Supporting Information

Visible-light-induced photoredox aerobic coupling of sulfonium ylides and amines leading to *E*-selective formation of 2-amino-2-butene-1,4-diones

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General information

The reactions via general procedure were carried out under an atmosphere of oxygen unless otherwise noted. Column chromatography was performed using silica gel (200-300 mesh). ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on Bruker-AV (400, 100 and 376 MHz, respectively) instrument internally referenced to tetramethylsilane (TMS) or chloroform signals. Mass spectra were measured on Agilent 5977 GC-MS instrument (EI). High-resolution mass spectra (ESI) were obtained with the Thermo Scientific LTQ Orbitrap XL mass spectrometer. Melting points were measured with a YUHUA X-5 melting point instrument and were uncorrected. The structures of known compounds were further corroborated by comparing their ^{1}H NMR, ^{13}C NMR data and HRMS data with those in literature. A commercially available blue LED (35W, HIPAR30, luminous flux is not less than 3200 lm) was purchased from Shenzhen Jing Feng Times Lighting Technology Co., Ltd as the reaction light source. All irradiation reactions were carried out in borosilicate glass vessel. The distance from the light source to the irradiation vessel is around 4-5 cm. Unless otherwise noted, all photocatalysts and other reagents were obtained from commercial suppliers and used without further purification.

General procedure of the reactions

Reaction device diagram: Reaction set-up for irradiation of mixture with 35 W blue LEDs and a fan was used to maintain the reaction temperature at 25-35 °C **(Figure S1).**

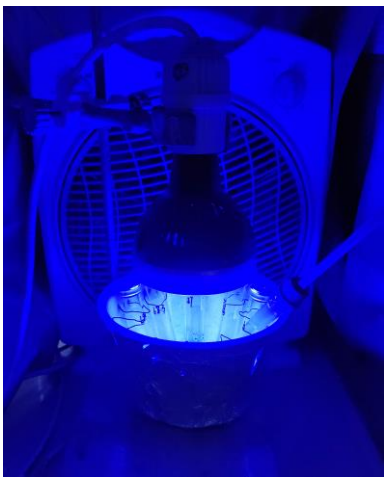
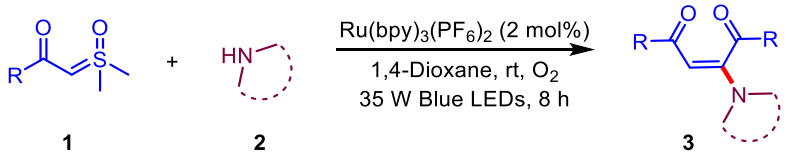


Figure S1. Temperature controled reaction setup (35 W blue LED)



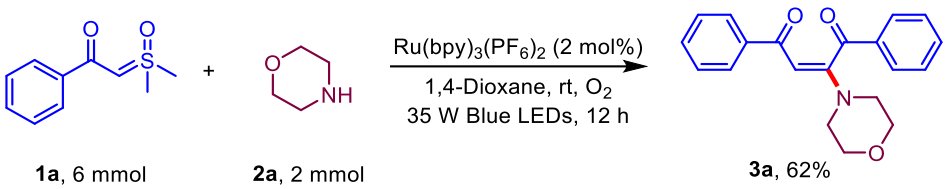
General procedure A: A 10 mL reaction vessel was charged with $Ru(bpy)_3(PF_6)_2$ (3.4 mg, 0.004 mmol, 2 mol%) and sulfoxonium ylides (**1**, 0.6 mmol, 1.5 equiv). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was repeated for three times). Then, amine (**2**, 0.2 mmol, 1.0 equiv) and 1,4-dioxane (2 mL) were added. The resulting mixture was stirred for 8 h under irradiation with a 35 W blue LEDs. After completion, the crude reaction mixture was filtered and the volatiles were removed under reduced pressure. Column chromatography was performed using silica gel (200-300 mesh) to give cross-olefin products **3**.

General procedure B: A 10 mL reaction vessel was charged with $Ru(bpy)_3(PF_6)_2$ (3.4 mg, 0.004 mmol, 2 mol%) and sulfoxonium ylides (**1**, 0.6 mmol, 1.5 equiv). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was repeated for three times). Then, amine (**2**, 0.2 mmol, 1.0 equiv) and DCM (2 mL) were added. The resulting mixture was stirred for 8 h under irradiation with a 35 W blue LEDs. After completion, the crude reaction mixture was filtered and the volatiles were removed under reduced pressure. Column chromatography was performed using silica gel (200-300 mesh) to give cross-olefin products **3**.

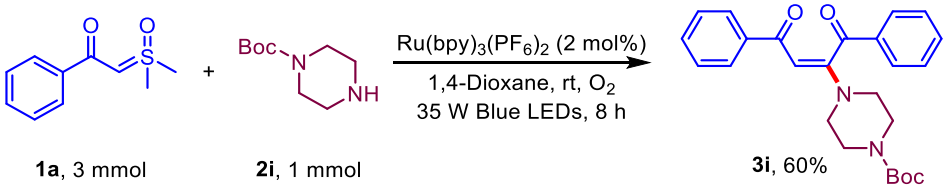
General procedure C: A 10 mL reaction vessel was charged with $Ru(bpy)_3(PF_6)_2$ (3.4 mg, 0.004 mmol, 2 mol%) and sulfoxonium ylides (**1**, 0.6 mmol, 1.5 equiv). The

atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was repeated for three times). Then, amine (**2**, 0.2 mmol, 1.0 equiv) and MeCN (2 mL) were added. The resulting mixture was stirred for 8 h under irradiation with a 35 W blue LEDs. After completion, the crude reaction mixture was filtered and the volatiles were removed under reduced pressure. Column chromatography was performed using silica gel (200-300 mesh) to give cross-olefin products **3**.

Scale-up experiments:



2 mmol scale reaction (3a): A 100 mL reaction vessel was charged with 2-(dimethyl(oxo)-λ^6-sulfaneylidene)-1-phenylethan-1-one (1.17 g, 6 mmol) and $Ru(bpy)_3(PF_6)_2$ (34 mg, 0.004 mmol, 2 mol%), followed by the addition of 1,4-dioxane (20 mL). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was conducted for three times). Then, morpholine (174 μL, 2 mmol) was added. The resulting mixture was stirred for 12 hours under irradiation with a 35 W blue LEDs. The reaction was monitored by TLC. The crude reaction mixture was filtered, and the volatiles were removed under reduced pressure. Column chromatography was performed using silica gel (petroleum ether/ethyl acetate = 3:1) to give product **3a** (0.399 g, 62% yield, *E*/*Z* > 20:1).

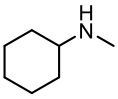


1 mmol scale reaction (3i): A 50 mL reaction vessel was charged with 2-(dimethyl(oxo)-λ^6-sulfaneylidene)-1-phenylethan-1-one (585 mg, 3 mmol), tert-butyl piperazine-1-carboxylate (186 mg, 1 mmol) and $Ru(bpy)_3(PF_6)_2$ (34 mg, 0.004 mmol, 2 mol%), followed by the addition of 1,4-dioxane (20 mL). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was conducted for three times). The resulting mixture was stirred for 8 hours under irradiation with a 35 W blue LEDs. The reaction was monitored by TLC. The crude reaction mixture was filtered, and the volatiles were removed under reduced pressure. Column chromatography was performed using silica gel (petroleum ether/ethyl acetate = 4:1) to give product **3i** (0.253 g, 60% yield, *E*/*Z* > 20:1).

Unsuccessful substrates:

Amines:

complex



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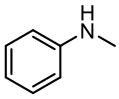

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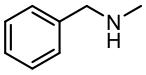

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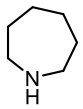

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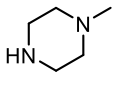

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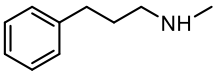

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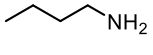

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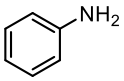

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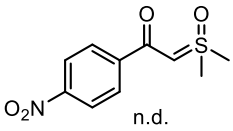
Ylides:



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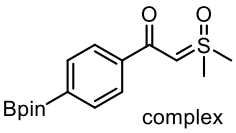

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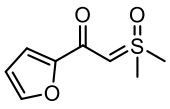

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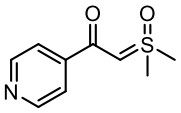

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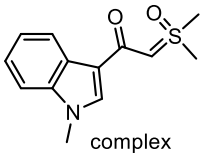

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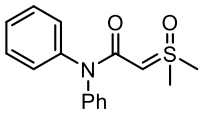

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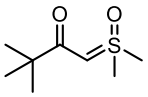

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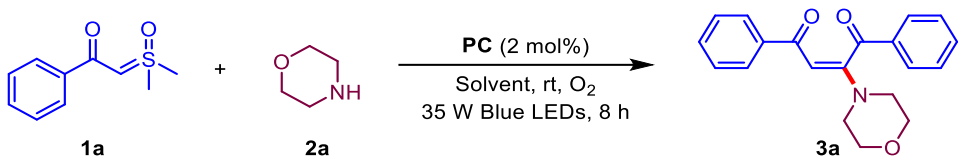
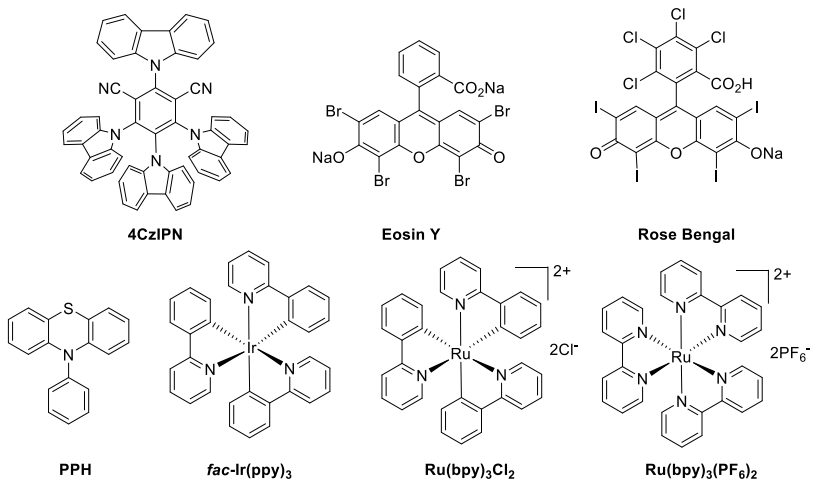
Optimization of reaction conditions[a]

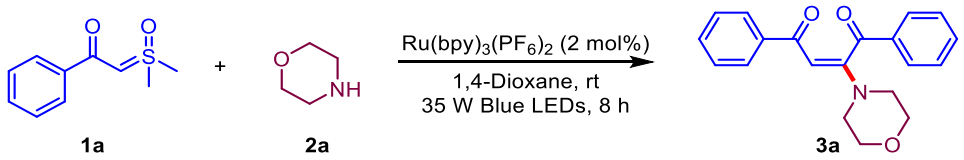


entry	PC	solvent	yield (%)[b]
1	$Ru(bpy)_3(PF_6)_2$	MeCN	62
2	$Ru(bpy)_3(PF_6)_2$	DCM	69
3	$Ru(bpy)_3(PF_6)_2$	Toluene	trace
4	$Ru(bpy)_3(PF_6)_2$	EtOAc	48
5	$Ru(bpy)_3(PF_6)_2$	THF	30
6	$Ru(bpy)_3(PF_6)_2$	DMF	59
7	$Ru(bpy)_3(PF_6)_2$	EA	66
8	$Ru(bpy)_3(PF_6)_2$	1,4-Dioxane	70
9	$Ru(bpy)_3(PF_6)_2$	2-Methylfuran	25
10	4CzIPN	1,4-Dioxane	trace
11	*fac*-$Ir(ppy)_3$	1,4-Dioxane	20
12	Rose bengal	1,4-Dioxane	28
13	Eosin Y	1,4-Dioxane	0
14	$Ru(bpy)_3Cl_2$	1,4-Dioxane	24
15	PPH	1,4-Dioxane	0
16[c]	$Ru(bpy)_3(PF_6)_2$	1,4-Dioxane	35
17[d]	$Ru(bpy)_3(PF_6)_2$	1,4-Dioxane	56
18	-	1,4-Dioxane	0
19[e]	$Ru(bpy)_3(PF_6)_2$	1,4-Dioxane	0
20[e]	-	1,4-Dioxane	0
21[f]	-	1,4-Dioxane	0
22[g]	$Ru(bpy)_3(PF_6)_2$	1,4-Dioxane	0
23[h]	$Ru(bpy)_3(PF_6)_2$	1,4-Dioxane	trace
24[i]	$Ru(bpy)_3(PF_6)_2$	1,4-Dioxane	0



[a] Reaction conditions: **1a** (0.6 mmol, 3.0 equiv), **2a** (0.2 mmol, 1.0 equiv) and photocatalyst (PC, 0.004, 2 mol %) in solvent (2 mL) at room temperature (25-30 °C) under O_2 atmosphere and 35 W blue LEDs irradiation for 8 h. [b] Yield of isolated product. [c] **1a** (0.4 mmol, 1.0 equiv) and **2a** (0.2 mmol, 1.0 equiv) were used. [d] **1a** (0.48 mmol, 1.2 equiv) and **2a** (0.2 mmol, 1.0 equiv) were used. [e] No light. [f] No light and Air atmosphere. [g] Under Ar atmosphere. [h] Under Air atmosphere. [i] Under Ar atmosphere with 1.5 equiv. TBHP.

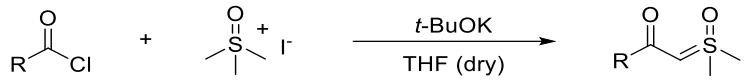
Screening of dioxygen concentration



entry	oxygen concentration	yield (%)
1	O_2 (2 mL)	32
2	O_2 (4 mL)	51
3	O_2 (8 mL)	67
4	O_2/N_2 (3:1)	17
5	O_2/N_2 (2:1)	12
6	O_2/N_2 (1:1)	<5

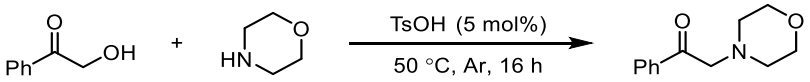
Procedures for the preparation of the corresponding raw materials

General procedure for preparation of sulfoxonium ylides [1]:



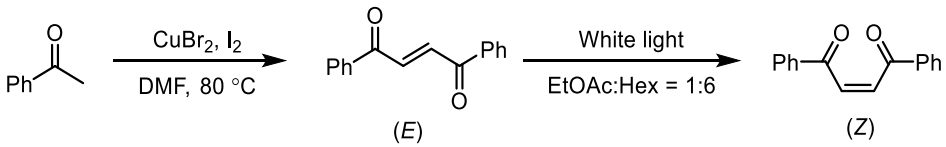
To a stirred solution of potassium tert-butoxide (3.0 g, 27.2 mmol) in THF (dry, 30 mL) was added trimethylsulfoxonium iodide (5.0 g, 20.6 mmol) at rt. The resulting mixture is refluxed for 2 h. The reaction mixture is then cooled to 0 ºC, followed by addition of acyl chlorides (7 mmol) in THF (7 mL). The reaction was allowed to rt and stirred for 3 h. Next, the solvent was evaporated and water (15 mL) and dichloromethane (20 mL) were added to the resulting slurry. The layers were separated and the aqueous layer was washed with dichloromethane (3×50 mL) and the organic layers were combined. The organic solution was dried over anhydrous sodium sulphate (Na_2SO_4), filtered over a sintered funnel and evaporated to dryness. The crude product was purified by flash chromatography over silica gel using DCM/MeOH (95:5) to afford the corresponding sulfoxonium ylides.

General procedure for preparation of 2-morpholino-1-phenylethan-1-one [2]:



2-Hydroxyacetophenone (0.2 mmol) and morpholine (0.2 mmol) were added in a reaction vessel followed by catalytic TsOH (0.005 mmol). The reaction mixture was stirred under nitrogen atmosphere overnight at 50 ºC. After full conversion of the starting material, the reaction mixture was cooled down to room temperature and purified by silica gel flash column directly (eluent: Petroleum ether/ EtOAc = 3:1).

General procedure for preparation of (*Z*)-1,4-diphenylbut-2-ene-1,4-dione [3]:



To a sealed tube (15 mL) was added acetophenone (0.5 mmol), $CuBr_2$ (0.1 mmol), I_2 (1 mmol) and dry DMF (0.5 mL). The mixture was heated to 80 ºC and reacted at the same temperature for 20 h. After the reaction was finished, saturated $Na_2S_2O_4$ solvent (10 mL) was added. The resulting mixture was extracted with ethyl acetate (3 × 10 mL). The combined organic phase was dried with Na_2SO_4 and evaporated in vacuum. Purification of the residue by flash column chromatography (Petroleum ether/ EtOAc = 1:15- 1:10) afforded the desired *E*-1,4-enedione.

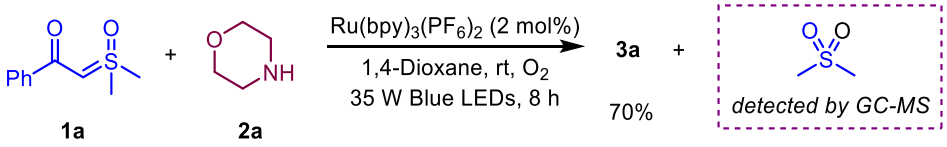
To a tube (10 mL) was added *E*-1,4-enedione (0.3 mmol) and solvent (2.1 mL, EtOAc/ Hex = 1:6), then the mixture was irradiated by white light (white light, 23 W) for 5 h.

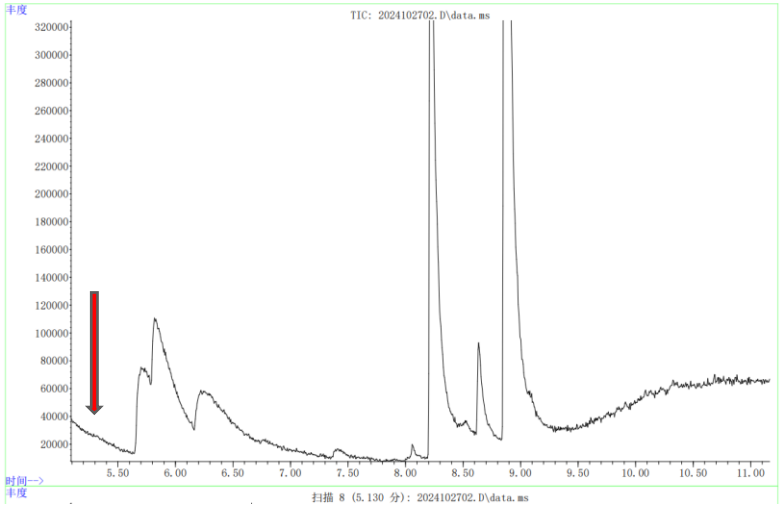
After the reaction was finished, the solvent was evaporated in vacuum. Purification of the residue by column chromatography (Al_2O_3, Petroleum ether/ EtOAc = 1:3) afforded the *Z*-1,4-enedione as a white solid.

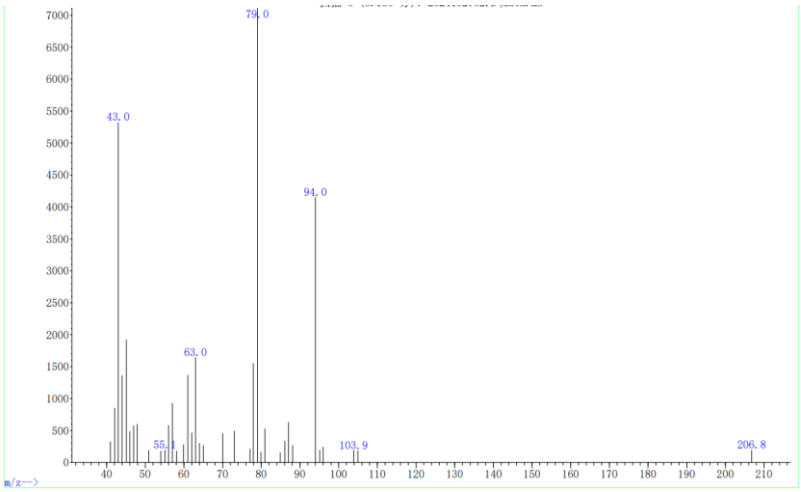
Mechanistic studies

Control experiments

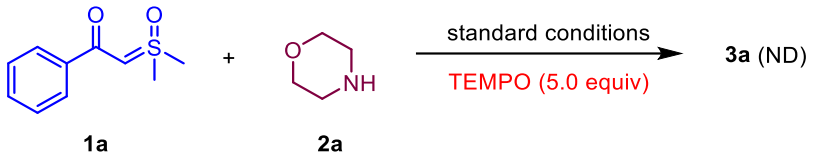
(a) The following reaction was carried out under standard conditions: A 10 mL reaction vessel was charged with $Ru(bpy)_3(PF_6)_2$ (3.4 mg, 0.004 mmol), **1a** (118 mg, 0.6 mmol), 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (156 mg, 1 mmol, 5.0 equiv) and 1,4-dioxane (2 mL). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was conducted for three times). Then, **2a** (17 μL, 0.2 mmol) was added. The resulting mixture was stirred for 8 h under irradiation with a 35 W blue LEDs. After completion, the crude reaction mixture was filtered and the volatiles were removed under reduced pressure. The crude residues were analyzed by GC-MS. Dimethyl sulfone was detected as a byproduct.



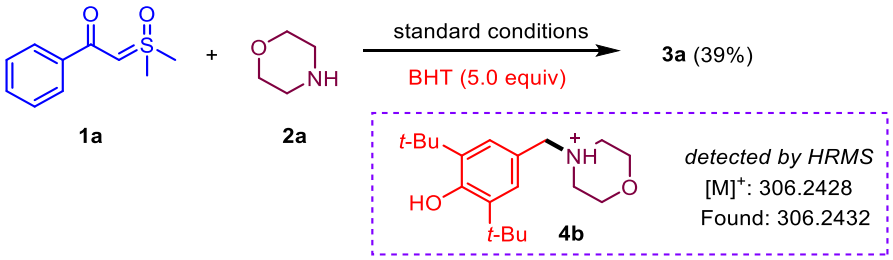


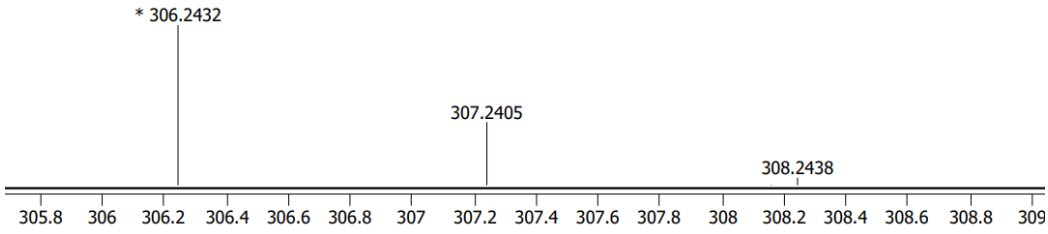


(b) The following reaction was carried out under standard conditions: A 10 mL reaction vessel was charged with $Ru(bpy)_3(PF_6)_2$ (3.4 mg, 0.004 mmol), **1a** (118 mg, 0.6 mmol), 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (156 mg, 1 mmol, 5.0 equiv) and 1,4-dioxane (2 mL). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was conducted for three times). Then, **2a** (17 μL, 0.2 mmol) was added. The resulting mixture was stirred for 8 h under irradiation with a 35 W blue LEDs. After completion, the crude reaction mixture was filtered and the volatiles were removed under reduced pressure. The crude residues were analyzed by GC-MS and HRMS. The formation of product **3a** was completely suppressed.



