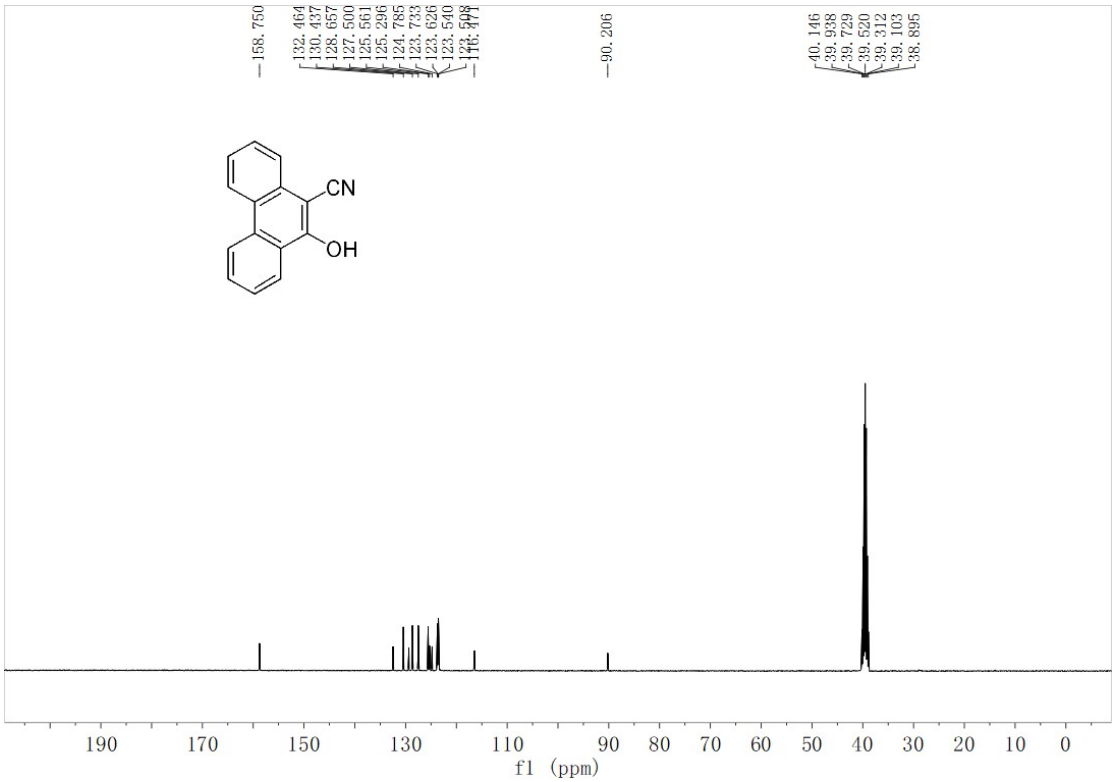
2ad – ^{13}C NMR (101 MHz, DMSO-d_6)



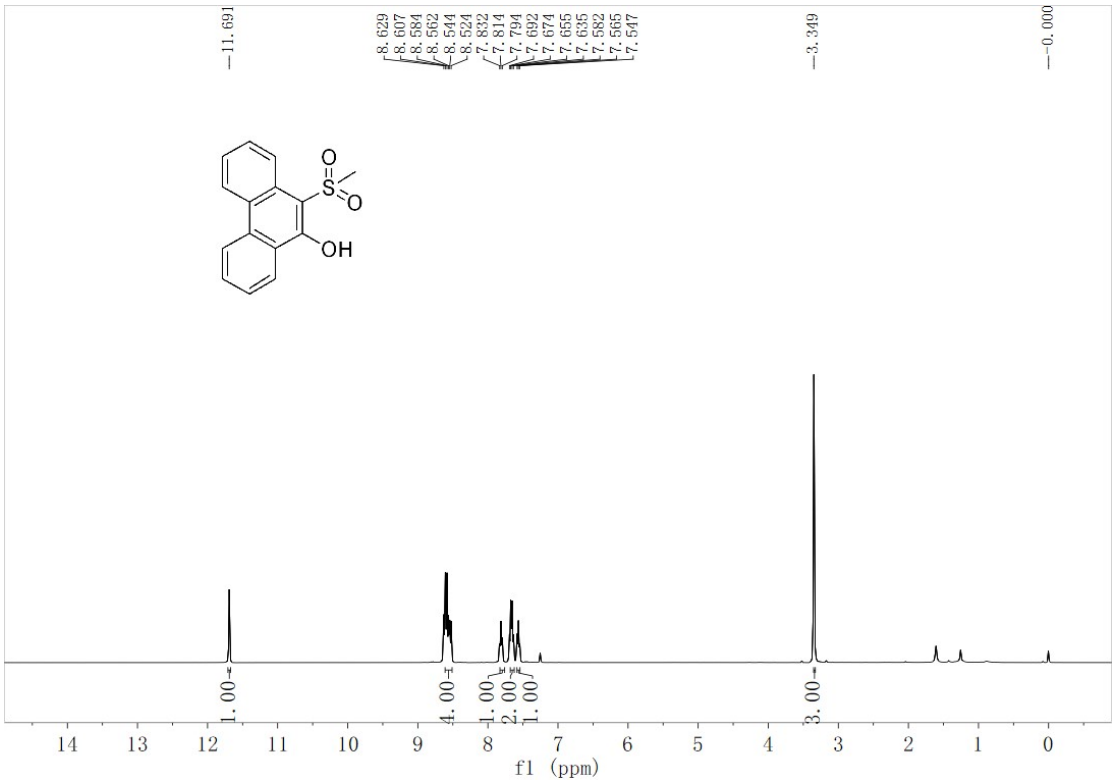