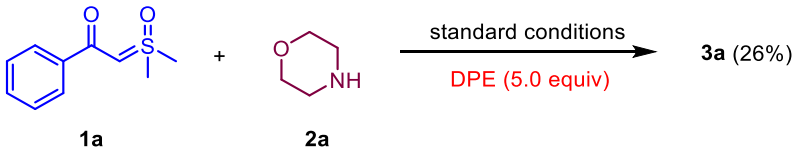
(c) The following reaction was carried out under standard conditions: A 10 mL reaction vessel was charged with $Ru(bpy)_3(PF_6)_2$ (3.4 mg, 0.004 mmol), **1a** (118 mg, 0.6 mmol), hydroxytoluene (BHT) (220 mg, 1 mmol, 5.0 equiv) and 1,4-dioxane (2 mL). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was conducted for three times). Then, **2a** (17 μL, 0.2 mmol) was added. The resulting mixture was stirred for 8 h under irradiation with a 35 W blue LEDs. After completion, the crude reaction mixture was filtered and the volatiles were removed under reduced pressure. The crude residues were analyzed by GC-MS and HRMS. BHT-trapped amination adduct **4b** was detected by HRMS and the formation of product **3a** was reduced to 39%.



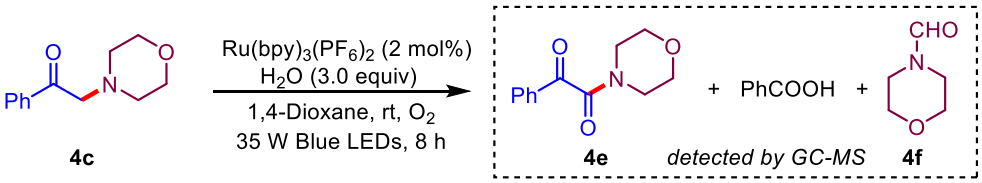


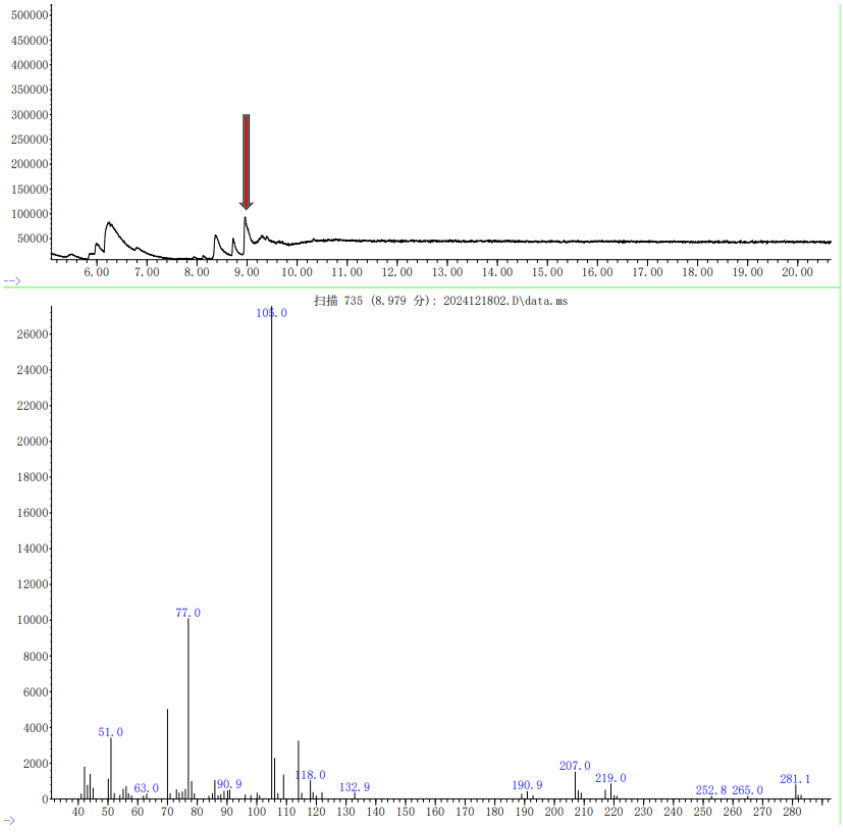
(d) The following reaction was carried out under standard conditions: A 10 mL reaction vessel was charged with $Ru(bpy)_3(PF_6)_2$ (3.4 mg, 0.004 mmol), **1a** (118 mg, 0.6 mmol), 1,1-Diphenylethylene (DPE) (177 μL, 1 mmol, 5.0 equiv) and 1,4-dioxane (2 mL). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was conducted for three times). Then, **2a** (17 μL, 0.2 mmol) was added. The resulting mixture was stirred for 8 h under irradiation with a 35 W blue LEDs.

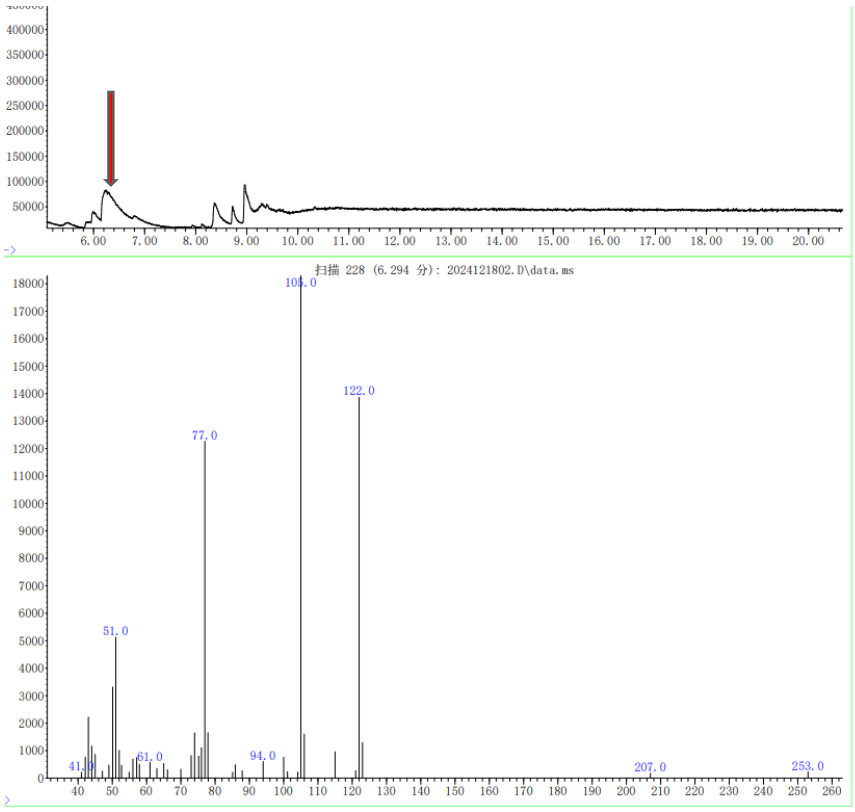
After completion, the crude reaction mixture was filtered and the volatiles were removed under reduced pressure. The crude residues were analyzed by GC-MS and HRMS. The formation of product **3a** was reduced to 28%.

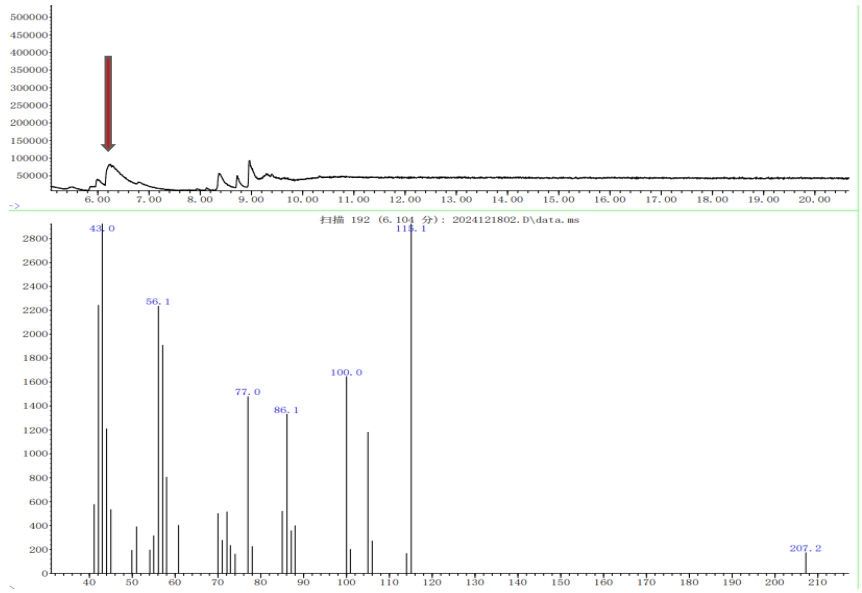


(d) The following reaction was carried out under standard conditions: A 10 mL reaction vessel was charged with $Ru(bpy)_3(PF_6)_2$ (3.4 mg, 0.004 mmol), 2-morpholino-1-phenylethan-1-one **4c** (41 mg, 0.2 mmol) and 1,4-dioxane (2 mL). The atmosphere was exchanged by applying vacuum and backfilling with O_2 (this process was conducted for three times). Then, H_2O (11 μL, 0.6 mmol) was added. The resulting mixture was stirred for 8 h under irradiation with a 35 W blue LEDs. After completion, the crude reaction mixture was filtered and the volatiles were removed under reduced pressure. The crude residues were analyzed by GC-MS. 1-Morpholino-2-phenylethane-1,2-dione **4e**, benzoic acid, and morpholine-4-carbaldehyde **4f** were detected by GC-MS.







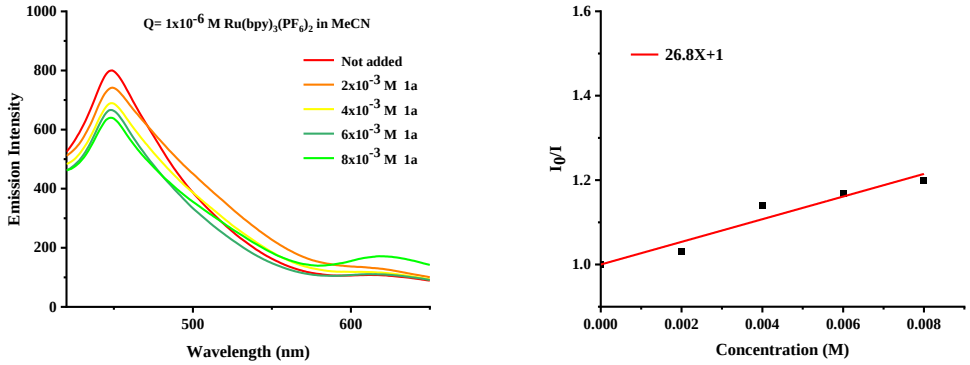


Stern–Volmer Quenching

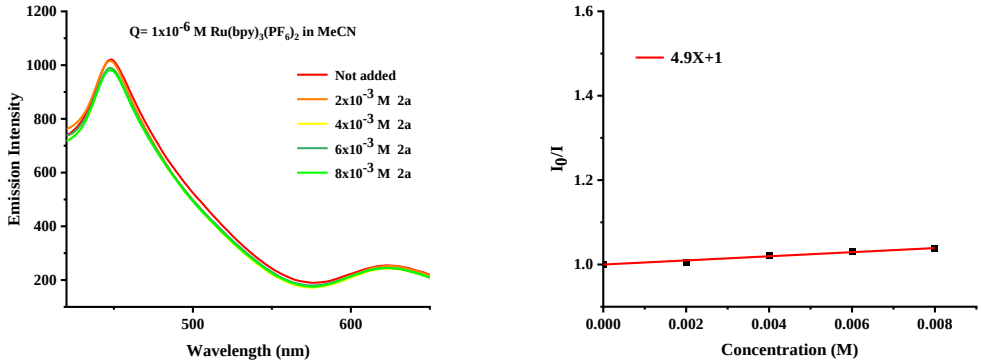
Formulation solution: Morpholine (**2a**, 1 mmol, 87 μL) was dissolved in MeCN in a 5 mL volumetric flask to set the concentration to be 0.2 M. 2-(dimethyl(oxo)-λ^6-sulfaneylidene)-1-phenylethan-1-one (**1a**, 1 mmol, 196 mg) was dissolved in MeCN in a 5 mL volumetric flask to set the concentration to be 0.2 M. Photocatalyst $Ru(bpy)_3(PF_6)_2$ (1.8 mg) was dissolved in MeCN (25.0 mL) to set the concentration to

be 0.1 mM.

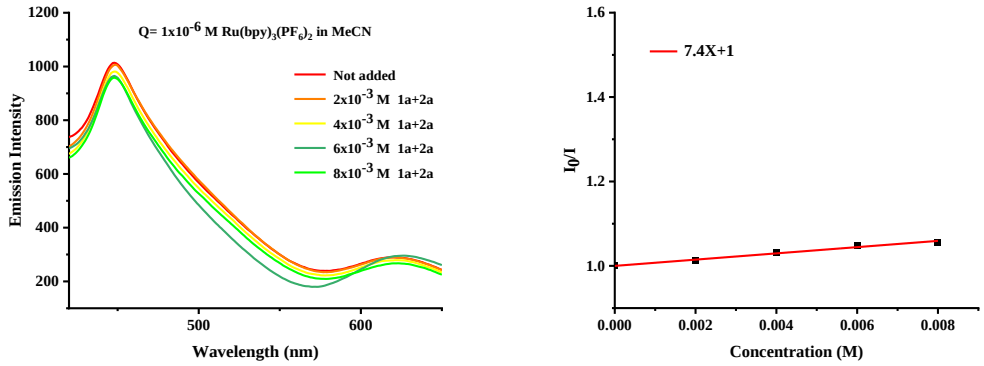
Experimental procedure A: The resulting 0.1 mM solution (20 μL) was added to cuvette to obtain different concentrations of catalyst solution. This solution was then diluted to a volume of 2.0 mL by adding MeCN to prepare a 1.0 μM solution. The resulting mixture was sparged with oxygen for 2 minutes and then irradiated at 390 nm. Fluorescence emission spectra were recorded (3 trials per sample). Into this solution, 20.0 μL of a 2-(dimethyl(oxo)-λ^6-sulfaneylidene)-1-phenylethan-1-one solution was successively added and uniformly stirred, and the resulting mixture was bubbled with oxygen for 2 minutes and irradiated at 390 nm. Fluorescence emission spectra of 0 μL, 20.0 μL, 40.0 μL, 60.0 μL, 80.0 μL fluorescence intensity. Follow this method and make changes to the amount to obtain the Stern–Volmer relationship in turn. The results were shown in the following figures.



Emission quenching of $Ru(bpy)_3(PF_6)_2$ with 2-(dimethyl(oxo)-λ^6-sulfaneylidene)-1-phenylethan-1-one (**1a**) in MeCN

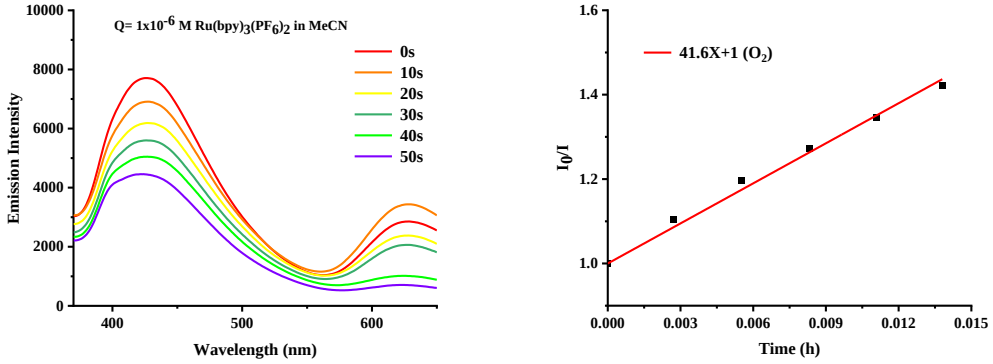


Emission quenching of $Ru(bpy)_3(PF_6)_2$ with morpholine (**2a**) in MeCN



Emission quenching of $Ru(bpy)_3(PF_6)_2$ with 2-(dimethyl(oxo)-λ^6-sulfaneylidene)-1-phenylethan-1-one (**1a**) and morpholine (**2a**) in MeCN

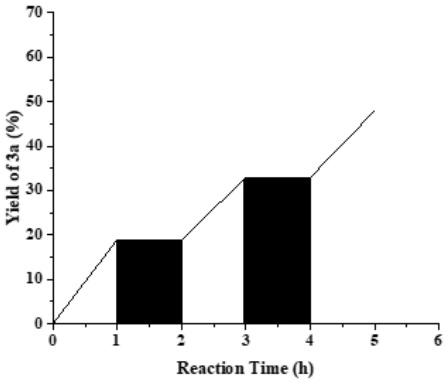
Experimental procedure B: The resulting 0.1 mM solution (20 μL) was added to cuvette to obtain different concentrations of catalyst solution. This solution was then diluted to a volume of 2.0 mL by adding MeCN to prepare a 1.0 μM solution. The resulting mixture was sparged with argon for 2 minutes and then irradiated at 350 nm. Fluorescence emission spectra were recorded (3 trials per sample). The resulting mixture was bubbled with oxygen for 10s and irradiated at 350 nm. Fluorescence emission spectra of 0s, 10s, 20s, 30s, 40s, 50s fluorescence intensity. Follow this method and make changes to the amount to obtain the Stern–Volmer relationship in turn. The results were shown in the following figures.



The emission intensity of the $Ru(bpy)_3(PF_6)_2$ catalyst solution affected by the gradual increase of the amount of O_2

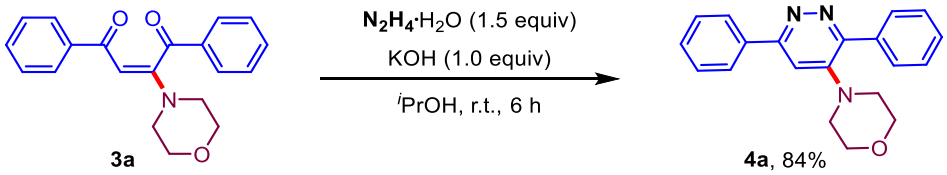
Light on/off experiment

Conducted the relationship of products with light on-off under standard conditions. Subsequent samples (each 20 μL) taken at regular time intervals and determined by GC with dodecane as the internal standard.



Plot of light on-off experiments

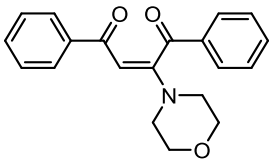
Late-stage derivation and application



To a stirred solution of the product *E*-**3a** (64.2 mg, 0.2 mmol, 1.0 equiv) and KOH (11.2 mg, 0.2 mmol, 1.0 equiv) in ^{i}PrOH (1 mL) at room temperature was dropwise added $N_2H_4{\cdot}H_2O$ (0.3 mmol, 1.5 equiv).[4] The resulting solution was stirred for 2 h. The reaction was quenched with water (10 mL), and the aqueous phase was extracted with EtOAc (3 x 10 mL). The combined organic layers were concentrated, and the residue was purified by flash chromatography (petroleum ether/ethyl acetate = 3:1) to afford the desired product **4a** (53.3 mg, 84% yield) as yellow liquid.

Characterization data of all products

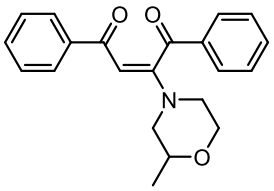
(*E*)-2-morpholino-1,4-diphenylbut-2-ene-1,4-dione (3a)[5]



Following the general procedure A, **3a** (70%, 45.0 mg, *E*/*Z* > 20:1) as a white solid was obtained after flash chromatography (Petroleum ether/ EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 7.99 (m, 2H), 7.89 – 7.81 (m, 2H), 7.60 – 7.54 (m, 1H), 7.52 – 7.42 (m, 3H), 7.40 – 7.32 (m, 2H), 6.16 (s, 1H), 3.87 – 3.62 (m, 4H), 3.51 – 3.22 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 187.1, 160.9, 138.7, 135.6, 133.5, 131.6, 128.9, 128.1, 128.0, 127.7, 93.3, 66.1, 47.5.

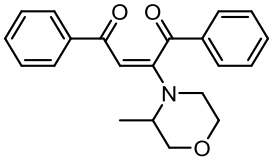
(*E*)-2-(2-methylmorpholino)-1,4-diphenylbut-2-ene-1,4-dione (3b)



Following the general procedure A, **3b** (66%, 44.2 mg, *E*/*Z* > 20:1) as a yellow solid (mp 114-116 ºC) was obtained after flash chromatography (Petroleum ether/ EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.1 Hz, 2H), 7.84 (d, *J* = 6.9 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.52 – 7.40 (m, 3H), 7.37 (t, *J* = 7.6 Hz, 2H), 6.15 (s, 1H), 3.98 – 2.71 (m, 7H), 1.16 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.0, 187.1, 160.8, 138.8, 135.7, 133.5, 131.6, 128.9, 128.1, 128.0, 127.7, 93.3, 71.5, 65.8, 53.2, 46.8, 18.6. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{21}H_{21}NNaO_3^+$ 358.1414; Found 358.1406.

(*E*)-2-(3-methylmorpholino)-1,4-diphenylbut-2-ene-1,4-dione (3c)

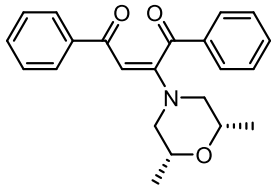


Following the general procedure C, **3c** (43%, 28.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 180-182 ºC) was obtained after flash chromatography (Petroleum ether/ EtOAc = 4:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.86 – 7.78 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.51 – 7.39 (m, 3H), 7.36 (t, *J* = 7.5 Hz, 2H), 6.10 (s, 1H), 3.96 – 3.07 (m, 7H), 1.41 (d, *J* = 37.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.5, 187.1, 171.1,

160.8, 160.5, 138.9, 135.7, 133.4, 131.5, 128.9, 128.2, 127.9, 127.7, 93.0, 92.7, 70.6, 66.4, 60.4, 51.2, 50.5, 43.0, 42.5, 21.0, 14.2. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{21}H_{21}NNaO_3^+$ 358.1414; Found 358.1408.

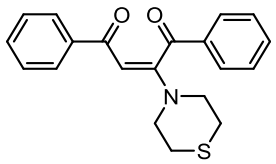
(*E*)-2-((2*R*,6*S*)-2,6-dimethylmorpholino)-1,4-diphenylbut-2-ene-1,4-dione (3d)



Following the general procedure A, **3d** (59%, 41.2 mg, *E*/*Z* > 20:1) as a white solid (mp 195-197 ºC) was obtained after flash chromatography (Petroleum ether/ EtOAc = 4:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.6 Hz, 2H), 7.84 (d, *J* = 7.6 Hz, 2H), 7.60 – 7.30 (m, 6H), 6.15 (s, 1H), 3.88 – 3.14 (m, 4H), 2.91 – 2.54 (m, 2H), 1.16 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.0, 187.0, 160.6, 138.8, 135.6, 133.4, 131.5, 128.9, 128.1, 128.0, 127.6, 93.2, 71.2, 52.5, 18.6. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{22}H_{23}NNaO_3^+$ 372.1570; Found 372.1573.

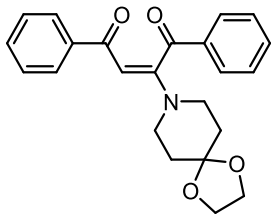
(*E*)-1,4-diphenyl-2-thiomorpholinobut-2-ene-1,4-dione (3e)



Following the general procedure A, **3e** (52%, 35.1 mg, *E*/*Z* > 20:1) as a white solid (mp 96-98 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.0 Hz, 2H), 7.83 (d, *J* = 7.0 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.51 – 7.40 (m, 3H), 7.36 (t, *J* = 7.5 Hz, 2H), 6.11 (s, 1H), 3.68 (s, 4H), 2.88 – 2.44 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 187.0, 159.9, 138.8, 135.5, 133.5, 131.5, 128.9, 128.1, 128.0, 127.6, 93.1, 50.9, 26.6. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{20}H_{19}NNaO_2S^+$ 360.1029; Found 360.1017.

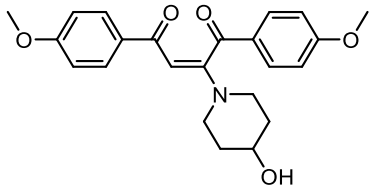
(*E*)-1,4-diphenyl-2-(1,4-dioxa-8-azaspiro[4.5]decan-8-yl)but-2-ene-1,4-dione (3f)



Following the general procedure A, **3f** (46%, 34.7 mg, *E*/*Z* > 20:1) as a yellow solid (mp 146-148 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 6.9 Hz, 2H), 7.84 (d, *J* = 7.0 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.51 – 7.40 (m, 3H), 7.36 (t, *J* = 7.6 Hz, 2H), 6.16 (s, 1H), 3.95 (s, 4H), 3.49 (s, 4H), 1.78 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.0, 187.0, 160.6, 139.0, 135.7, 133.3, 131.3, 128.9, 128.1, 128.0, 127.6, 106.1, 92.7, 64.5, 46.0, 34.7. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{23}H_{23}NNaO_4^+$ 400.1519; Found 400.1511.

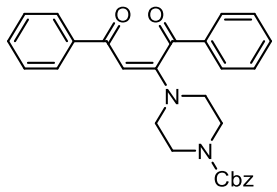
(*E*)-2-(4-hydroxypiperidin-1-yl)-1,4-bis(4-methoxyphenyl)but-2-ene-1,4-dione (3g)



Following the general procedure A, **3g** (43%, 34.0 mg, *E*/*Z* > 20:1) as a white solid (mp 124-126 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.82 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.08 (s, 1H), 3.95 – 3.86 (m, 1H), 3.82 (d, *J* = 9.4 Hz, 6H), 3.70 – 3.43 (m, 2H), 3.17 (s, 2H), 2.46 (s, 1H), 1.86 (s, 2H), 1.71 – 1.46 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.0, 185.9, 163.7, 162.1, 160.4, 131.9, 130.4, 129.6, 129.1, 114.1, 113.2, 92.0, 65.8, 55.4, 55.3, 44.9, 33.6. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{26}NO_5^+$ 396.1805; Found 396.1796.

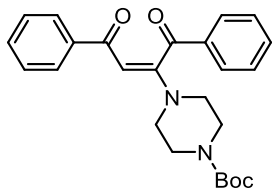
benzyl (*E*)-4-(1,4-dioxo-1,4-diphenylbut-2-en-2-yl)piperazine-1-carboxylate (3h)



Following the general procedure A, **3h** (67%, 60.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 75-77 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 7.2 Hz, 2H), 7.84 (d, *J* = 7.2 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.49 – 7.41 (m, 3H), 7.39 – 7.29 (m, 7H), 6.15 (s, 1H), 5.13 (s, 2H), 3.71 – 3.23 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 187.1, 160.5, 154.9, 138.6, 136.0, 135.5, 133.6, 131.6, 128.9, 128.5, 128.2, 128.1, 128.0, 127.6, 93.5, 67.5, 47.0, 42.8. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{28}H_{26}N_2NaO_4^+$ 477.1785; Found 477.1787.

***tert*-butyl (*E*)-4-(1,4-dioxo-1,4-diphenylbut-2-en-2-yl)piperazine-1-carboxylate (3i)**

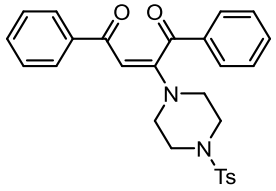


Following the general procedure A, **3i** (73%, 61.4 mg, *E*/*Z* > 20:1) as a white solid (mp

101-103 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 4:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.2 Hz, 2H), 7.84 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.50 – 7.41 (m, 3H), 7.37 (t, *J* = 7.5 Hz, 2H), 6.15 (s, 1H), 3.61 – 3.23 (m, 8H), 1.45 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 187.0, 160.6, 154.3, 138.7, 135.6, 133.5, 131.5, 128.9, 128.1, 128.0, 127.6, 93.3, 80.5, 47.1, 28.2. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{25}H_{28}N_2NaO_4^+$ 443.1941; Found 443.1937.

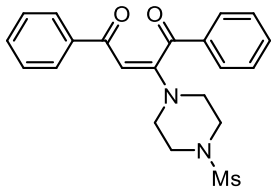
(*E*)-1,4-diphenyl-2-(4-tosylpiperazin-1-yl)but-2-ene-1,4-dione (3j)



Following the general procedure A, **3j** (70%, 66.4 mg, *E*/*Z* > 20:1) as a white solid (mp 246-248 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 4:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.2 Hz, 2H), 7.79 (d, *J* = 7.1 Hz, 2H), 7.61 – 7.50 (m, 3H), 7.47 – 7.39 (m, 3H), 7.37 – 7.30 (m, 4H), 6.12 (s, 1H), 3.43 (d, *J* = 5.7 Hz, 4H), 3.05 (d, *J* = 18.6 Hz, 4H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 187.1, 160.0, 144.4, 138.5, 135.4, 133.6, 131.8, 131.7, 129.9, 128.9, 128.2, 127.9, 127.64, 127.62, 94.0, 47.0, 45.4, 21.5. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{27}H_{26}N_2NaO_4S^+$ 497.1505; Found 497.1493.

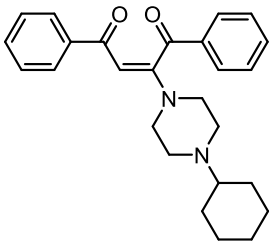
(*E*)-2-(4-(methylsulfonyl)piperazin-1-yl)-1,4-diphenylbut-2-ene-1,4-dione (3k)



Following the general procedure A, **3k** (74%, 59.0 mg, *E*/*Z* > 20:1) as a white solid (mp 219-221 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 1:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 7.2 Hz, 2H), 7.84 (d, *J* = 7.2 Hz, 2H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.52 – 7.41 (m, 3H), 7.41 – 7.33 (m, 2H), 6.21 (s, 1H), 3.47 (t, *J* = 5.1 Hz, 4H), 3.29 (d, *J* = 16.4 Hz, 4H), 2.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 187.2, 160.2, 138.4, 135.4, 133.7, 131.8, 129.0, 128.2, 128.0, 127.7, 94.3, 47.2, 45.0, 34.9. ^{19}F NMR (376 MHz, $CDCl_3$) δ -119.1. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{21}H_{22}N_2NaO_4S^+$ 421.1192; Found 421.1204.

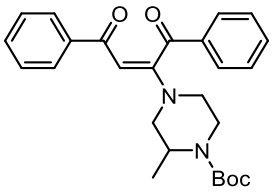
(*E*)-2-(4-cyclohexylpiperazin-1-yl)-1,4-diphenylbut-2-ene-1,4-dione (3l)



Following the general procedure A, **3l** (47%, 37.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 190-192 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.2 Hz, 2H), 7.84 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.50 – 7.40 (m, 3H), 7.39 – 7.32 (m, 2H), 6.12 (s, 1H), 3.39 (d, *J* = 42.0 Hz, 4H), 2.63 (s, 4H), 2.34 – 2.21 (m, 1H), 1.87 – 1.75 (m, 4H), 1.62 (d, *J* = 12.5 Hz, 1H), 1.30 – 1.05 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.1, 186.9, 161.0, 139.0, 135.8, 133.4, 131.4, 128.9, 128.08, 128.05, 127.7, 92.7, 63.4, 48.5, 28.8, 26.1, 25.7. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{26}H_{31}N_2O_2^+$ 403.2380; Found 403.2381.

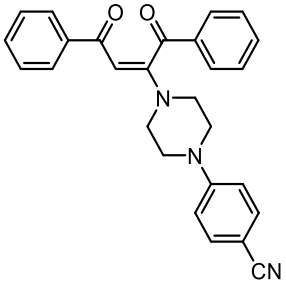
***tert*-butyl (*E*)-4-(1,4-dioxo-1,4-diphenylbut-2-en-2-yl)-2-methylpiperazine-1-carboxylate (3m)**



Following the general procedure A, **3m** (65%, 56.5 mg, *E*/*Z* > 20:1) as a white solid (mp 89-91 ºC) was obtained after flash chromatography (Petroleum ether/Acetone = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.6 Hz, 2H), 7.85 (d, *J* = 7.1 Hz, 2H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.51 – 7.33 (m, 5H), 6.13 (s, 1H), 4.37 – 2.91 (m, 7H), 1.45 (s, 9H), 1.31 – 0.92 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.7, 186.9, 161.4, 154.2, 138.8, 133.5, 131.5, 128.9, 128.8, 128.1, 127.9, 127.6, 93.1, 80.4, 50.8, 47.2, 28.3, 16.1. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{26}H_{31}N_2O_4^+$ 435.2278; Found 435.2271.

(*E*)-4-(4-(1,4-dioxo-1,4-diphenylbut-2-en-2-yl)piperazin-1-yl)benzonitrile (3n)

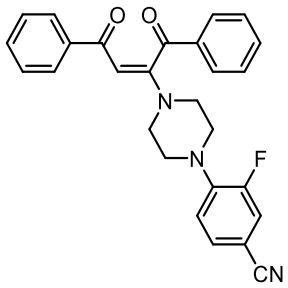


Following the general procedure A, **3n** (57%, 61.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 135-137 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc =

2:1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.92 (d, J = 7.7 Hz, 2H), 7.85 (d, J = 7.6 Hz, 2H), 7.66 – 7.44 (m, 7H), 7.39 (t, J = 7.5 Hz, 2H), 6.94 (d, J = 8.7 Hz, 2H), 6.34 (s, 1H), 3.69 – 3.37 (m, 8H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.7, 185.8, 160.9, 152.9, 139.0, 136.2, 134.0, 133.9, 132.1, 129.6, 128.8, 128.3, 128.1, 120.5, 114.3, 99.0, 92.8, 46.9. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{24}N_3O_2^+$ 422.1863; Found 422.1853.

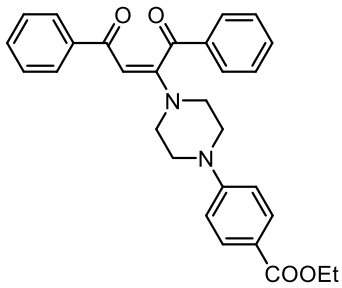
(*E*)-4-(4-(1,4-dioxo-1,4-diphenylbut-2-en-2-yl)piperazin-1-yl)-3-fluorobenzonitrile (3o)



Following the general procedure A, **3o** (59%, 51.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 198-200 ºC) was obtained after flash chromatography (Petroleum ether/Acetone = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 7.1 Hz, 2H), 7.85 (d, J = 7.2 Hz, 2H), 7.57 (t, J = 7.3 Hz, 1H), 7.52 – 7.42 (m, 3H), 7.41 – 7.33 (m, 3H), 7.28 (dd, J = 12.8, 2.2 Hz, 1H), 6.87 (t, J = 8.5 Hz, 1H), 6.21 (s, 1H), 3.55 (d, J = 27.0 Hz, 4H), 3.26 (d, J = 16.7 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 187.1, 160.5, 153.9 (d, J = 248.8 Hz), 143.0 (d, J = 7.7 Hz), 138.6, 135.6, 133.6, 131.7, 129.4 (d, J = 3.3 Hz), 129.0, 128.2, 128.0, 127.7, 119.8 (d, J = 24.5 Hz), 118.8 (d, J = 3.6 Hz), 118.0, 104.7 (d, J = 9.5 Hz), 93.7, 49.0, 47.2. ^{19}F NMR (376 MHz, $CDCl_3$) δ -119.6. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{27}H_{22}FN_3NaO_2^+$ 462.1588; Found 462.1571.

ethyl (*E*)-4-(4-(1,4-dioxo-1,4-diphenylbut-2-en-2-yl)piperazin-1-yl)benzoate (3p)

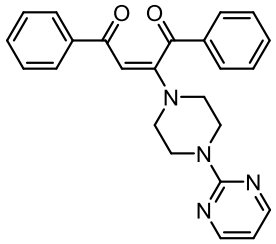


Following the general procedure A, **3p** (72%, 67.5 mg, *E*/*Z* > 20:1) as a yellow solid (mp 177-179 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 7.3 Hz, 2H), 7.93 (d, J = 8.9 Hz, 2H), 7.85 (d, J = 7.3 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.51 – 7.40 (m, 3H), 7.37 (t, J = 7.5 Hz, 2H), 6.83 – 6.75 (m, 2H), 6.18 (s, 1H), 4.32 (q, J = 7.1 Hz, 2H), 3.69 – 3.28 (m, 8H),

1.36 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 187.0, 166.4, 160.5, 152.9, 138.7, 135.6, 133.6, 131.6, 131.2, 129.0, 128.2, 128.0, 127.7, 120.9, 113.6, 93.3, 60.4, 46.7, 14.3. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{29}H_{29}N_2O_4^+$ 469.2122; Found 469.2115.

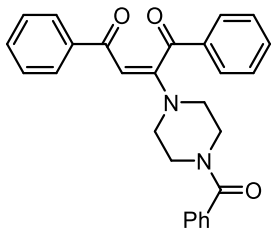
(*E*)-1,4-diphenyl-2-(4-(pyrimidin-2-yl)piperazin-1-yl)but-2-ene-1,4-dione (3q)



Following the general procedure A, **3q** (50%, 39.8 mg, *E*/*Z* > 20:1) as a white solid (mp 98-100 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 4.7 Hz, 2H), 8.06 (d, *J* = 7.0 Hz, 2H), 7.86 (d, *J* = 7.0 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.51 – 7.41 (m, 3H), 7.40 – 7.34 (m, 2H), 6.56 (t, *J* = 4.8 Hz, 1H), 6.18 (s, 1H), 4.03 – 3.77 (m, 4H), 3.62 – 3.35 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.0, 187.0, 161.1, 160.8, 157.7, 138.8, 135.7, 133.5, 131.5, 128.9, 128.1, 128.0, 127.7, 110.7, 93.1, 47.1, 42.9. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{23}N_4O_2^+$ 399.1816; Found 399.1802.

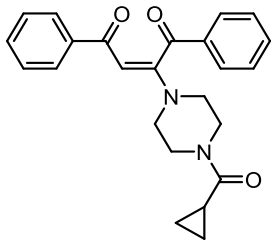
(*E*)-2-(4-benzoylpiperazin-1-yl)-1,4-diphenylbut-2-ene-1,4-dione (3r)



Following the general procedure A, **3r** (61%, 51.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 179-181 ºC) was obtained after flash chromatography (Petroleum ether/Acetone = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 7.2 Hz, 2H), 7.84 (d, *J* = 7.1 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.49 – 7.33 (m, 10H), 6.19 (s, 1H), 4.02 – 3.19 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 187.1, 170.5, 160.5, 138.6, 135.5, 134.6, 133.6, 131.7, 130.2, 129.0, 128.6, 128.2, 128.0, 127.7, 127.0, 93.9, 47.2, 42.5. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{25}N_2O_3^+$ 425.1860; Found 425.1866.

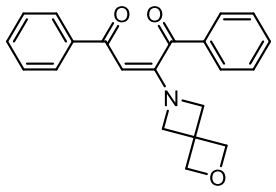
(*E*)-2-(4-(cyclopropanecarbonyl)piperazin-1-yl)-1,4-diphenylbut-2-ene-1,4-dione (3s)



Following the general procedure A, **3s** (66%, 51.1 mg, *E*/*Z* > 20:1) as a white solid (mp 94-96 ºC) was obtained after flash chromatography (Petroleum ether/ Acetone = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.2 Hz, 2H), 7.85 (d, *J* = 7.3 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.52 – 7.43 (m, 3H), 7.38 (dd, *J* = 8.3, 6.8 Hz, 2H), 6.16 (s, 1H), 3.75 (d, *J* = 25.2 Hz, 4H), 3.45 (d, *J* = 33.0 Hz, 4H), 1.71 – 1.61 (m, 1H), 1.06 – 0.95 (m, 2H), 0.87 – 0.74 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 187.1, 172.4, 160.5, 138.6, 135.5, 133.6, 131.7, 129.0, 128.2, 128.0, 127.7, 93.6, 47.0, 44.7, 10.9, 7.8. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{24}H_{24}N_2NaO_3^+$ 411.1679; Found 411.1682.

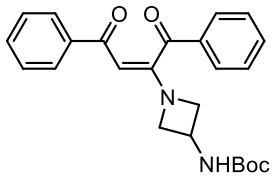
(*E*)-1,4-diphenyl-2-(2-oxa-6-azaspiro[3.3]heptan-6-yl)but-2-ene-1,4-dione (3t)



Following the general procedure A, **3t** (64%, 43.3 mg, *E*/*Z* > 20:1) as a white solid (mp 200-202 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 7.2 Hz, 2H), 7.85 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.50 – 7.41 (m, 3H), 7.38 (dd, *J* = 8.3, 6.7 Hz, 2H), 5.82 (s, 1H), 4.78 (s, 4H), 4.30 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.3, 186.2, 159.5, 138.4, 134.7, 133.7, 131.6, 128.9, 128.3, 128.2, 127.7, 92.3, 80.4, 60.6, 38.8. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{21}H_{19}NNaO_3^+$ 356.1257; Found 356.1253.

***tert*-butyl (*E*)-(1-(1,4-dioxo-1,4-diphenylbut-2-en-2-yl)azetidin-3-yl)carbamate (3u)**

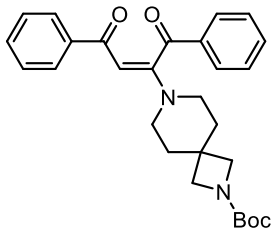


Following the general procedure A, **3u** (76%, 61.7 mg, *E*/*Z* > 20:1) as a yellow solid (mp 208-210 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 4:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.3 Hz, 2H), 7.81 (d, *J* = 7.6 Hz, 2H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.49 – 7.39 (m, 3H), 7.34 (t, *J* = 7.6 Hz, 2H), 5.76 (s, 1H), 5.71 – 5.08 (m, 1H), 4.74 – 3.65 (m, 5H), 1.43 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.5,

186.2, 159.7, 154.8, 138.5, 134.8, 133.6, 131.5, 128.8, 128.3, 128.1, 127.7, 92.1, 80.2, 58.9, 41.2, 28.2. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{24}H_{26}N_2NaO_4^+$ 429.1785; Found 429.1780.

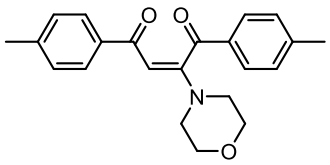
***tert*-butyl (*E*)-7-(1,4-dioxo-1,4-diphenylbut-2-en-2-yl)-2,7-diazaspiro[3.5]nonane-2-carboxylate (3v)**



Following the general procedure A, **3v** (68%, 62.6 mg, *E*/*Z* > 20:1) as a yellow solid (mp 210-212 ºC) was obtained after flash chromatography (Petroleum ether/Acetone = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.1 Hz, 2H), 7.84 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.52 – 7.41 (m, 3H), 7.36 (t, *J* = 7.4 Hz, 2H), 6.16 (s, 1H), 3.66 (d, *J* = 4.4 Hz, 4H), 3.33 (s, 3H), 1.82 (d, *J* = 20.8 Hz, 5H), 1.44 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.0, 187.0, 160.6, 156.2, 138.9, 135.7, 133.4, 131.4, 128.9, 128.10, 128.07, 128.0, 127.6, 92.9, 79.7, 58.6, 45.1, 34.8, 33.2, 28.3. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{28}H_{32}N_2NaO_4^+$ 483.2254; Found 483.2244.

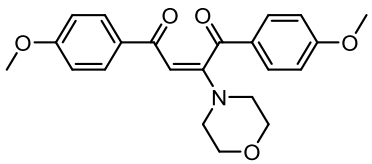
(*E*)-2-morpholino-1,4-di-*p*-tolylbut-2-ene-1,4-dione (3w)[6]



Following the general procedure A, **3w** (65%, 45.4 mg, *E*/*Z* > 20:1) as a yellow solid was obtained after flash chromatography (Petroleum ether/EtOAc = 4:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.9 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 6.14 (s, 1H), 3.80 – 3.61 (m, 4H), 3.47 – 3.20 (m, 4H), 2.38 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.7, 186.7, 160.7, 144.4, 142.0, 136.1, 133.3, 129.6, 128.8, 128.1, 127.7, 93.2, 66.1, 47.4, 21.7, 21.4. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{24}NO_3^+$ 350.1751; Found 350.1752.

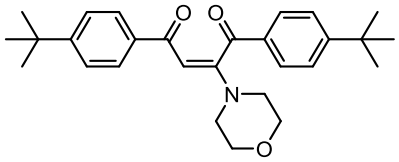
(*E*)-1,4-bis(4-methoxyphenyl)-2-morpholinobut-2-ene-1,4-dione (3x)



Following the general procedure A, **3x** (66%, 50.3 mg, *E*/*Z* > 20:1) as a white solid (mp 135-137 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 1:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.5 Hz, 2H), 7.84 (d, *J* = 8.9 Hz, 2H), 6.94 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.12 (s, 1H), 3.84 (s, 3H), 3.81 (s, 3H), 3.78 – 3.63 (m, 4H), 3.47 – 3.21 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.8, 185.8, 163.8, 162.3, 160.5, 131.6, 130.4, 129.7, 129.0, 114.2, 113.2, 93.0, 66.1, 55.4, 55.2, 47.3. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{22}H_{23}NNaO_5^+$ 404.1468; Found 404.1445.

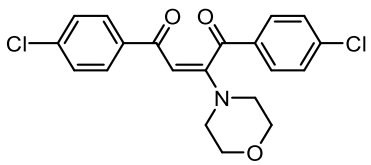
(*E*)-1,4-bis(4-(tert-butyl)phenyl)-2-morpholinobut-2-ene-1,4-dione (3y)



Following the general procedure A, **3y** (77%, 66.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 224-226 °C) was obtained after flash chromatography (Petroleum ether/EtOAc = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.2 Hz, 2H), 7.80 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.2 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 6.16 (s, 1H), 3.81 – 3.62 (m, 4H), 3.49 – 3.18 (m, 4H), 1.31 (d, *J* = 6.2 Hz, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.7, 186.7, 160.7, 157.0, 154.9, 136.1, 133.1, 127.9, 127.5, 125.8, 125.0, 93.3, 66.1, 47.4, 35.0, 34.8, 31.02, 30.95. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{28}H_{36}NO_3^+$ 434.2690; Found 434.2692.

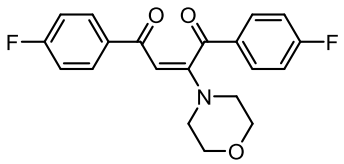
(*E*)-1,4-bis(4-chlorophenyl)-2-morpholinobut-2-ene-1,4-dione (3z)



Following the general procedure A, **3z** (55%, 42.9 mg, *E*/*Z* > 20:1) as a yellow solid (mp 62-64 °C) was obtained after flash chromatography (Petroleum ether/EtOAc = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.5 Hz, 2H), 7.77 (d, *J* = 8.6 Hz, 2H), 7.46 (d, *J* = 8.6 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 6.09 (s, 1H), 3.83 – 3.62 (m, 4H), 3.49 – 3.17 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.5, 185.8, 160.7, 140.0, 137.9, 136.8, 134.0, 129.4, 129.3, 129.1, 128.4, 92.9, 66.0, 47.6. HRMS (ESI) m/z: $[M+K]^+$ Calcd for $C_{20}H_{17}Cl_2NKO_3^+$ 428.0217; Found 428.0230.

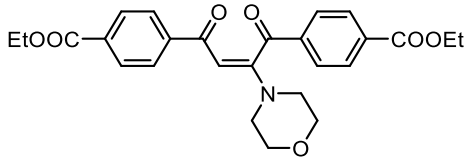
(*E*)-1,4-bis(4-fluorophenyl)-2-morpholinobut-2-ene-1,4-dione (3aa)



Following the general procedure B, **3aa** (61%, 43.6 mg, *E*/*Z* > 20:1) as a yellow solid (mp 171-173 °C) was obtained after flash chromatography (Petroleum ether/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 8.6, 5.5 Hz, 2H), 7.85 (dd, *J* = 8.7, 5.6 Hz, 2H), 7.16 (t, *J* = 8.6 Hz, 2H), 7.04 (t, *J* = 8.6 Hz, 2H), 6.10 (s, 1H), 3.84 – 3.64 (m, 4H), 3.49 – 3.21 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.3, 185.7, 165.9 (d, *J* = 255.6 Hz), 164.9 (d, *J* = 252.5 Hz), 160.7, 134.9 (d, *J* = 3.0 Hz), 132.2 (d, *J* = 2.9 Hz), 130.6 (d, *J* = 9.5 Hz), 130.1 (d, *J* = 9.0 Hz), 116.2 (d, *J* = 22.1 Hz), 115.2 (d, *J* = 21.6 Hz), 93.0, 66.1, 47.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ -104.0, -107.7. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{20}H_{17}F_2NNaO_3^+$ 380.1069; Found 380.1051.