2ae – ^{1}H NMR (400 MHz, DMSO-d_6)



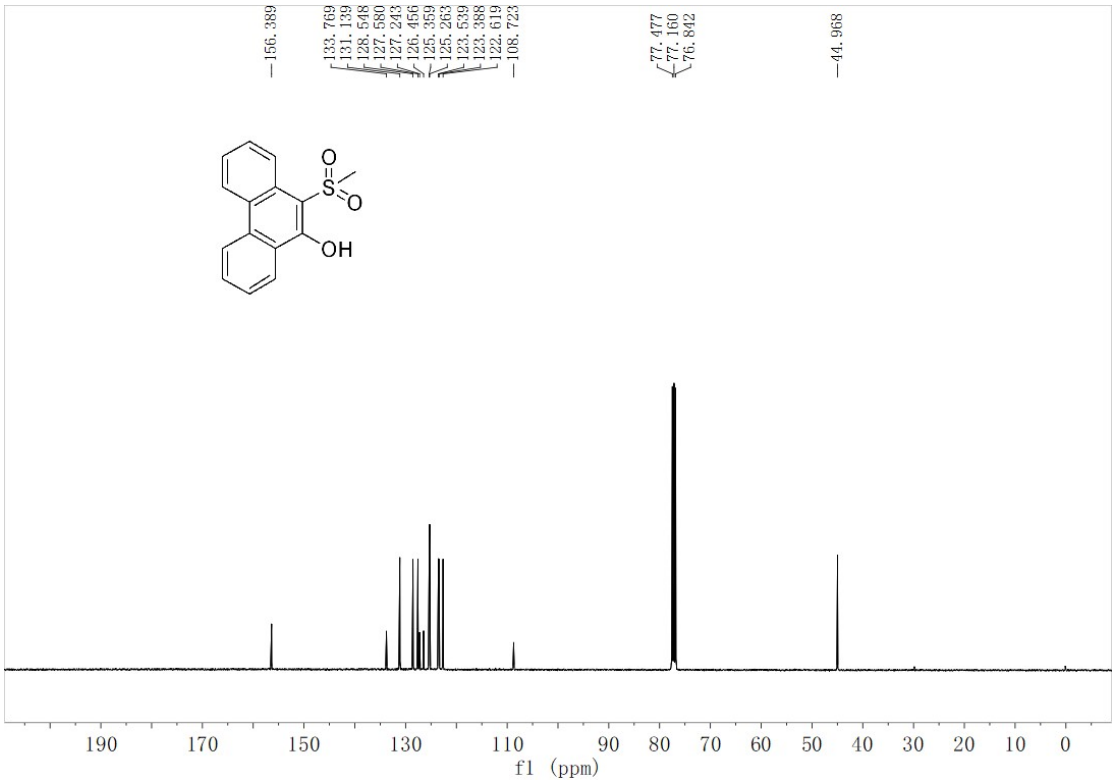
2ae – ^{13}C NMR (101 MHz, DMSO-d_6)



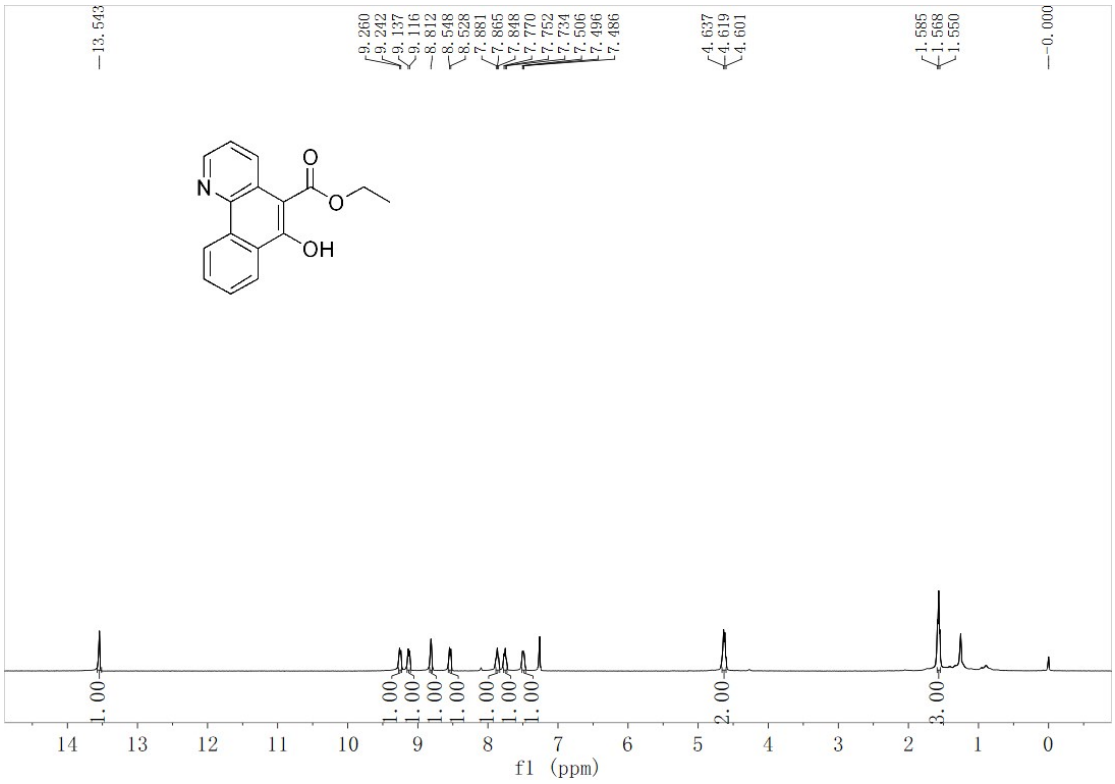
2af – ^{1}H NMR (400 MHz, $CDCl_3$)



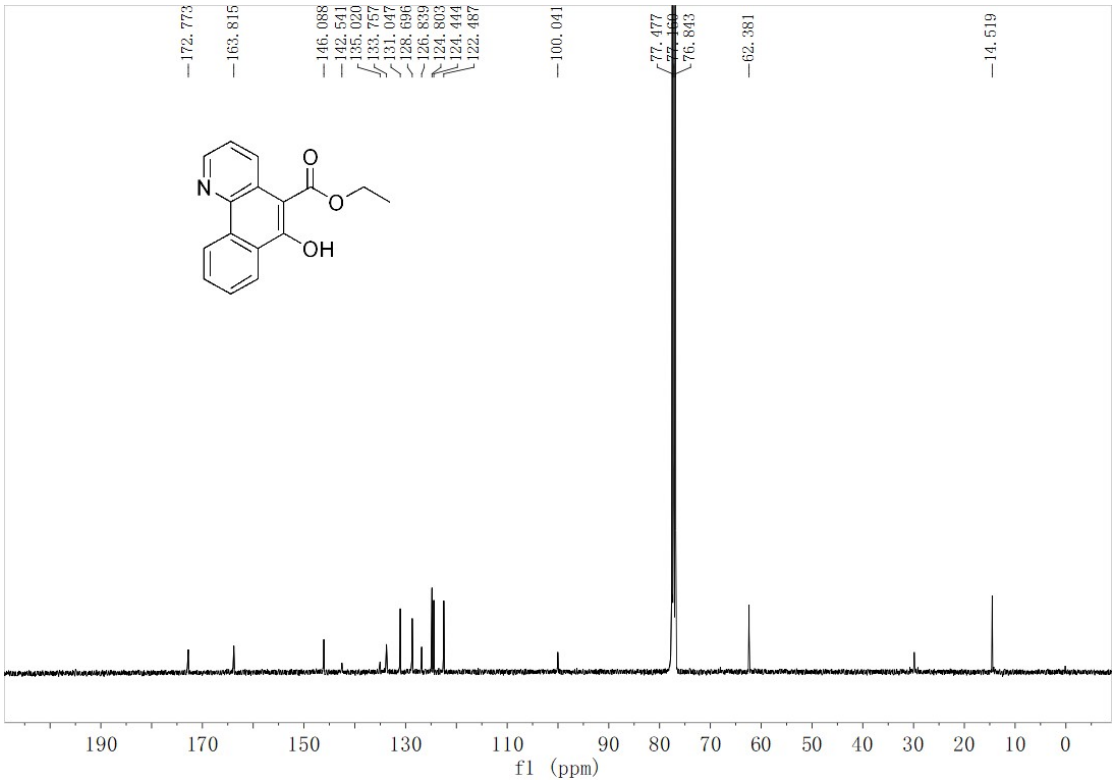
2af – ^{13}C NMR (101 MHz, $CDCl_3$)