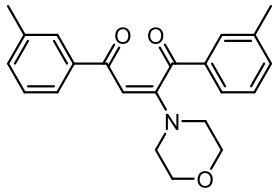
(*E*)- diethyl 4,4'-(2-morpholinomaleoyl)dibenzoate (3ab)



Following the general procedure C, **3ab** (63%, 58.7 mg, *E*/*Z* > 20:1) as a yellow solid (mp 194-196 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.4 Hz, 2H), 8.09 (d, *J* = 8.4 Hz, 2H), 8.04 (d, *J* = 8.4 Hz, 2H), 7.87 (d, *J* = 8.5 Hz, 2H), 6.17 (s, 1H), 4.45 – 4.31 (m, 4H), 4.45 – 4.31 (m, 4H), 3.83 – 3.67 (m, 4H), 1.44 – 1.35 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.8, 186.4, 165.9, 165.6, 161.1, 142.0, 138.6, 134.6, 132.9, 130.2, 129.4, 127.7, 127.5, 93.4, 66.1, 61.4, 61.2, 47.8, 14.2. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{26}H_{27}NNaO_7^+$ 488.1680; Found 488.1671.

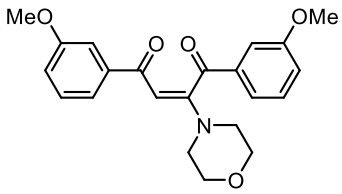
(*E*)-2-morpholino-1,4-di-*m*-tolylbut-2-ene-1,4-dione (3ac)



Following the general procedure A, **3ac** (59%, 41.2 mg, *E*/*Z* > 20:1) as a yellow solid (mp 79-81 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 4:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (s, 1H), 7.79 (d, *J* = 7.1 Hz, 1H), 7.69 – 7.61 (m, 2H), 7.41 – 7.30 (m, 2H), 7.29 – 7.23 (m, 2H), 6.14 (s, 1H), 3.82 – 3.66 (m, 4H), 3.49 – 3.23 (m, 4H), 2.40 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.1, 187.3, 161.0, 138.8, 138.7, 137.9, 135.7, 134.4, 132.3, 128.8, 128.4, 128.2, 128.0, 125.4, 124.8, 93.4, 66.1, 47.5, 21.4, 21.3. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{22}H_{23}NNaO_3^+$ 372.1570; Found 372.1540.

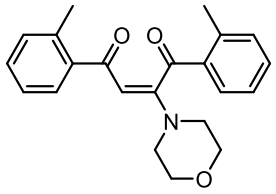
(*E*)-1,4-bis(3-methoxyphenyl)-2-morpholinobut-2-ene-1,4-dione (3ad)



Following the general procedure A, **3ad** (54%, 41.2 mg, *E*/*Z* > 20:1) as a yellow solid (mp 125-127 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 2.7, 1.5 Hz, 1H), 7.56 (d, *J* = 7.6 Hz, 1H), 7.44 (d, *J* = 7.7 Hz, 1H), 7.40 – 7.34 (m, 2H), 7.32 – 7.25 (m, 1H), 7.15 – 7.09 (m, 1H), 7.03 – 6.98 (m, 1H), 6.13 (s, 1H), 3.87 (s, 3H), 3.80 – 3.66 (m, 7H), 3.47 – 3.24 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.6, 186.9, 161.0, 160.1, 159.6, 140.2, 137.0, 130.0, 129.0, 121.0, 120.3, 120.1, 118.3, 112.1, 111.6, 93.4, 66.1, 55.4, 55.3, 47.6. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{22}H_{23}NNaO_5^+$ 404.1468; Found 404.1463.

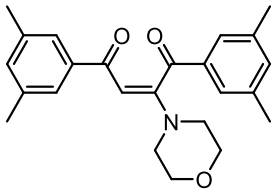
(*E*)-2-morpholino-1,4-di-o-tolylbut-2-ene-1,4-dione (3ae)



Following the general procedure A, **3ae** (43%, 30.0 mg, *E*/*Z* > 20:1) as a white solid (mp 130-132 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 8:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.45 – 7.36 (m, 1H), 7.38 – 7.27 (m, 2H), 7.28 – 7.17 (m, 3H), 7.17 – 7.06 (m, 2H), 5.67 (s, 1H), 3.75 (t, *J* = 4.9 Hz, 4H), 3.36 (s, 4H), 2.84 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.0, 192.2, 161.2, 141.3, 141.2, 135.8, 134.5, 132.4, 130.8, 129.9, 129.3, 127.0, 125.4, 125.2, 97.2, 66.2, 47.5, 21.9, 19.8. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{22}H_{23}NNaO_3^+$ 372.1570; Found 372.1564.

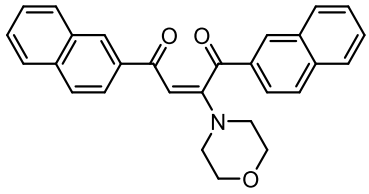
(*E*)-1,4-bis(3,5-dimethylphenyl)-2-morpholinobut-2-ene-1,4-dione (3af)



Following the general procedure A, **3af** (57%, 43.0 mg, *E*/*Z* > 20:1) as a yellow solid (mp 142-144 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 4:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (s, 2H), 7.48 (s, 2H), 7.18 (s, 1H), 7.08 (s, 1H), 6.13 (s, 1H), 3.83 – 3.67 (m, 4H), 3.50 – 3.24 (m, 4H), 2.34 (s, 6H), 2.31 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.2, 187.3, 161.0, 138.7, 138.5, 137.6, 135.7, 135.3, 133.2, 125.7, 125.5, 93.4, 66.1, 47.5, 21.2, 21.1. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{24}H_{27}NNaO_3^+$ 400.1883; Found 400.1860.

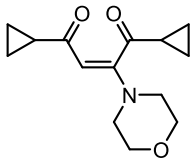
(*E*)-2-morpholino-1,4-di(naphthalen-2-yl)but-2-ene-1,4-dione (3ag)



Following the general procedure C, **3ag** (56%, 47.2 mg, *E*/*Z* > 20:1) as a yellow solid (mp 84-86 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 1.8 Hz, 1H), 8.40 (d, *J* = 1.7 Hz, 1H), 8.19 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.98 – 7.77 (m, 7H), 7.60 – 7.45 (m, 4H), 6.39 (s, 1H), 3.84 – 3.63 (m, 4H), 3.60 – 3.30 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 186.9, 161.1, 136.0, 135.9, 134.9, 133.2, 132.7, 132.5, 129.7, 129.6, 129.2, 129.1, 128.54, 128.50, 127.9, 127.8, 127.64, 127.57, 126.7, 126.3, 124.3, 123.5, 93.6, 66.2, 47.6. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{28}H_{24}NO_3^+$ 422.1751; Found 422.1747.

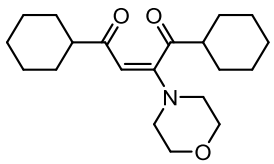
(*E*)-1,4-dicyclopropyl-2-morpholinobut-2-ene-1,4-dione (3ah)



Following the general procedure C, **3ah** (44%, 21.9 mg, *E*/*Z* > 20:1) as a white solid (mp 140-142 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.29 (s, 1H), 3.71 – 3.64 (t, *J* = 4.8 Hz, 4H), 3.16 (t, *J* = 4.9 Hz, 4H), 2.15 – 2.05 (m, 1H), 1.79 – 1.68 (m, 1H), 1.18 (s, 2H), 1.04 – 0.96 (m, 2H), 0.95 – 0.87 (m, 2H), 0.75 – 0.65 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.1, 196.5, 160.6, 95.7, 65.9, 47.2, 22.4, 20.4, 12.2, 9.7. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{14}H_{19}NNaO_3^+$ 272.1257; Found 272.1251.

(*E*)-1,4-dicyclohexyl-2-morpholinobut-2-ene-1,4-dione (3ai)

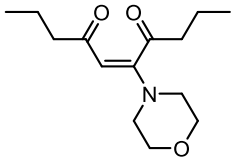


Following the general procedure C, **3ai** (38%, 25.3 mg, *E*/*Z* > 20:1) as a white solid (mp 100-102 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc =

6:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.18 (s, 1H), 3.67 (t, *J* = 4.8 Hz, 4H), 3.14 (t, *J* = 4.8 Hz, 4H), 2.52 – 2.38 (m, 1H), 2.26 – 2.15 (m, 1H), 2.05 (d, *J* = 11.2 Hz, 2H), 1.80 – 1.57 (m, 8H), 1.34 – 1.08 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.8, 200.8, 161.0, 95.1, 66.0, 50.8, 50.1, 47.5, 29.2, 28.0, 25.9, 25.8. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{20}H_{31}NNaO_3^+$ 356.2196; Found 356.2194.

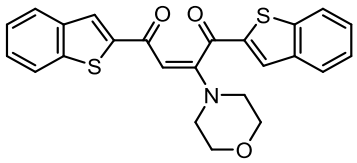
(*E*)-5-morpholinodec-5-ene-4,7-dione (3aj)



Following the general procedure C, **3aj** (36%, 18.2 mg, *E*/*Z* > 20:1) as a white solid (mp 82-84 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.16 (s, 1H), 3.74 (t, *J* = 4.9 Hz, 4H), 3.19 (t, *J* = 4.9 Hz, 4H), 2.65 – 2.46 (m, 2H), 2.31 (t, *J* = 7.5 Hz, 2H), 1.86 – 1.72 (m, 2H), 1.67 – 1.53 (m, 2H), 0.99 (t, *J* = 7.4 Hz, 3H), 0.91 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.1, 197.8, 161.4, 95.2, 66.0, 47.0, 45.2, 44.3, 18.6, 16.6, 13.9, 13.6. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{14}H_{23}NNaO_3^+$ 276.1570; Found 276.1557.

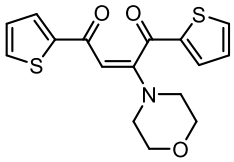
(*E*)-1,4-bis(benzo[b]thiophen-2-yl)-2-morpholinobut-2-ene-1,4-dione (3ak)



Following the general procedure C, **3ak** (49%, 42.5 mg, *E*/*Z* > 20:1) as a yellow solid (mp 184-186 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.91 – 7.76 (m, 5H), 7.43 (t, *J* = 7.3 Hz, 1H), 7.40 – 7.30 (m, 3H), 6.15 (s, 1H), 3.76 (s, 4H), 3.43 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.2, 180.7, 159.5, 145.4, 142.9, 142.3, 141.9, 139.3, 138.9, 130.1, 127.6, 126.5, 126.4, 126.1, 125.3, 125.0, 124.6, 123.0, 122.7, 93.5, 66.1, 47.6. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{20}NO_3S_2^+$ 434.0879; Found 434.0876.

(*E*)-2-morpholino-1,4-di(thiophen-2-yl)but-2-ene-1,4-dione (3al)

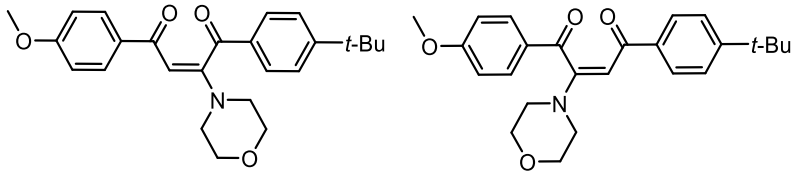


Following the general procedure A, **3al** (52%, 34.7 mg, *E*/*Z* > 20:1) as a yellow solid (mp 170-172 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc =

3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 7.66 (m, 2H), 7.63 (d, *J* = 3.7 Hz, 1H), 7.49 (d, *J* = 4.9 Hz, 1H), 7.14 – 7.03 (m, 2H), 5.96 (s, 1H), 3.75 (t, *J* = 4.9 Hz, 4H), 3.37 (t, *J* = 4.9 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 179.7, 159.6, 145.9, 142.9, 134.5, 133.0, 131.5, 129.6, 128.3, 127.8, 93.5, 66.1, 47.5. HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{16}H_{15}NNaO_3S_2^+$ 356.0386; Found 356.0361.

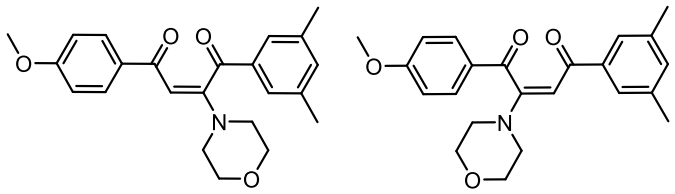
(*E*)-1-(4-(*tert*-butyl)phenyl)-4-(4-methoxyphenyl)-2-morpholinobut-2-ene-1,4-dione (3am) + (*E*)-4-(4-(*tert*-butyl)phenyl)-1-(4-methoxyphenyl)-2-morpholinobut-2-ene-1,4-dione (3am')



Following the general procedure A, **3am + 3am'** (50%, 40.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 76-78 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.94 (m, 2H), 7.88 – 7.77 (m, 2H), 7.50 – 7.35 (m, 2H), 6.95 – 6.83 (m, 2H), 6.14 (d, *J* = 4.6 Hz, 1H), 3.82 (d, *J* = 9.8 Hz, 3H), 3.77 – 3.63 (m, 4H), 3.47 – 3.22 (m, 4H), 1.31 (d, *J* = 6.1 Hz, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 192.8, 186.7, 186.0, 163.8, 162.3, 160.64, 160.60, 157.0, 155.0, 136.2, 133.2, 132.1, 131.5, 130.4, 129.8, 127.9, 127.5, 125.9, 125.0, 114.2, 113.3, 93.3, 93.2, 66.1, 55.4, 55.2, 47.4, 35.1, 34.8, 31.04, 30.97. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{25}H_{30}NO_4^+$ 408.2169; Found 408.2165.

(*E*)-1-(3,5-dimethylphenyl)-4-(4-methoxyphenyl)-2-morpholinobut-2-ene-1,4-dione (3an) + (*E*)-4-(3,5-dimethylphenyl)-1-(4-methoxyphenyl)-2-morpholinobut-2-ene-1,4-dione (3an')

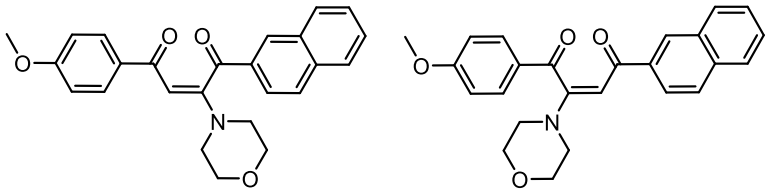


Following the general procedure A, **3an + 3an'** (47%, 35.7 mg, *E*/*Z* > 20:1) as a yellow solid (mp 74-76 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.81 (m, 2H), 7.63 (s, 0.86H), 7.45 (s, 1.14H), 7.18 (s, 0.43H), 7.07 (s, 0.56H), 6.95 – 6.82 (m, 2H), 6.12 (d, *J* = 1.8 Hz, 1H), 3.81 (d, *J* = 9.7 Hz, 3H), 3.79 – 3.67 (m, 4H), 3.47 – 3.24 (m, 4H), 2.32 (d, *J* = 17.0 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.4, 192.7, 187.3, 185.9, 163.8, 162.3, 160.72, 160.69, 138.9, 138.5, 137.6, 135.3, 133.1, 132.0, 131.5, 130.3, 129.8, 128.9, 125.7, 125.5, 114.2,

113.2, 93.3, 93.1, 66.1, 55.4, 55.2, 47.4, 21.2, 21.1. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{26}NO_4^+$ 380.1856; Found 380.1859.

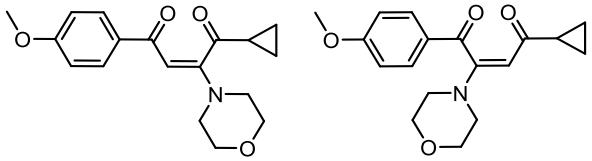
(*E*)-4-(4-methoxyphenyl)-2-morpholino-1-(naphthalen-2-yl)but-2-ene-1,4-dione (3ao) + (*E*)-1-(4-methoxyphenyl)-2-morpholino-4-(naphthalen-2-yl)but-2-ene-1,4-dione (3ao')



Following the general procedure C, **3ao + 3ao'** (53%, 42.5 mg, *E*/*Z* > 20:1) as a yellow solid (mp 123-125 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (s, 0.41H), 8.39 (s, 0.58H), 8.18 – 7.77 (m, 6H), 7.61 – 7.43 (m, 2H), 6.95 (d, *J* = 8.4 Hz, 1.23H), 6.84 (d, *J* = 8.4 Hz, 0.86H), 6.30 (s, 0.57H), 6.21 (s, 41H), 3.81 (d, *J* = 16.0 Hz, 3H), 3.78 – 3.64 (m, 4H), 3.52 – 3.24 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.7, 192.7, 186.8, 186.0, 163.9, 162.4, 161.0, 160.6, 136.3, 135.8, 134.8, 133.3, 132.7, 132.5, 131.3, 130.4, 129.8, 129.65, 129.59, 129.2, 129.0, 128.9, 128.5, 128.4, 127.84, 127.77, 127.6, 127.5, 126.7, 126.3, 124.4, 123.5, 114.2, 113.3, 93.5, 93.2, 66.1, 55.4, 55.3, 47.5. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{25}H_{24}NO_4^+$ 402.1700; Found 402.1696.

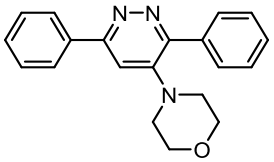
(*E*)-4-(4-methoxyphenyl)-2-morpholino-1-(naphthalen-2-yl)but-2-ene-1,4-dione (3ap) + (*E*)-1-(4-methoxyphenyl)-2-morpholino-4-(naphthalen-2-yl)but-2-ene-1,4-dione (3ap')



Following the general procedure C, **3ap + 3ap'** (41%, 25.8 mg, *E*/*Z* > 20:1) as a yellow solid (mp 101-103 ºC) was obtained after flash chromatography (Petroleum ether/EtOAc = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (dd, *J* = 16.4, 8.6 Hz, 2H), 6.92 (dd, *J* = 14.1, 8.6 Hz, 2H), 5.88 (s, 0.23H), 5.58 (s, 0.77H), 3.85 (d, *J* = 3.2 Hz, 3H), 3.81 – 3.63 (m, 4H), 3.36 – 3.13 (m, 4H), 2.33 – 2.21 (m, 0.25H), 1.83 – 1.75 (m, 0.86H), 1.38 – 1.30 (m, 0.56H), 1.09 (m, 0.48H). 0.96 – 0.79 (m, 1.73H), 0.75 – 0.65 (m, 1.57H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.1, 192.8, 163.8, 163.1, 162.5, 158.4, 130.5, 129.8, 128.9, 114.2, 113.4, 97.0, 92.5, 66.1, 55.5, 55.3, 47.7, 47.1, 22.7, 20.6, 12.4, 9.9, 9.7. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{18}H_{22}NO_4^+$ 316.1543; Found 316.1538.

4-(3,6-diphenylpyridazin-4-yl)morpholine (4a)



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.0 Hz, 2H), 7.97 (d, *J* = 7.6 Hz, 2H), 7.55 – 7.40 (m, 6H), 7.23 (s, 1H), 3.70 (t, *J* = 4.6 Hz, 4H), 3.01 (t, *J* = 4.7 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.1, 152.7, 149.0, 137.5, 136.7, 129.7, 129.1, 128.8, 128.6, 128.0, 127.0, 110.3, 66.1, 49.4. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{20}H_{20}N_3O^+$ 318.1601; Found 318.1606.

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(1) (a) N. Xian, J. Yin, X. Ji, G.-J. Deng and H. Huang, Visible-light-mediated photoredox carbon radical formation from aqueous sulfoxonium ylides, *Org. Lett.,* 2023, **25**, 1161–1165; (b) T. Zhou, P.-F. Qian, J.-Y. Li, Y.-B. Zhou, H.-C. Li, H.-Y. Chen and B.-F. Shi, Efficient synthesis of sulfur-stereogenic sulfoximines via Ru(II)-catalyzed enantioselective C–H functionalization enabled by chiral carboxylic acid, *J. Am. Chem. Soc.,* 2021, **143**, 6810−6816; (c) N. Xian, J. Yin, X. Ji, G.-J. Deng and H. Huang, Stereoselective three-component carbocyclization of 1,7-enynes with sulfoxonium ylides and water under photoredox catalysis, *Org. Lett.,* 2024, **26**, 6736−6741.