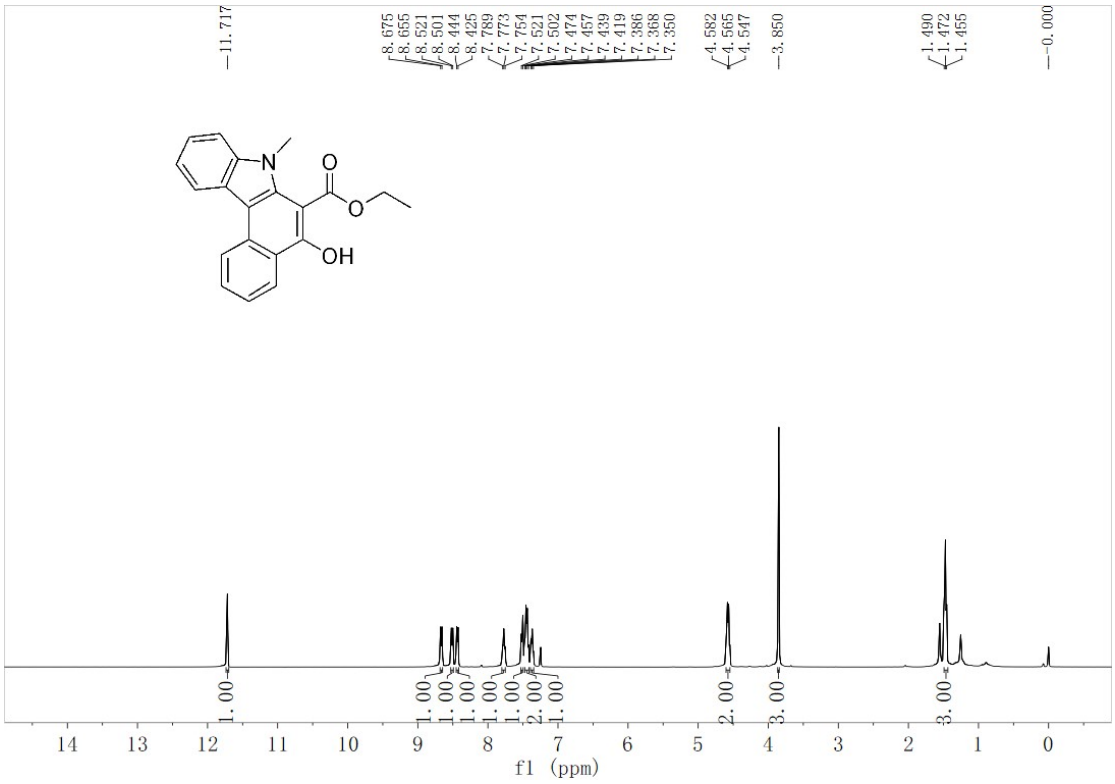
2ag – ^{1}H NMR (400 MHz, $CDCl_3$)



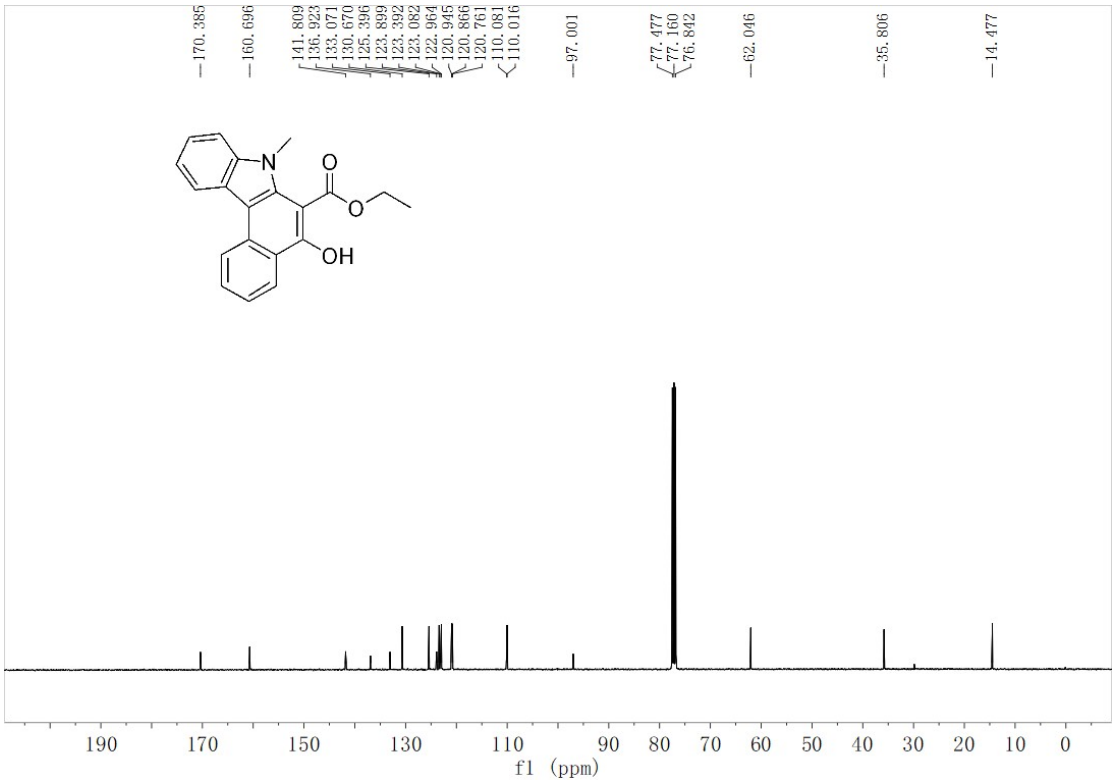
2ag – ^{13}C NMR (101 MHz, $CDCl_3$)



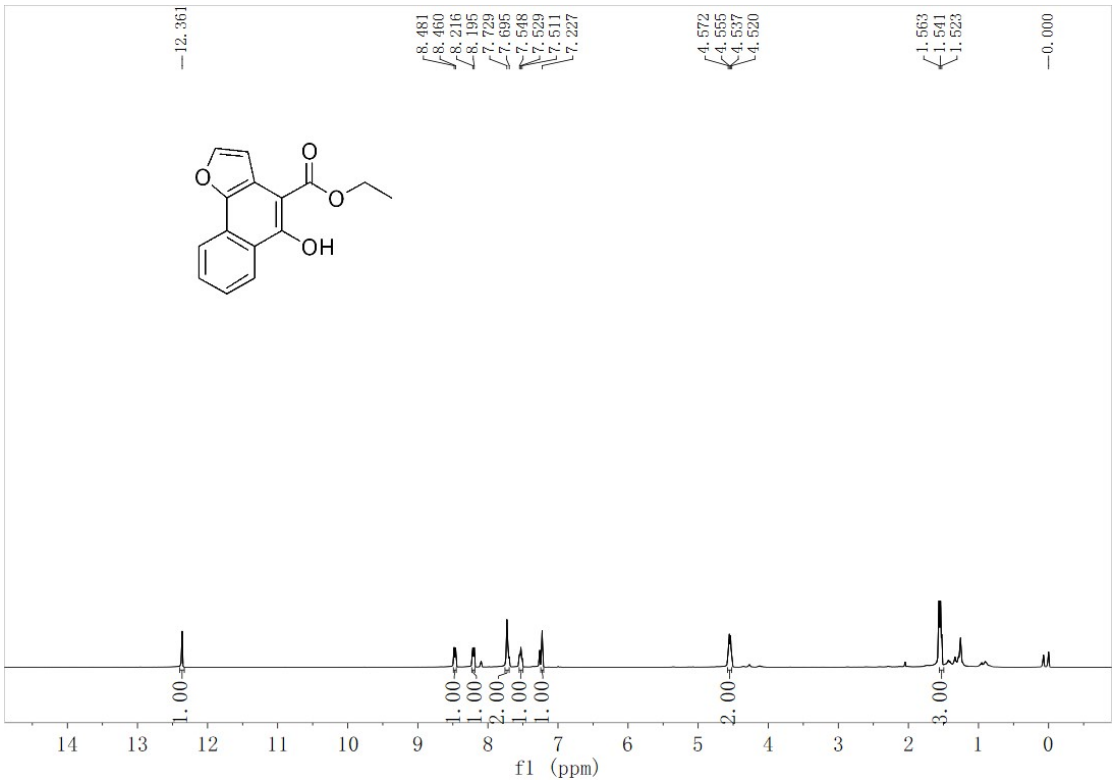
2ah – ^{1}H NMR (400 MHz, $CDCl_3$)