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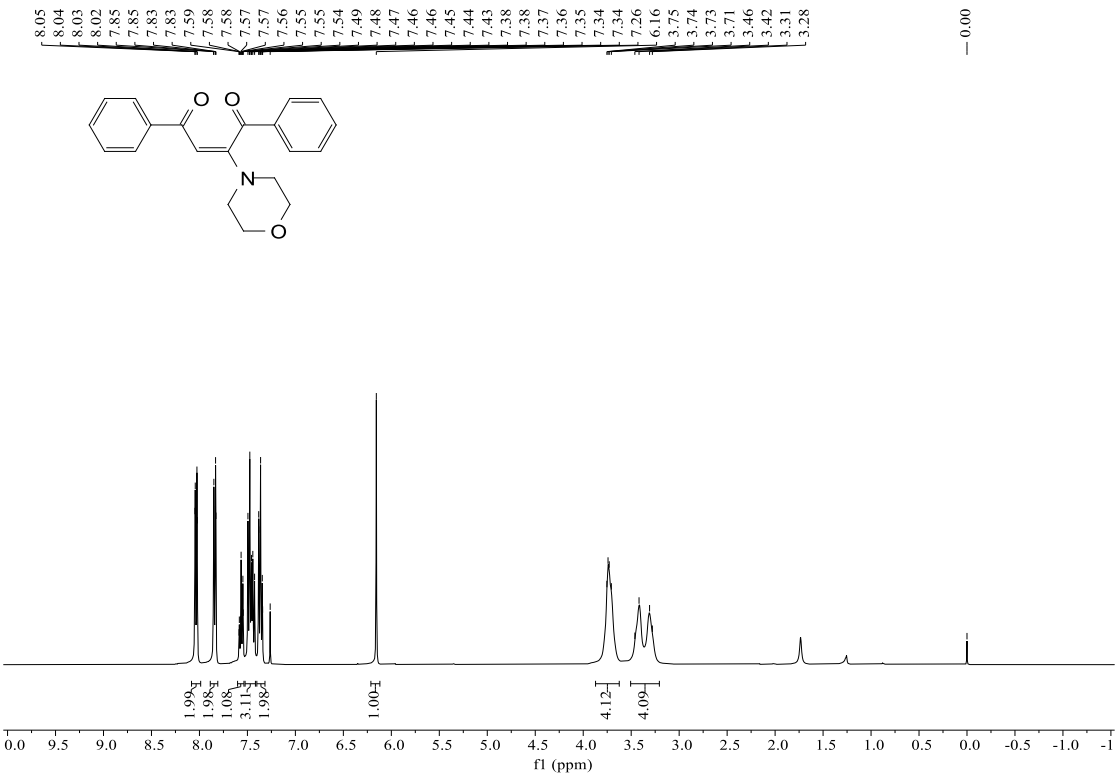
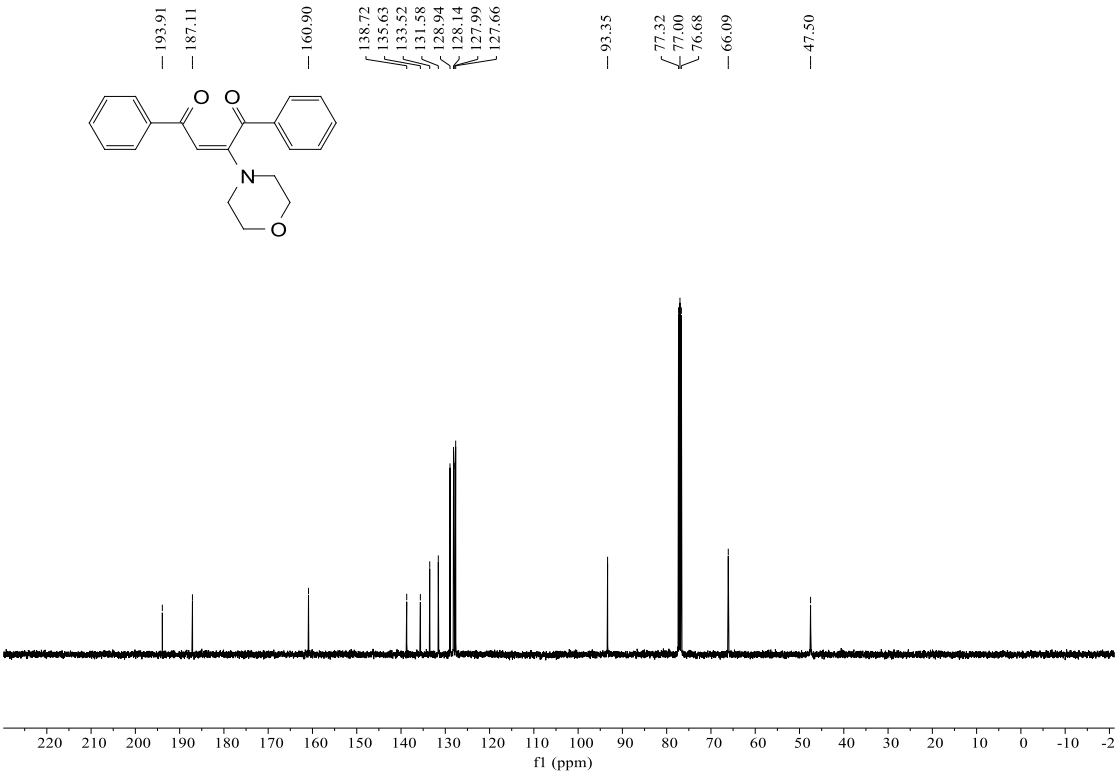
Copies of ^{1}H, ^{13}C and ^{19}F NMR spectra of all products

^{1}H NMR spectra of 3a (400 MHz, $CDCl_3$)



^{13}C NMR spectra of 3a (100 MHz, $CDCl_3$)



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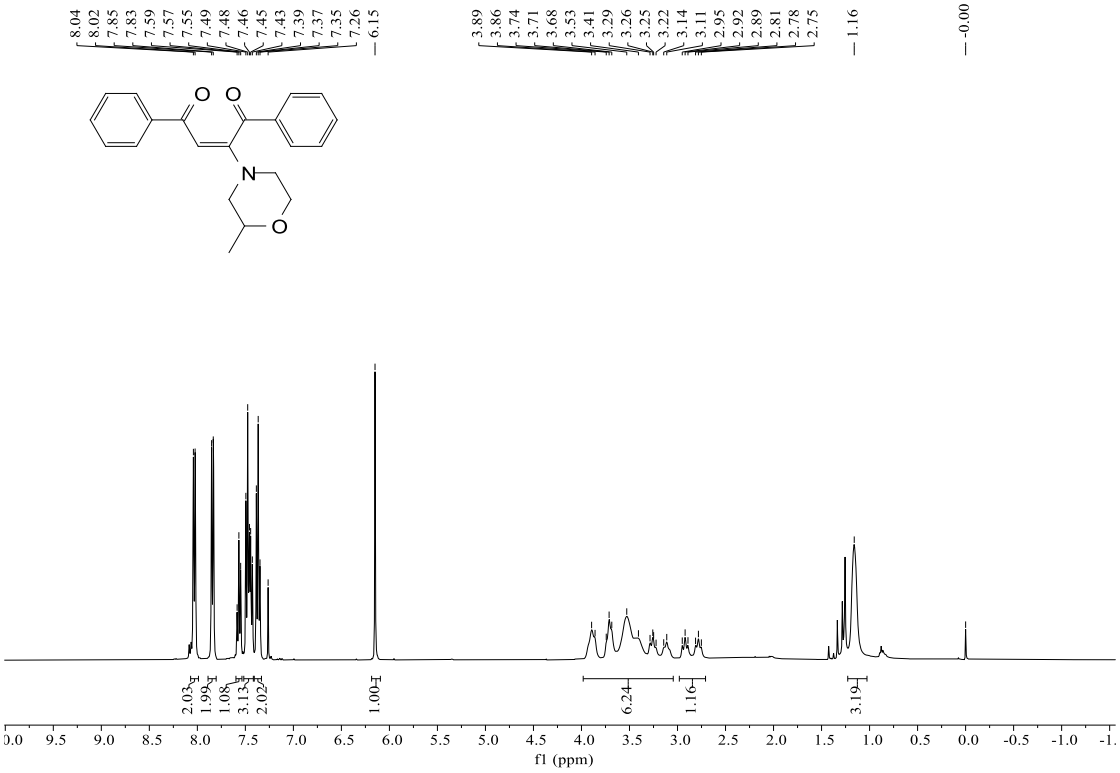

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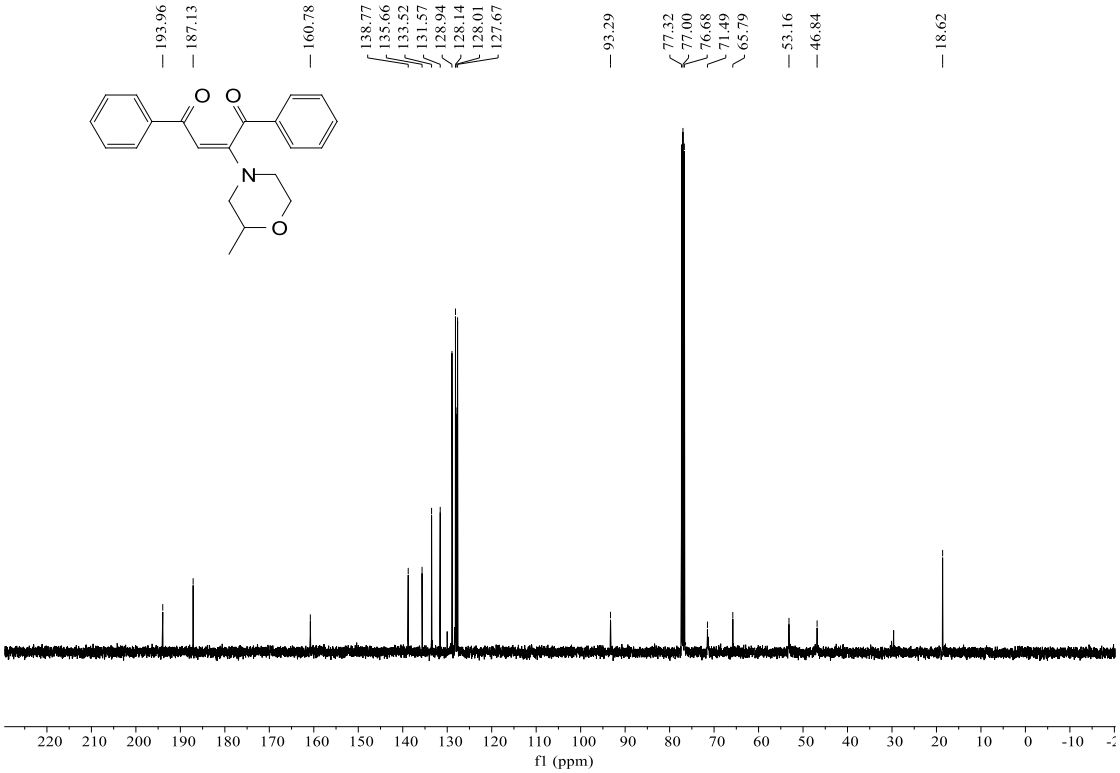
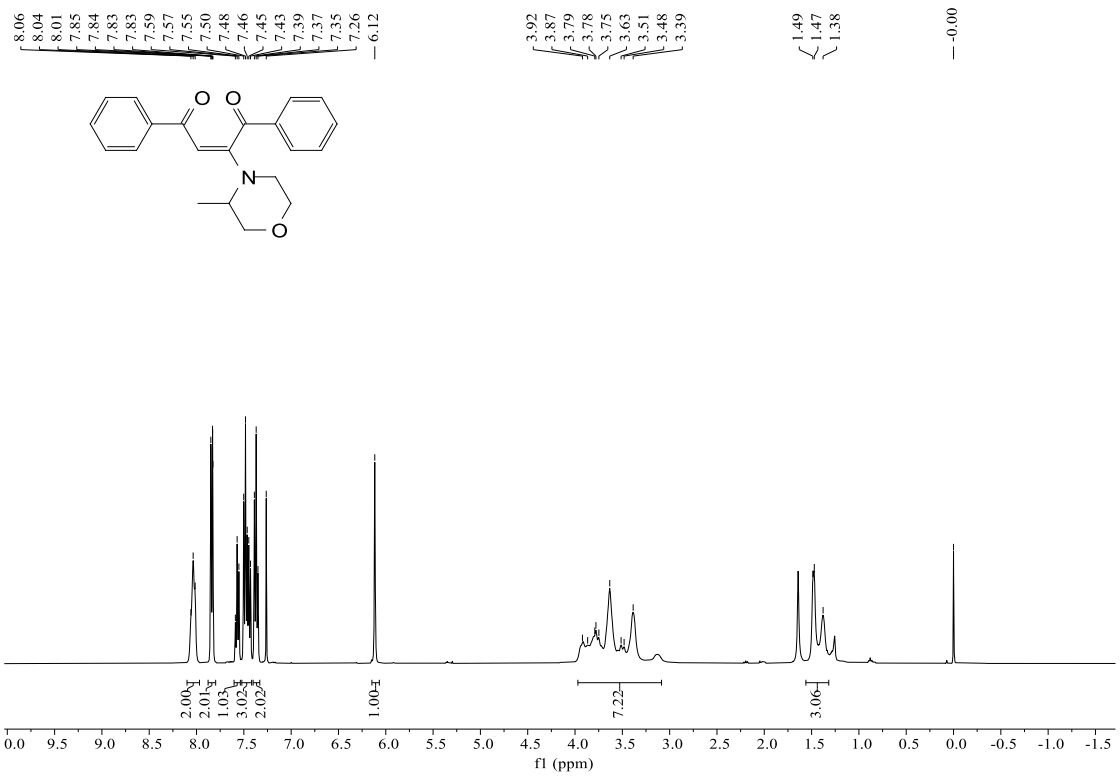


^{1}H NMR spectra of 3c (400 MHz, $CDCl_3$)



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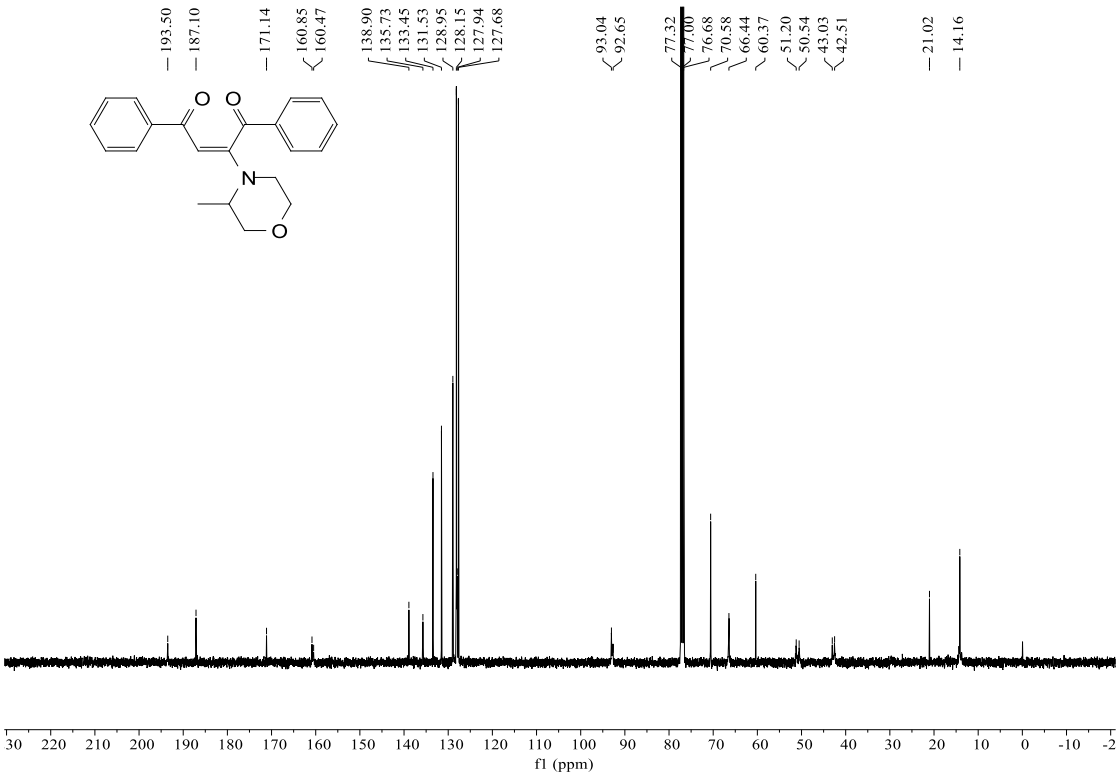

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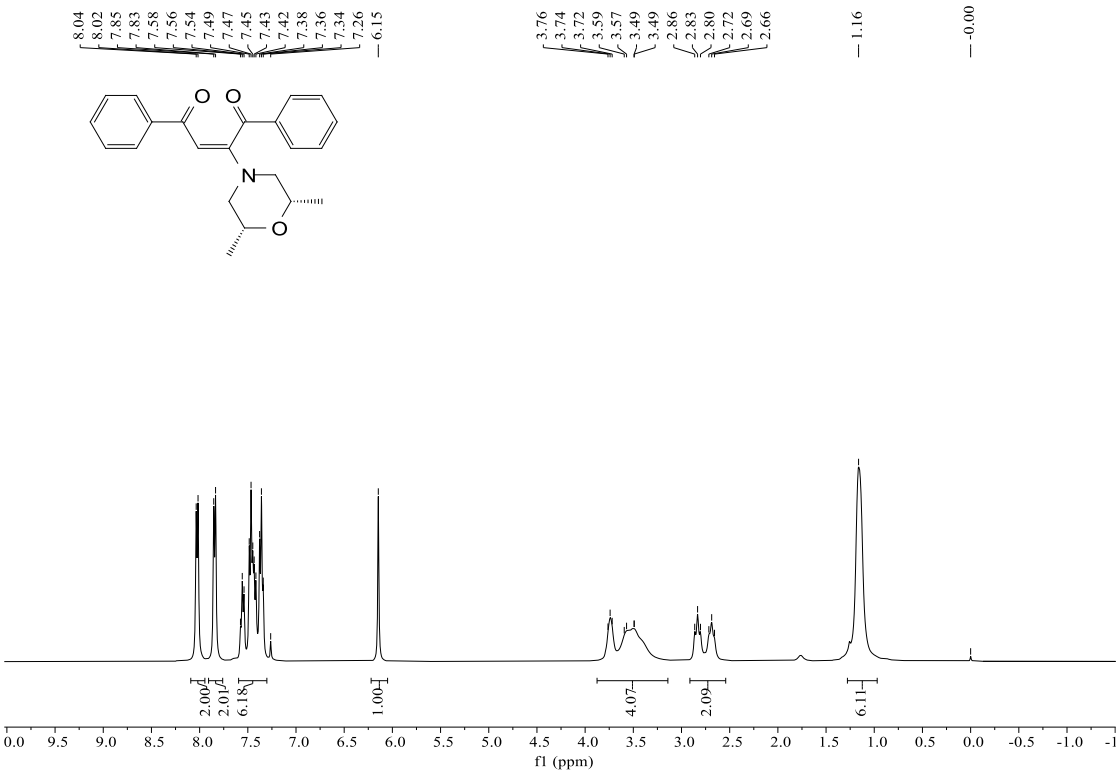

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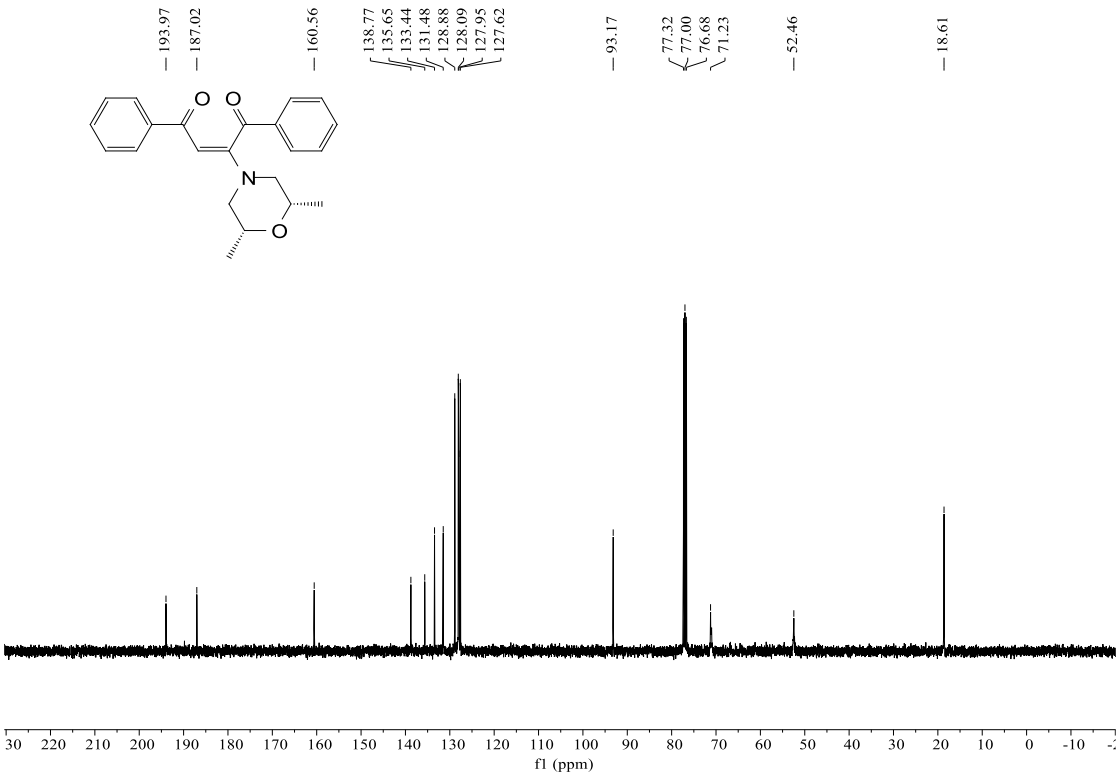

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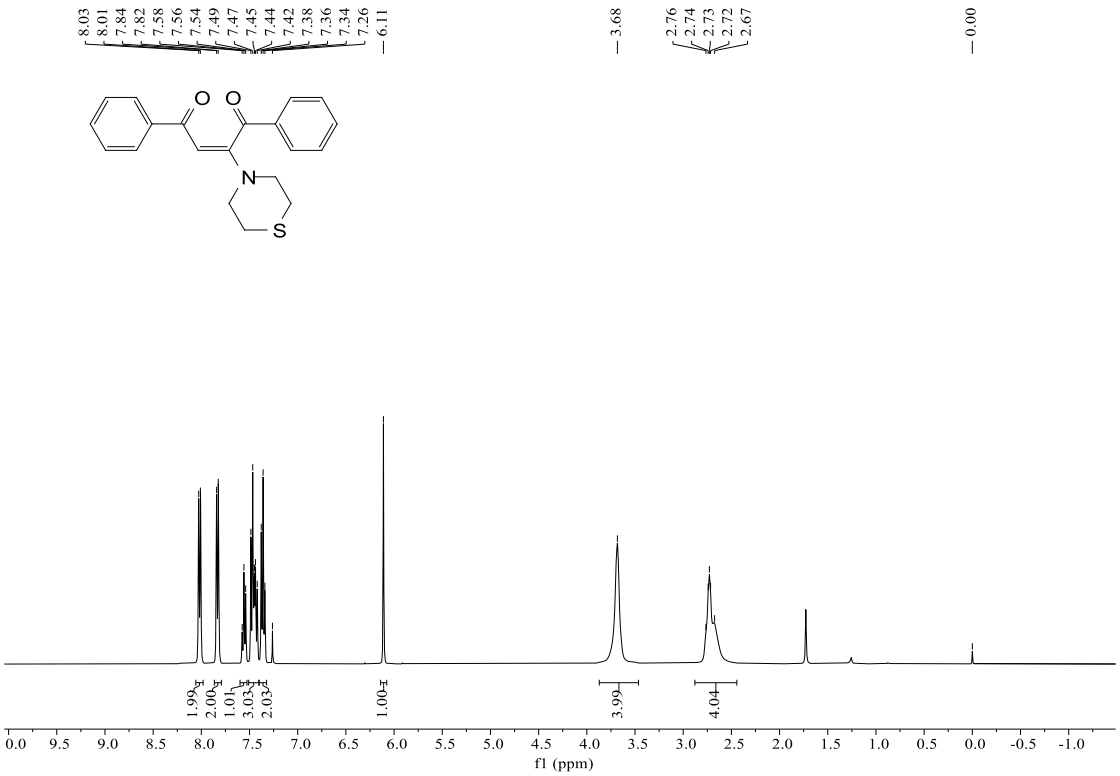