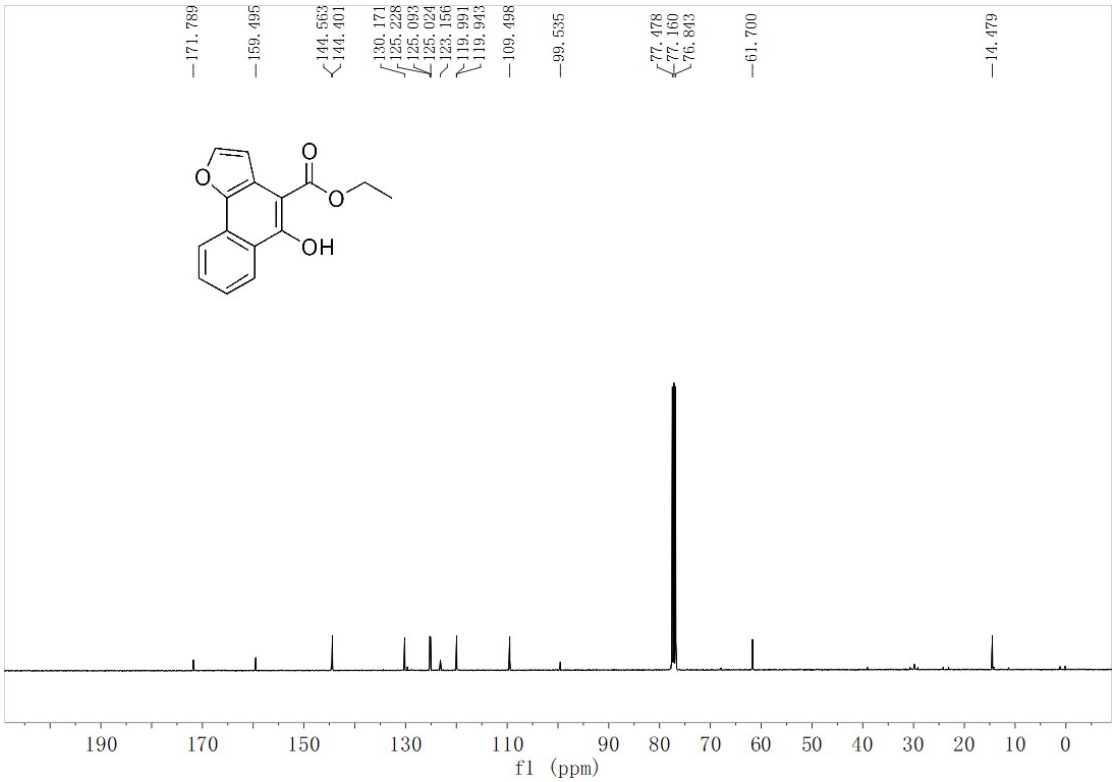
2ah – ^{13}C NMR (101 MHz, $CDCl_3$)



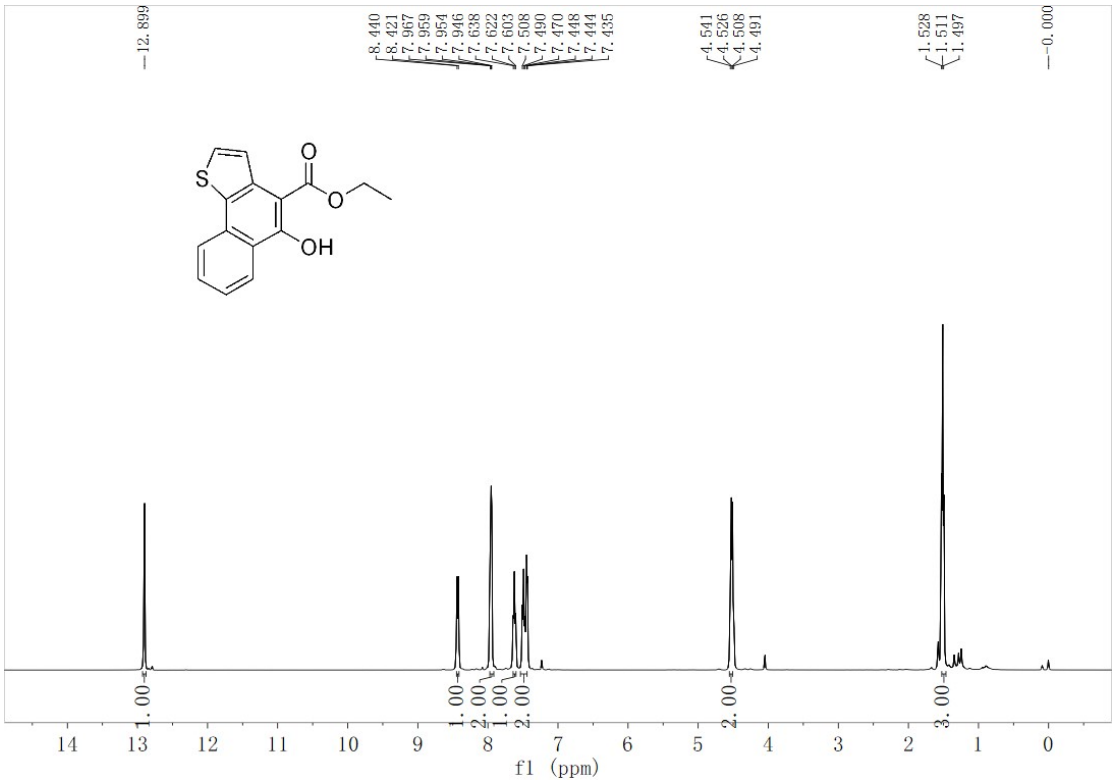
2ai – ^{1}H NMR (400 MHz, $CDCl_3$)



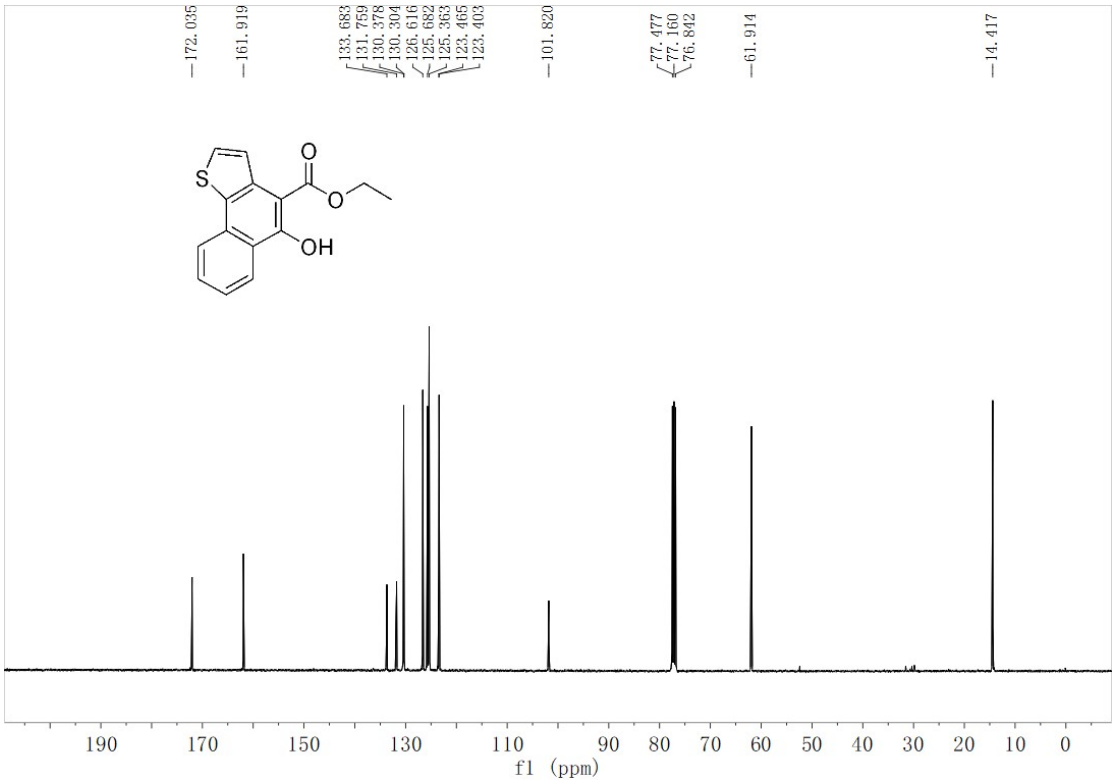
2ai – ^{13}C NMR (101 MHz, $CDCl_3$)



2aj – ^{1}H NMR (400 MHz, $CDCl_3$)



2aj – ^{13}C NMR (101 MHz, $CDCl_3$)



8. References

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