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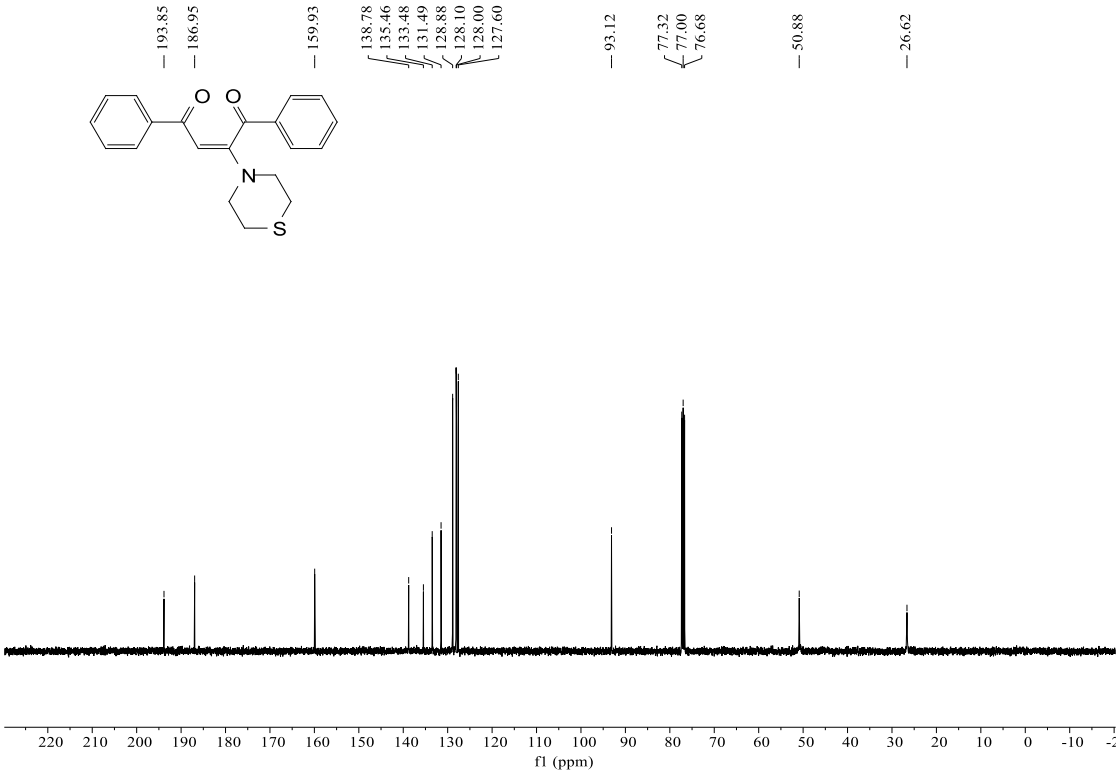
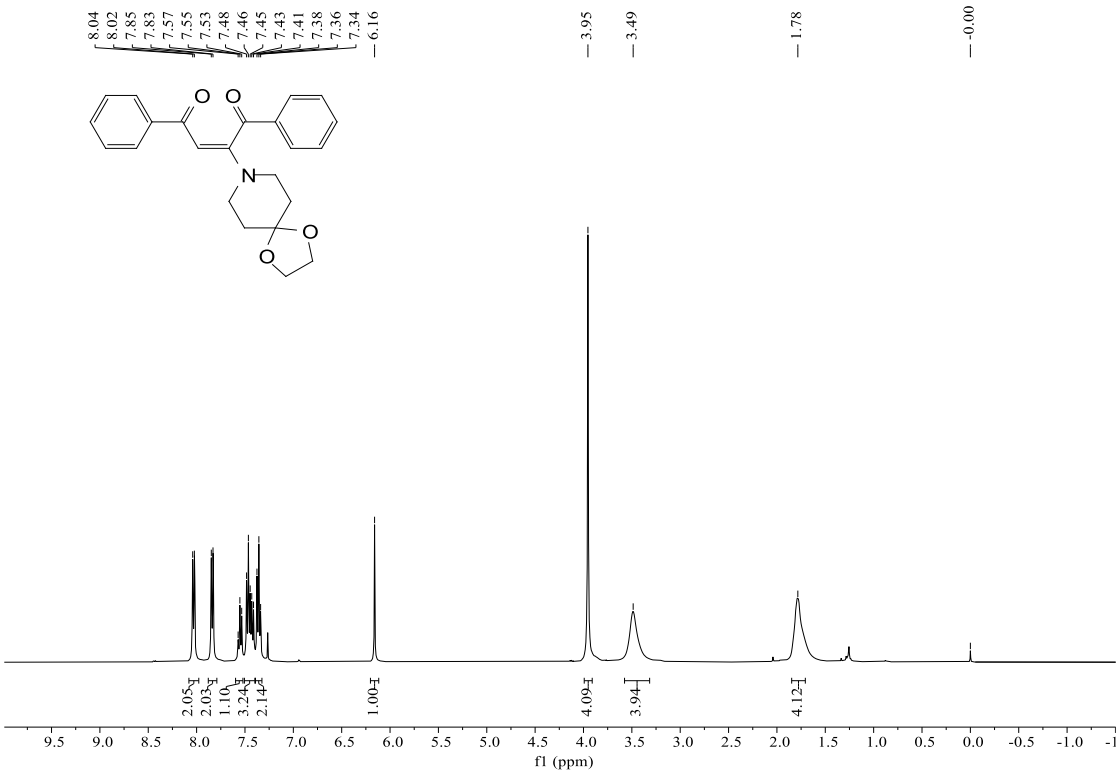
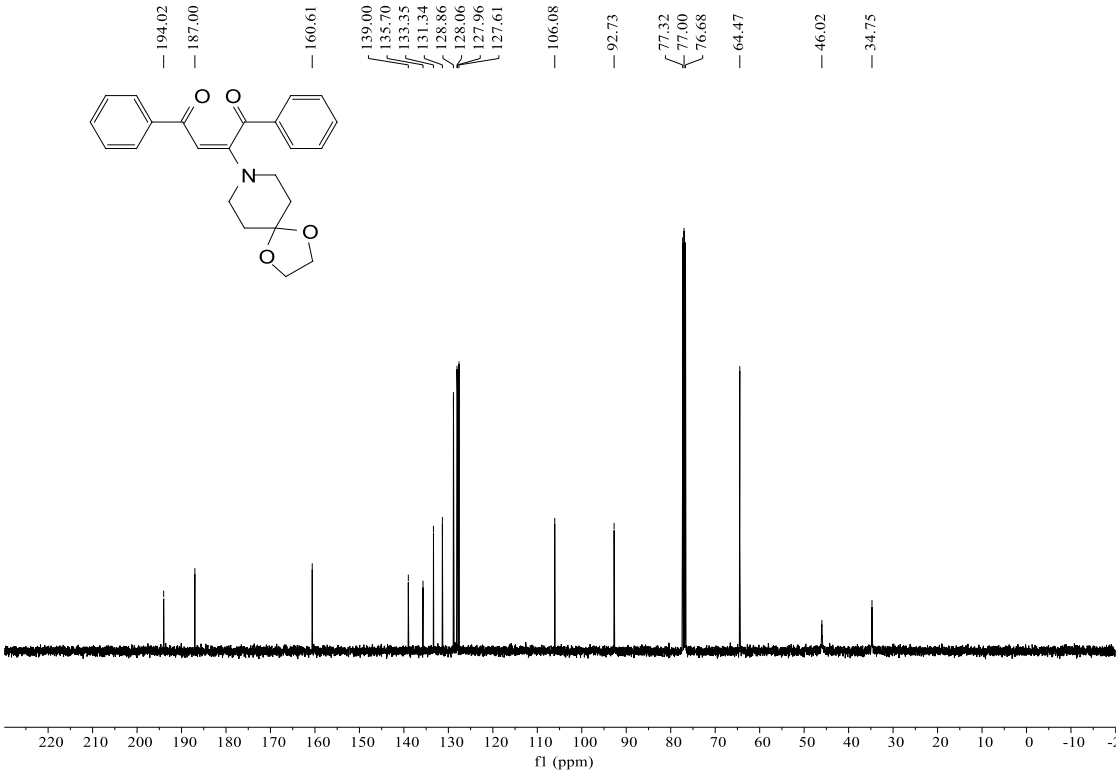


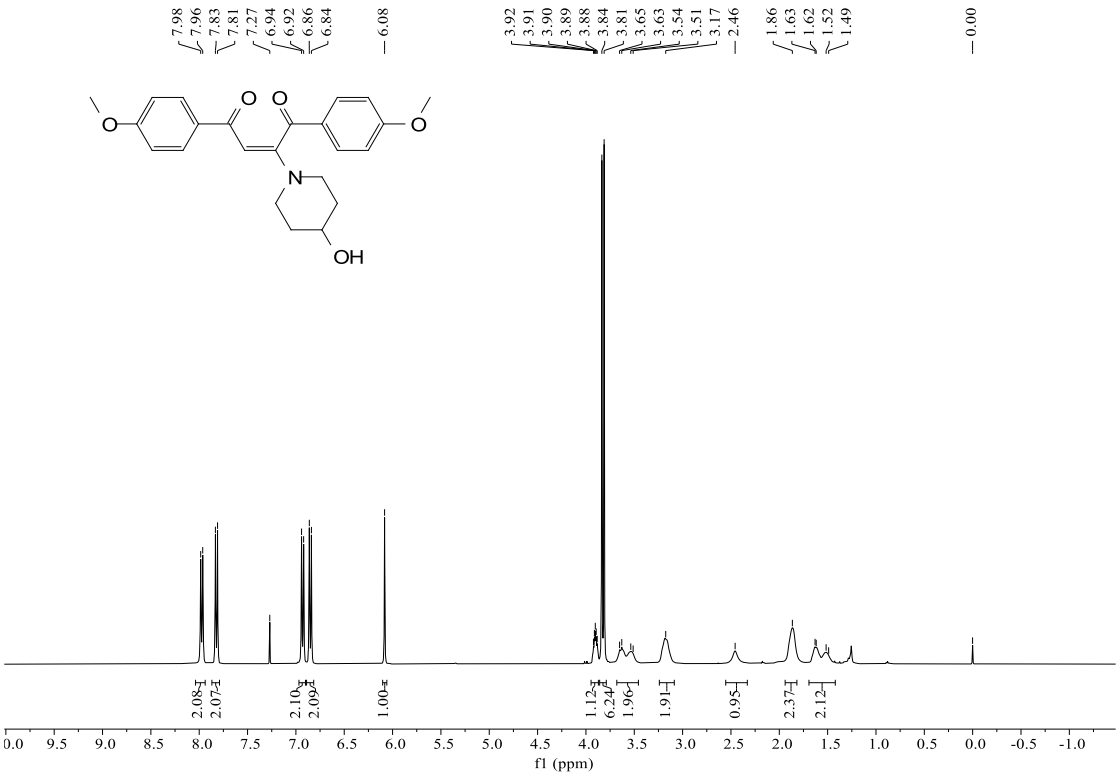
^{1}H NMR spectra of 3f (400 MHz, $CDCl_3$)



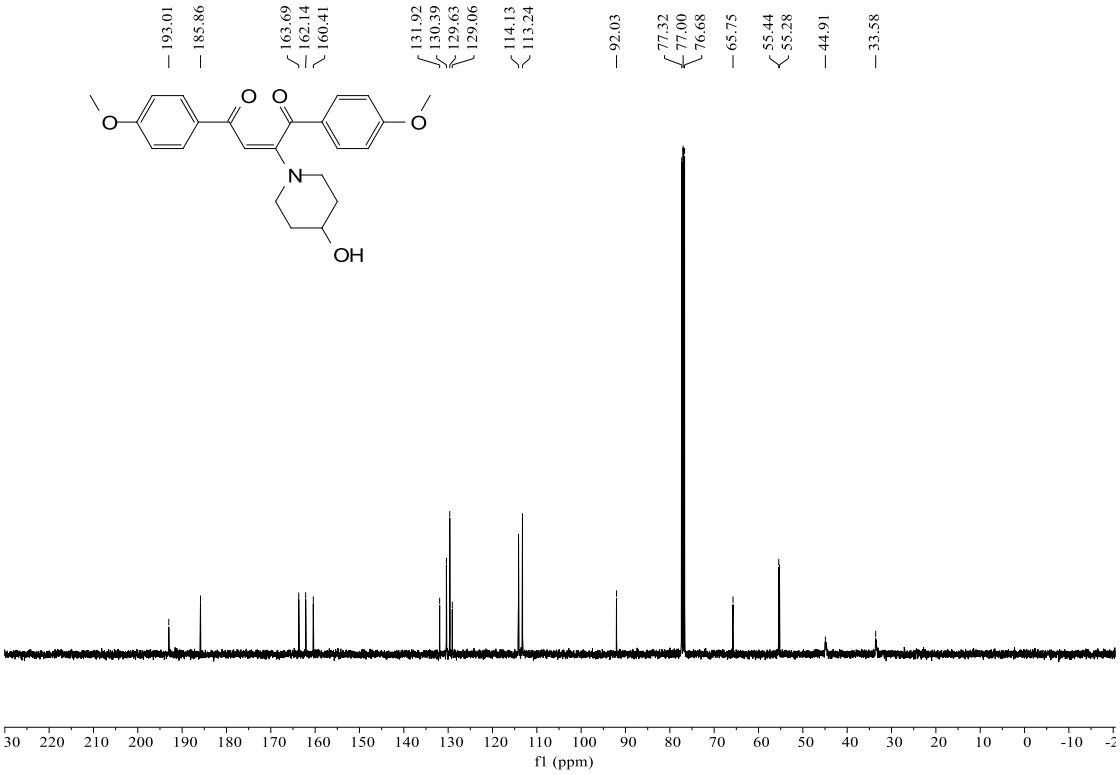
^{13}C NMR spectra of 3f (100 MHz, $CDCl_3$)



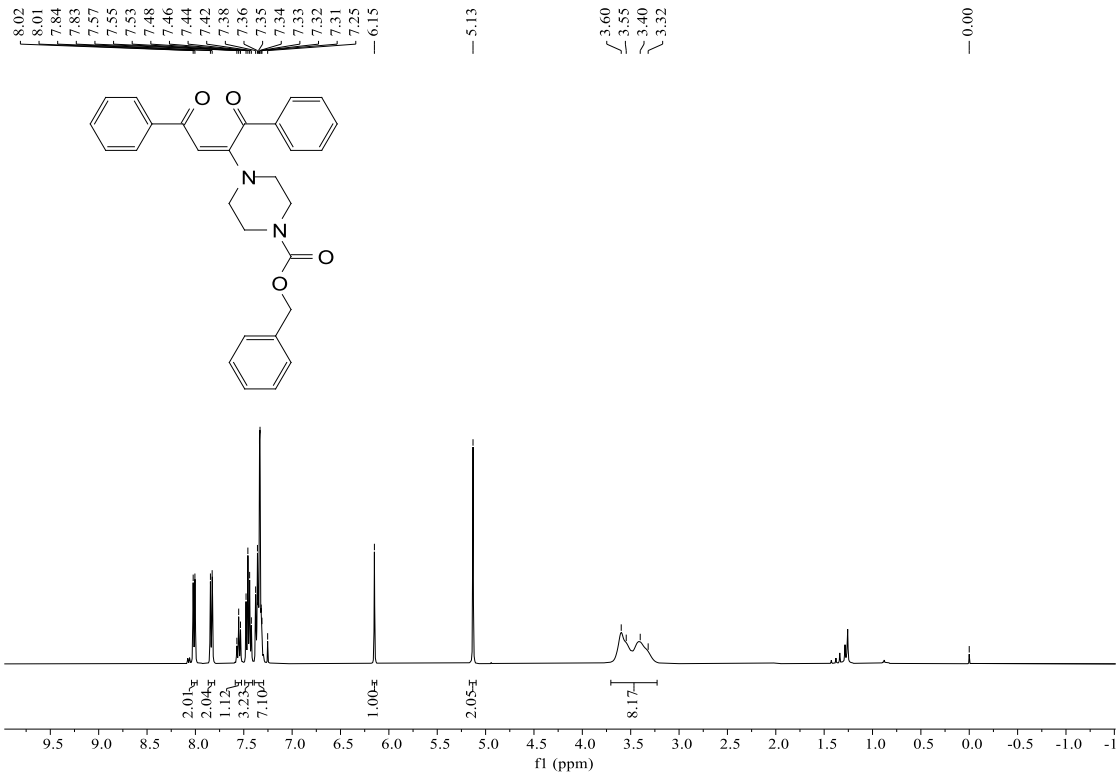
^{1}H NMR spectra of 3g (400 MHz, $CDCl_3$)



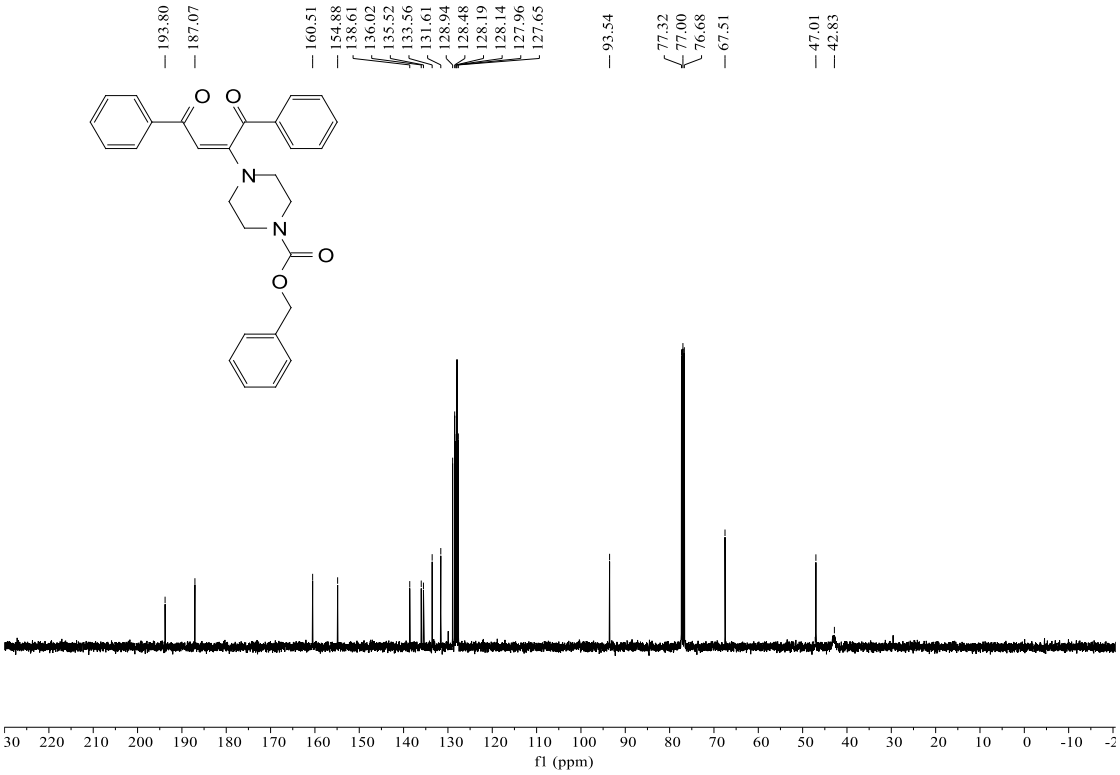
^{13}C NMR spectra of 3g (100 MHz, $CDCl_3$)



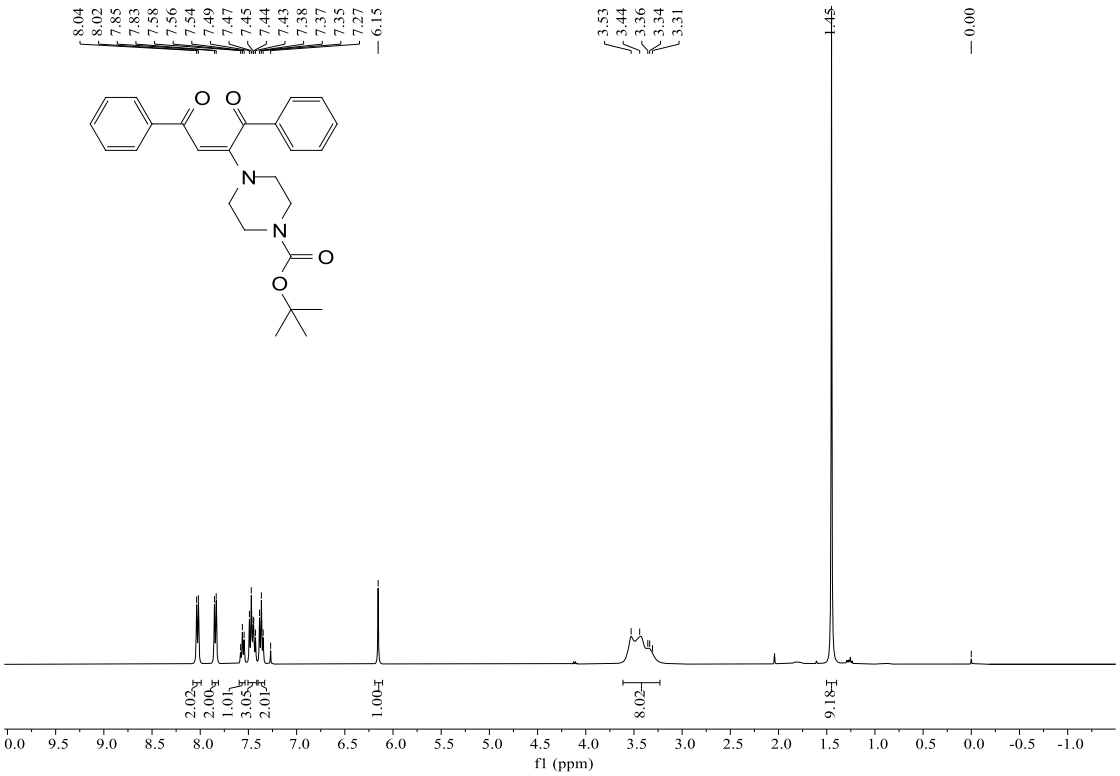
^{1}H NMR spectra of 3h (400 MHz, $CDCl_3$)



^{13}C NMR spectra of 3h (100 MHz, $CDCl_3$)



^{1}H NMR spectra of 3i (400 MHz, $CDCl_3$)



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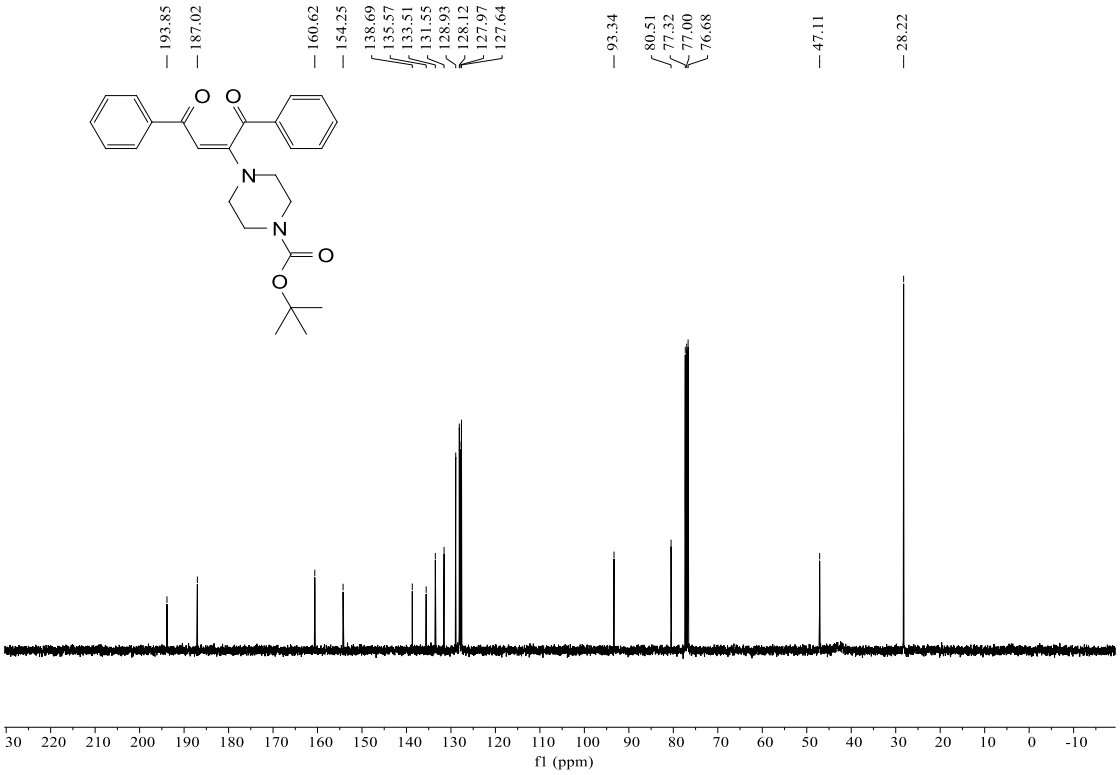

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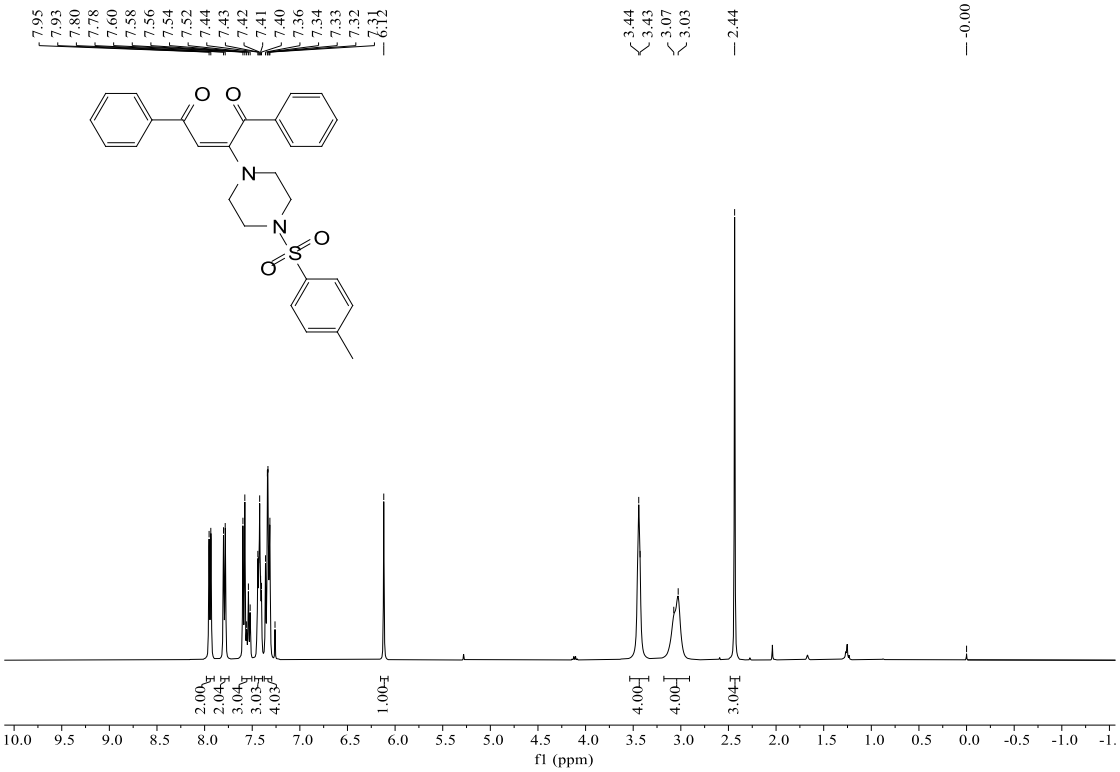

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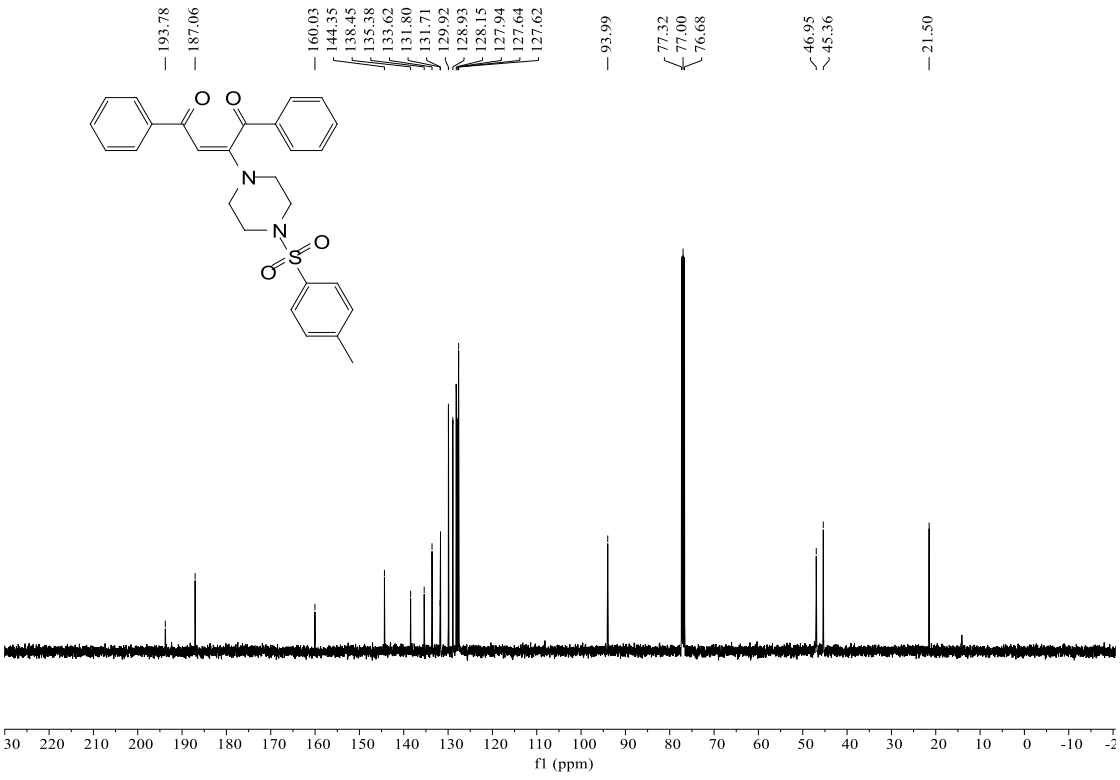
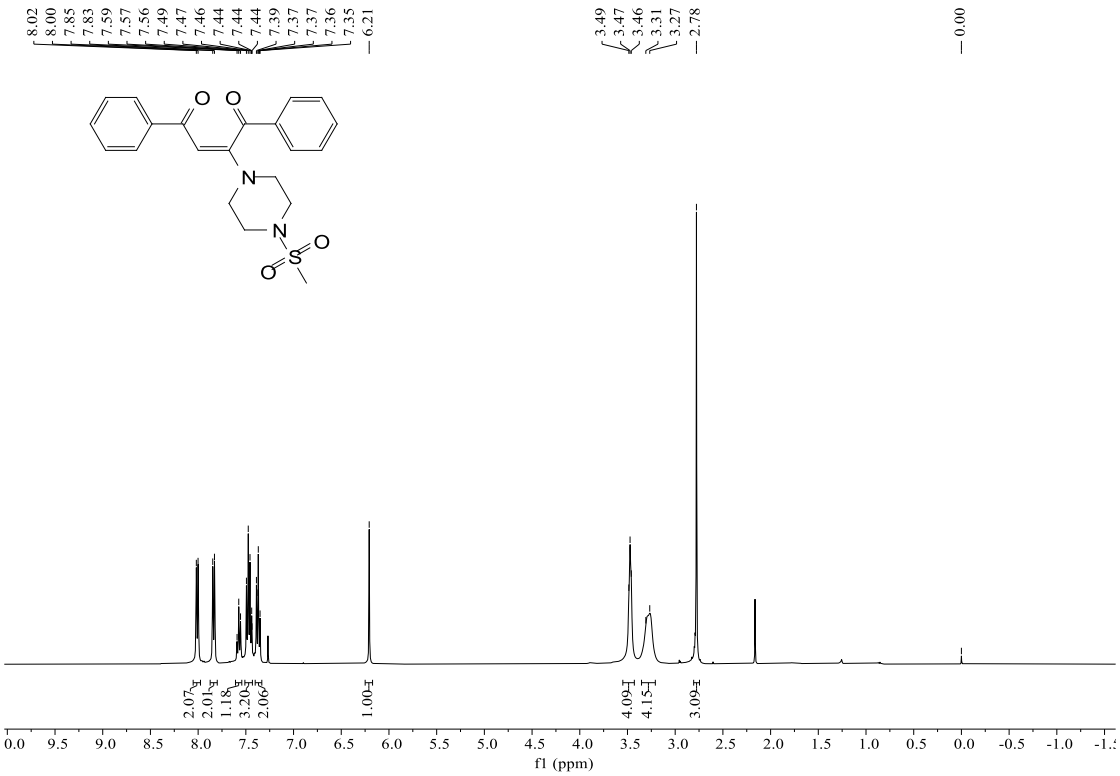


^{1}H NMR spectra of 3k (400 MHz, $CDCl_3$)



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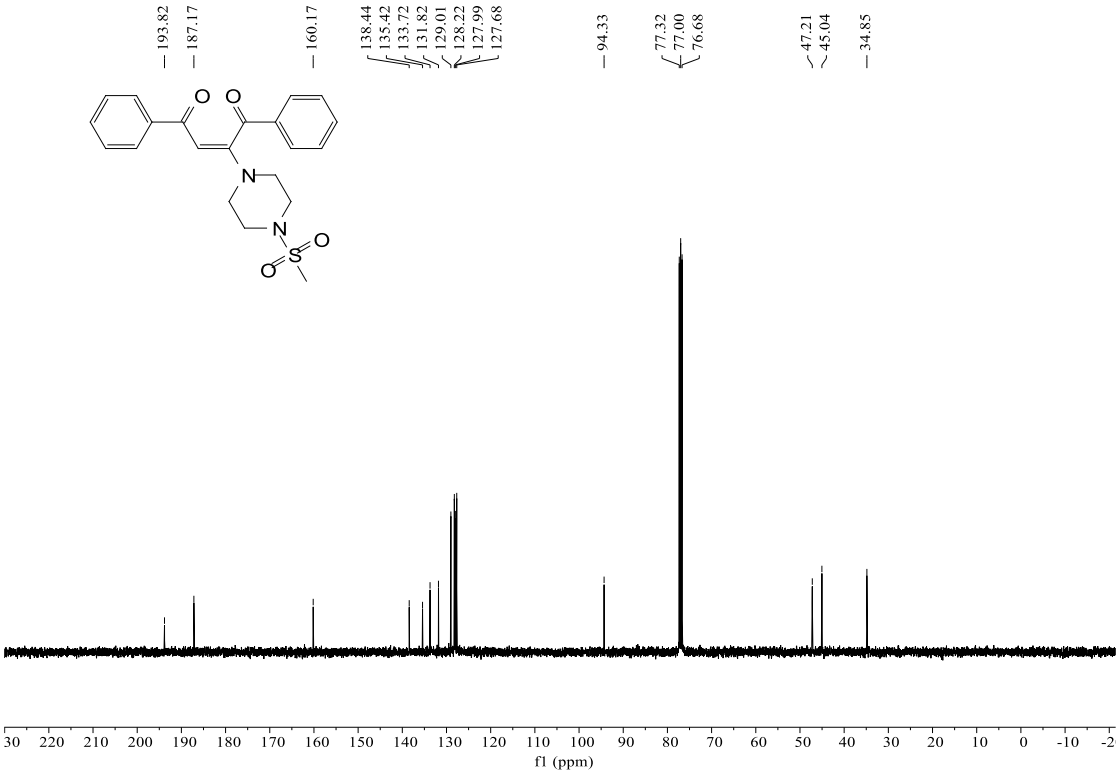
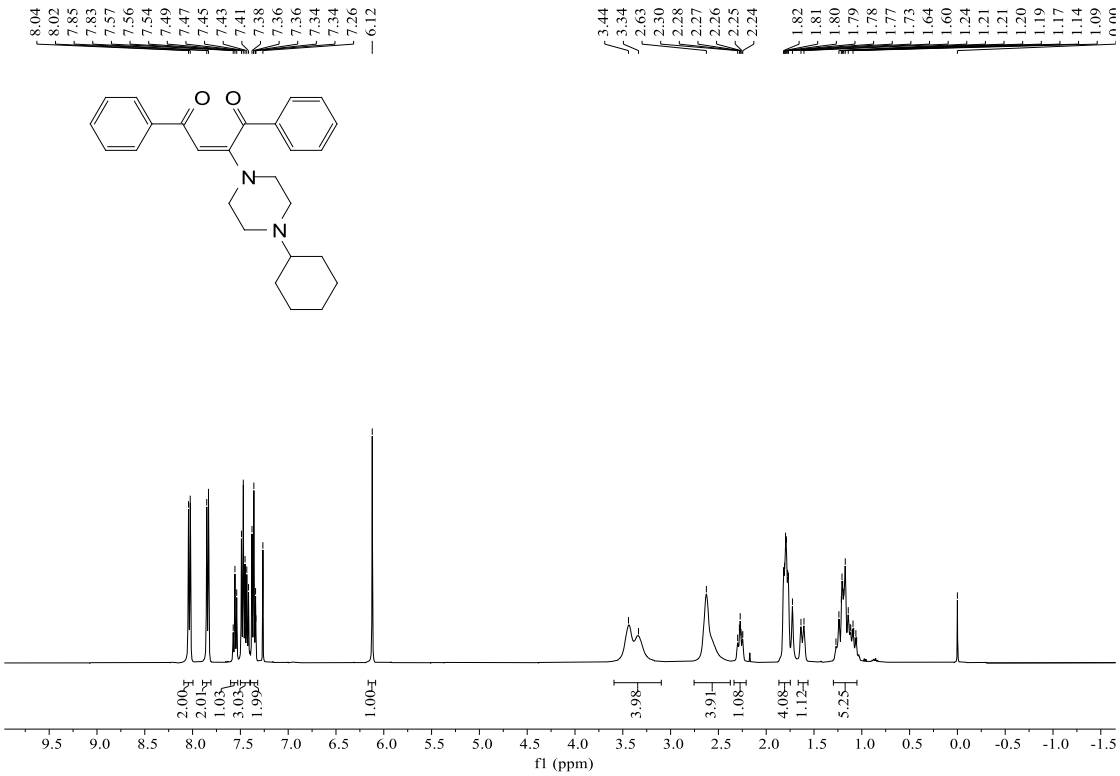


^{1}H NMR spectra of 3l (400 MHz, $CDCl_3$)



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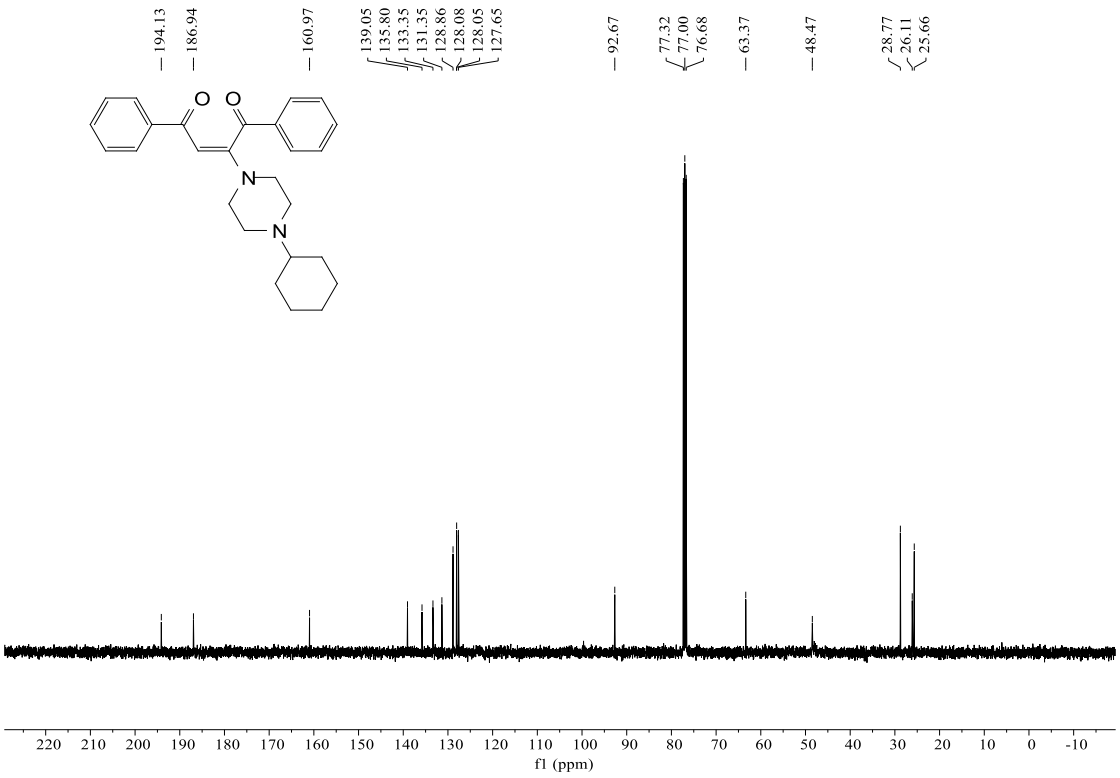
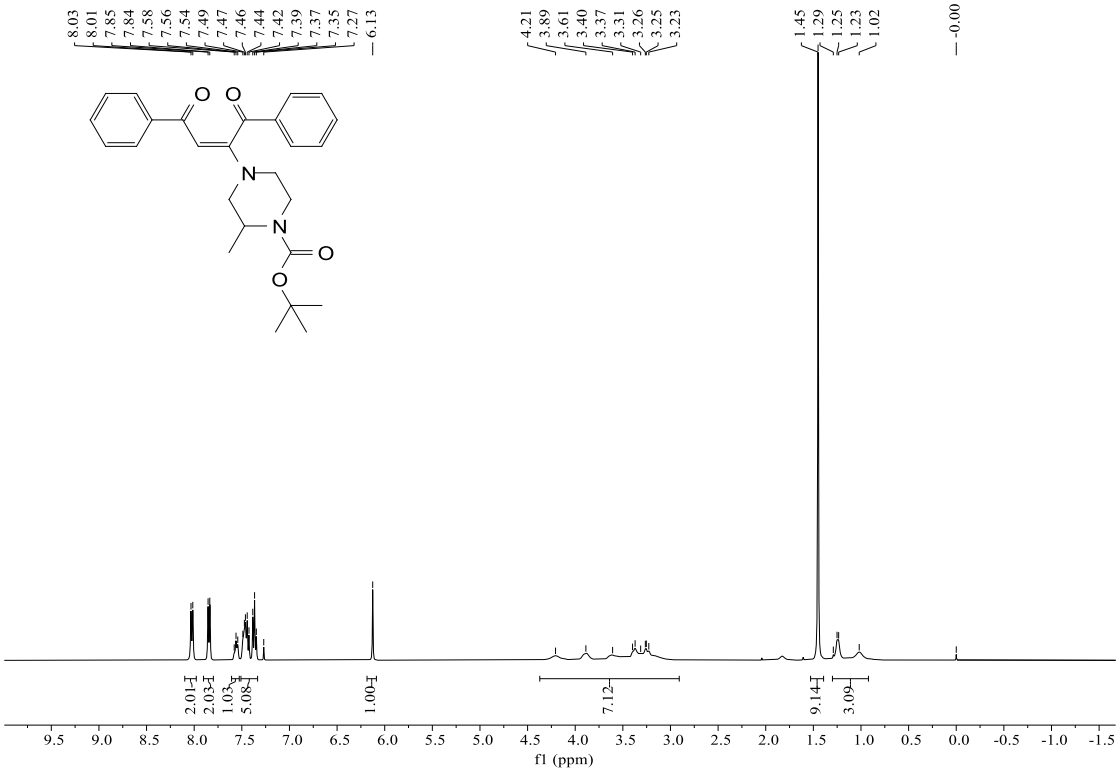
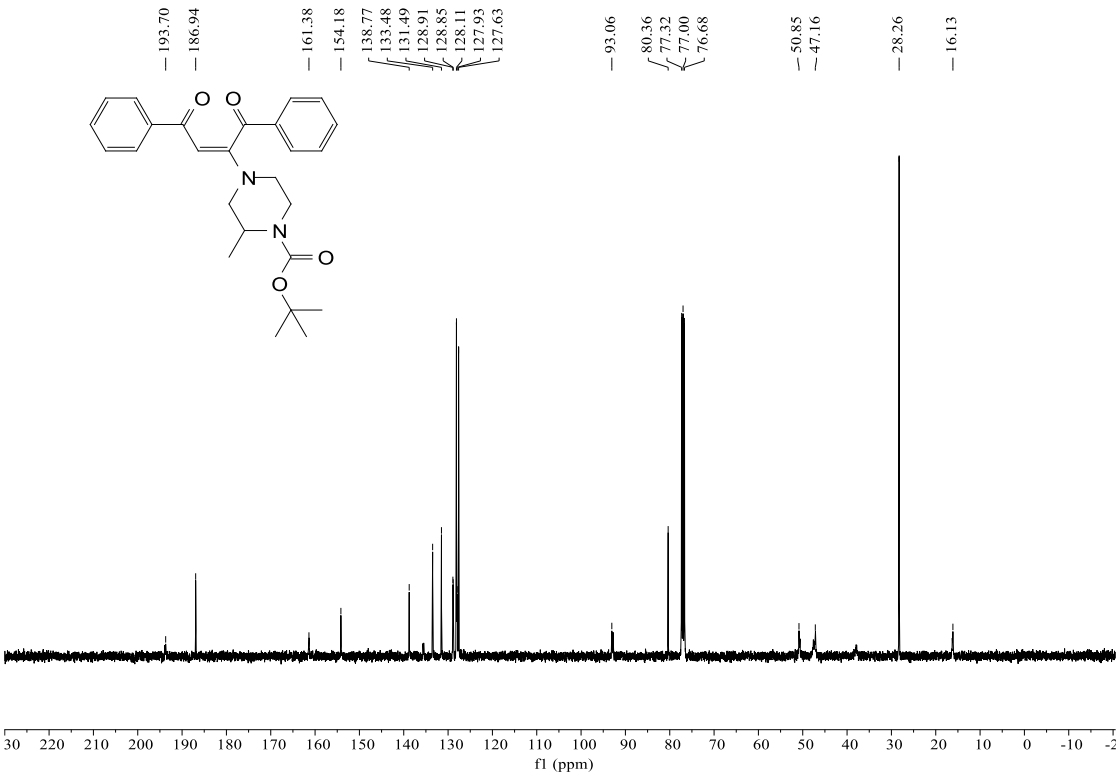


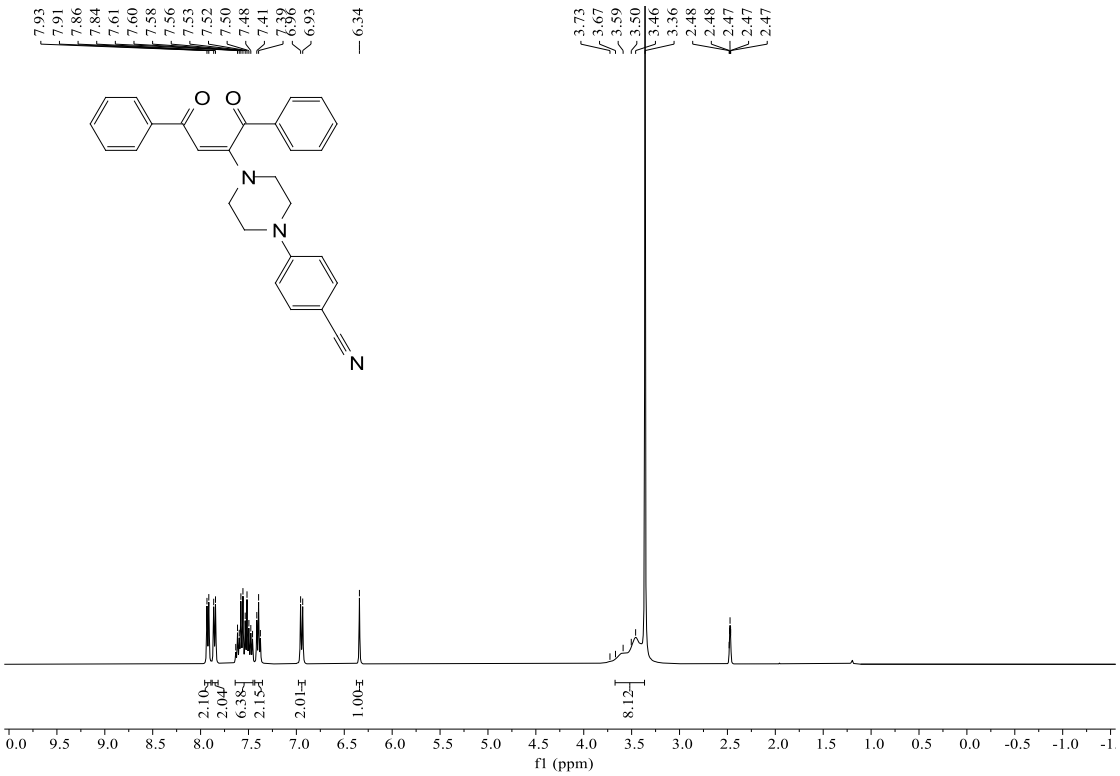
^{1}H NMR spectra of 3m (400 MHz, $CDCl_3$)



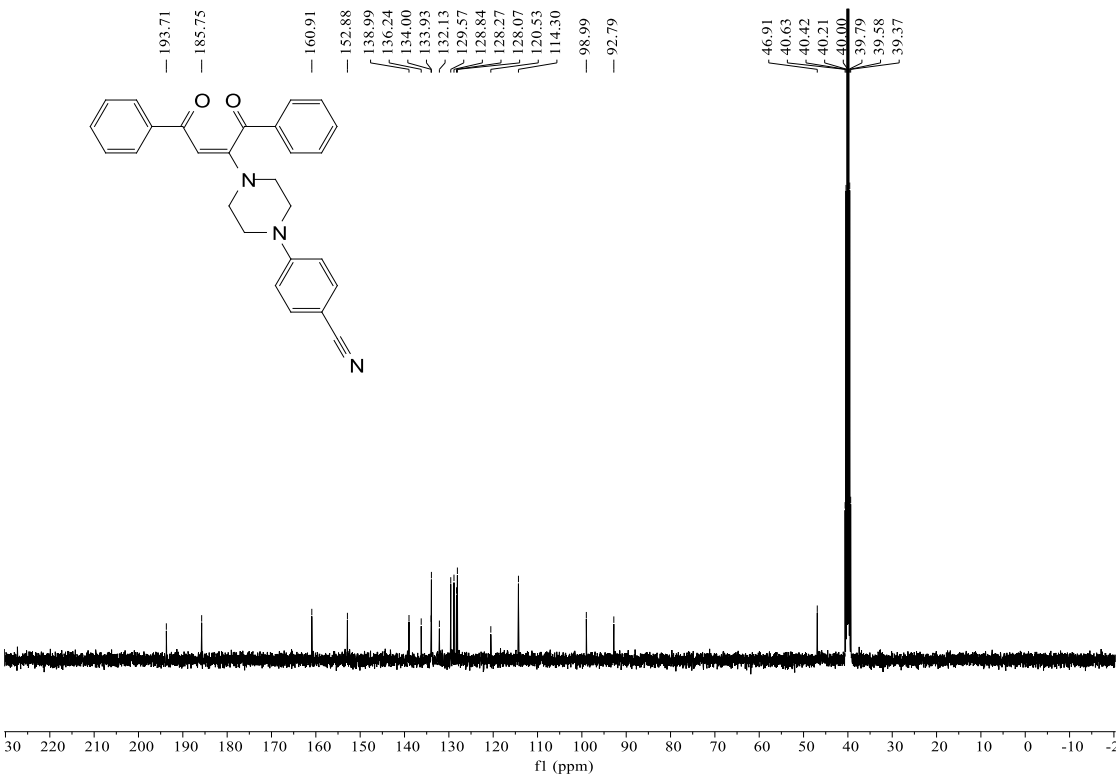
^{13}C NMR spectra of 3m (100 MHz, $CDCl_3$)



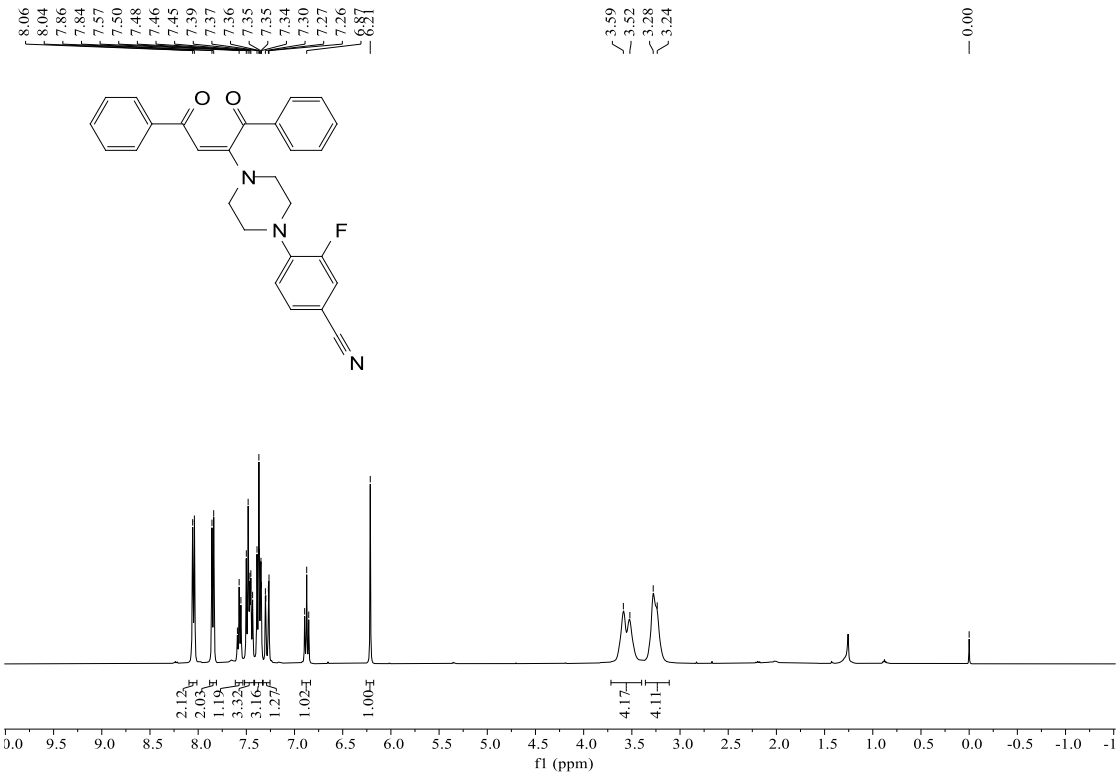
^{1}H NMR spectra of 3n (400 MHz, DMSO-d_6)



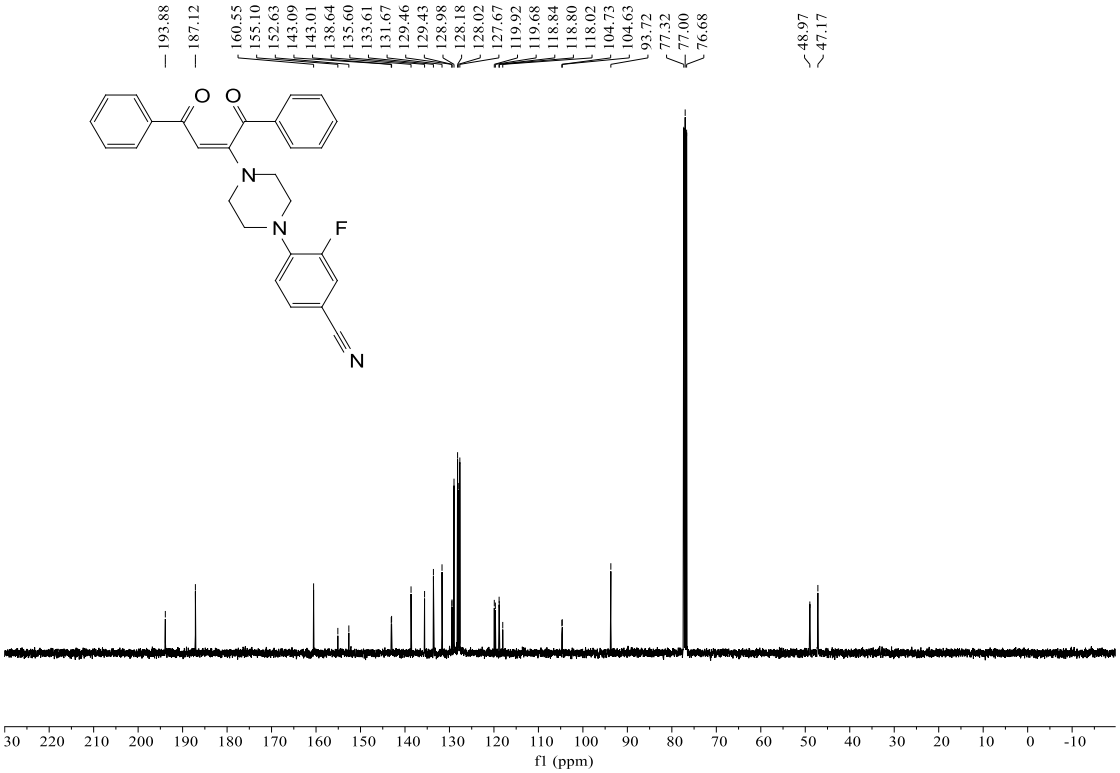
^{13}C NMR spectra of 3n (100 MHz, DMSO-d_6)



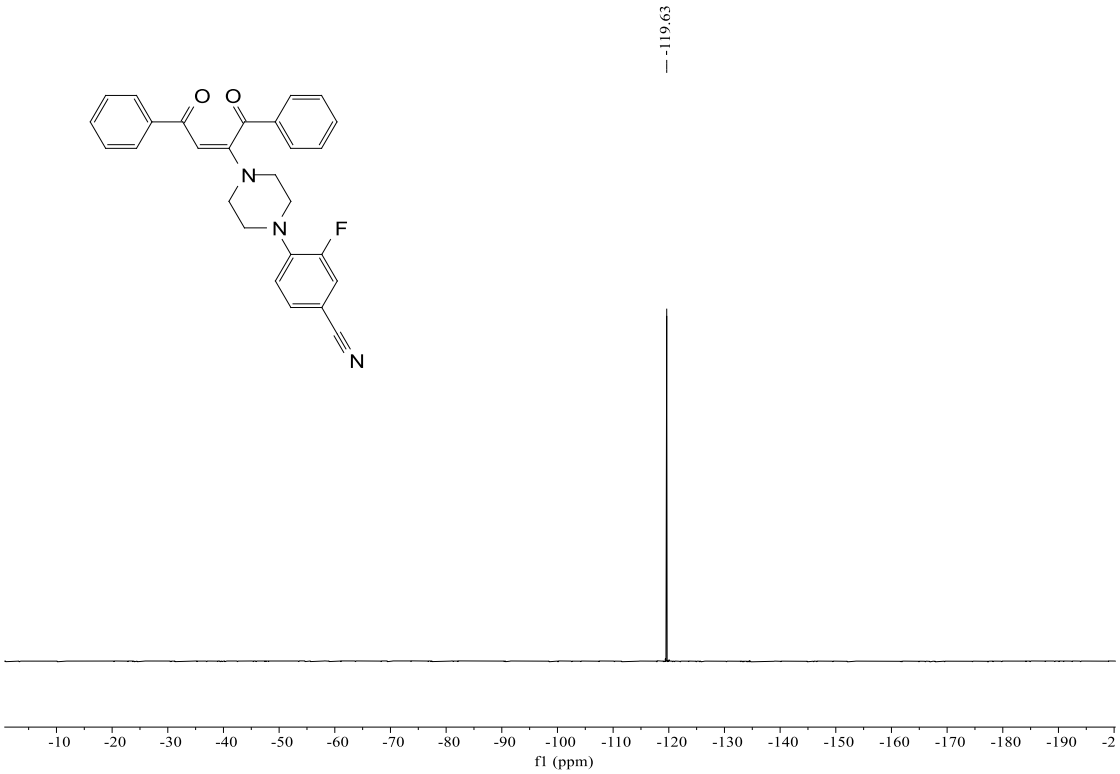
^{1}H NMR spectra of 3o (400 MHz, $CDCl_3$)



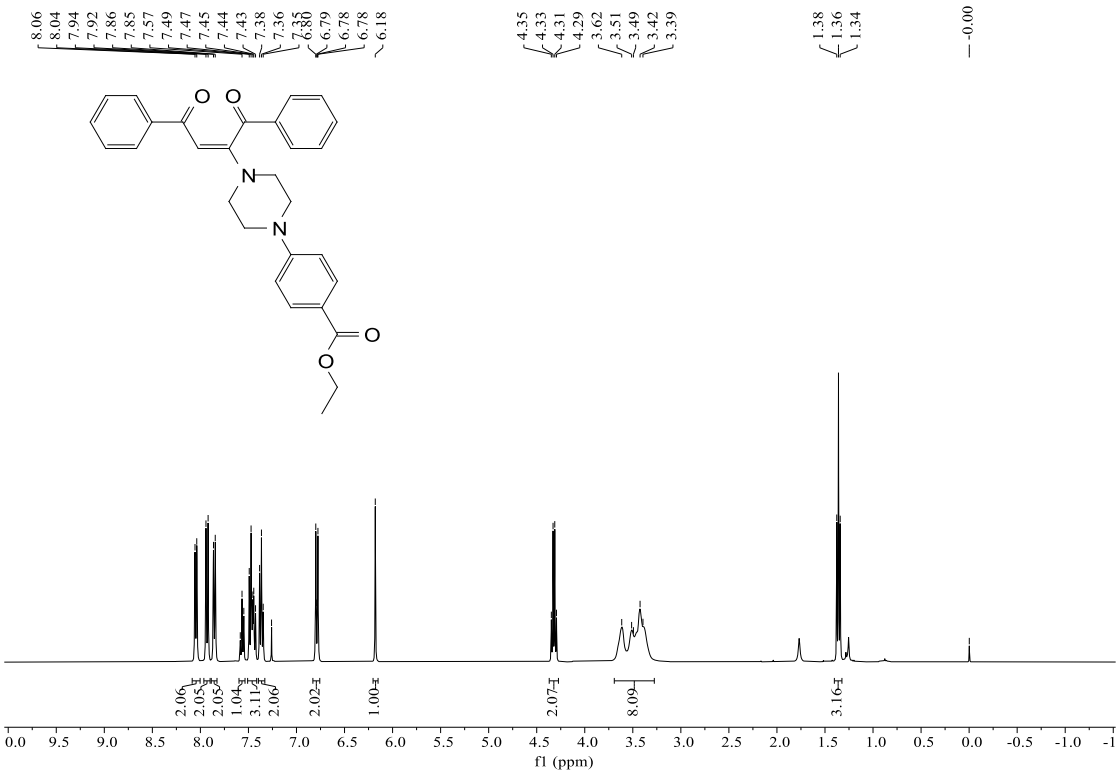
^{13}C NMR spectra of 3o (100 MHz, $CDCl_3$)



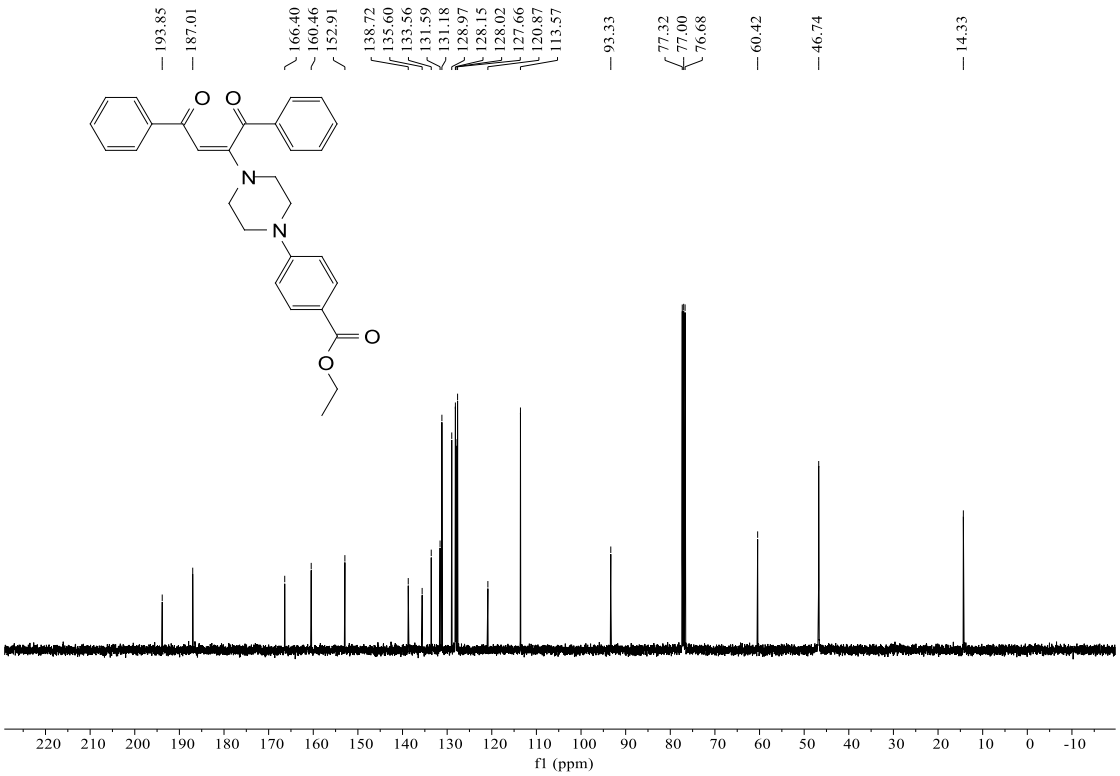
^{19}F NMR spectra of 3o (376 MHz, $CDCl_3$)



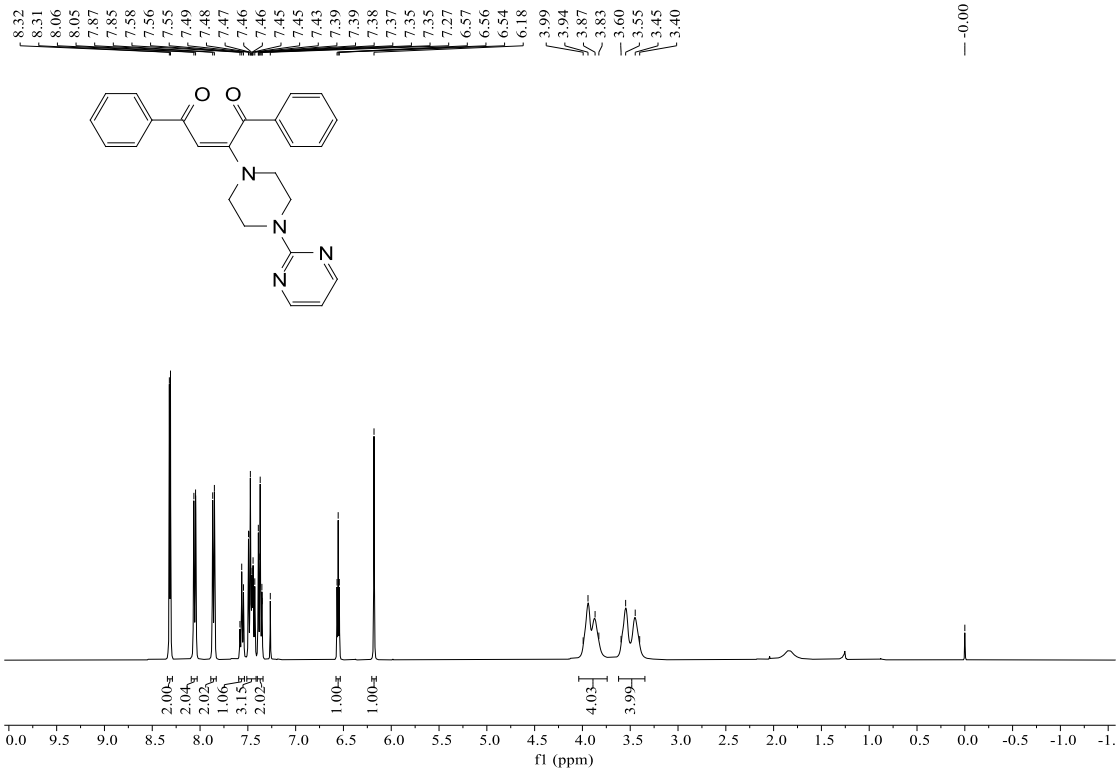
^{1}H NMR spectra of 3p (400 MHz, $CDCl_3$)



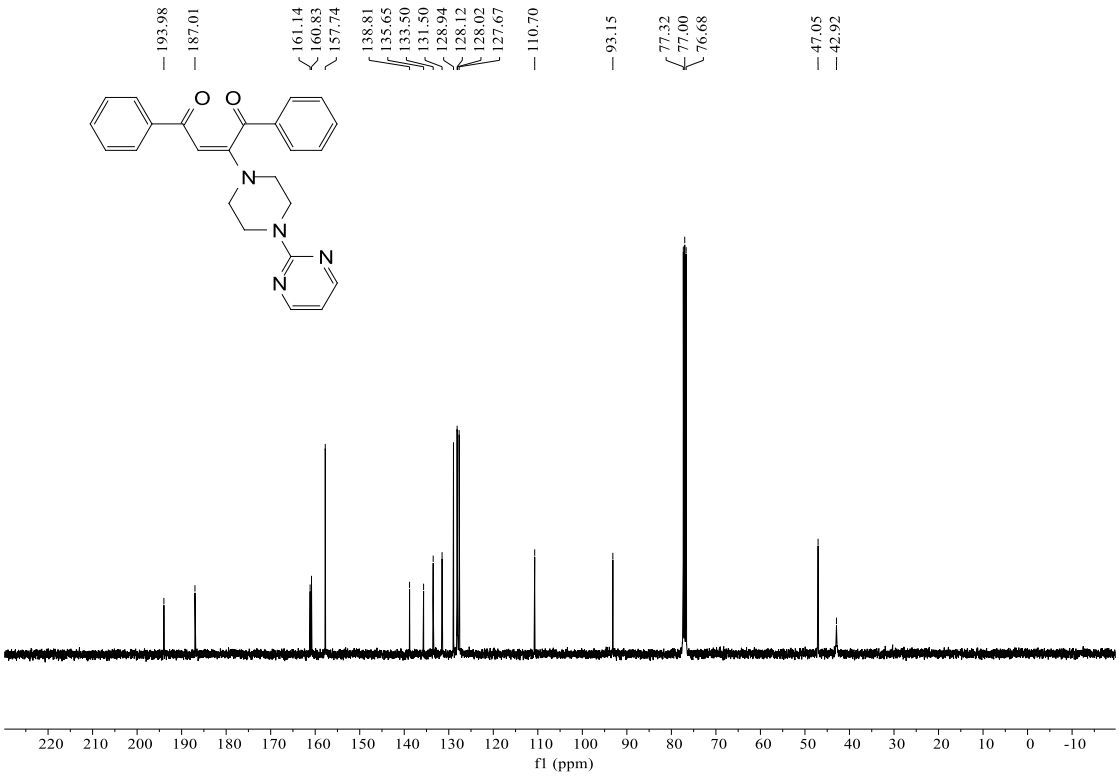
^{13}C NMR spectra of 3p (100 MHz, $CDCl_3$)



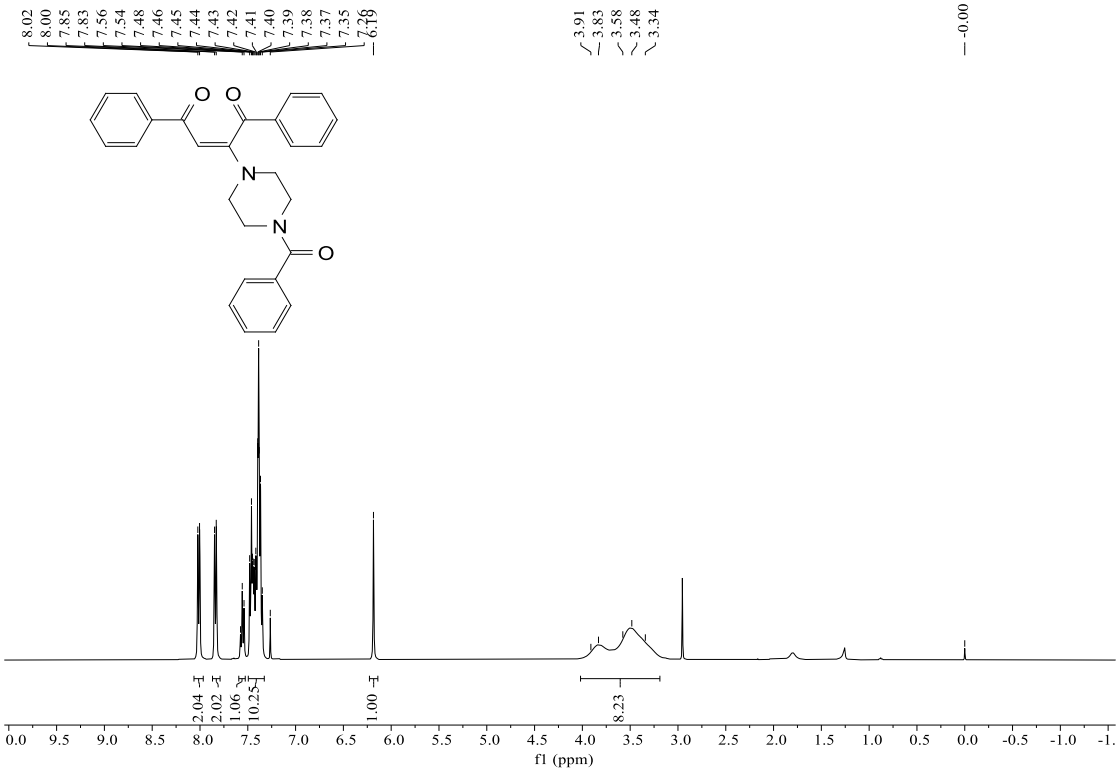
^{1}H NMR spectra of 3q (400 MHz, $CDCl_3$)



^{13}C NMR spectra of 3q (100 MHz, $CDCl_3$)



^{1}H NMR spectra of 3r (400 MHz, $CDCl_3$)



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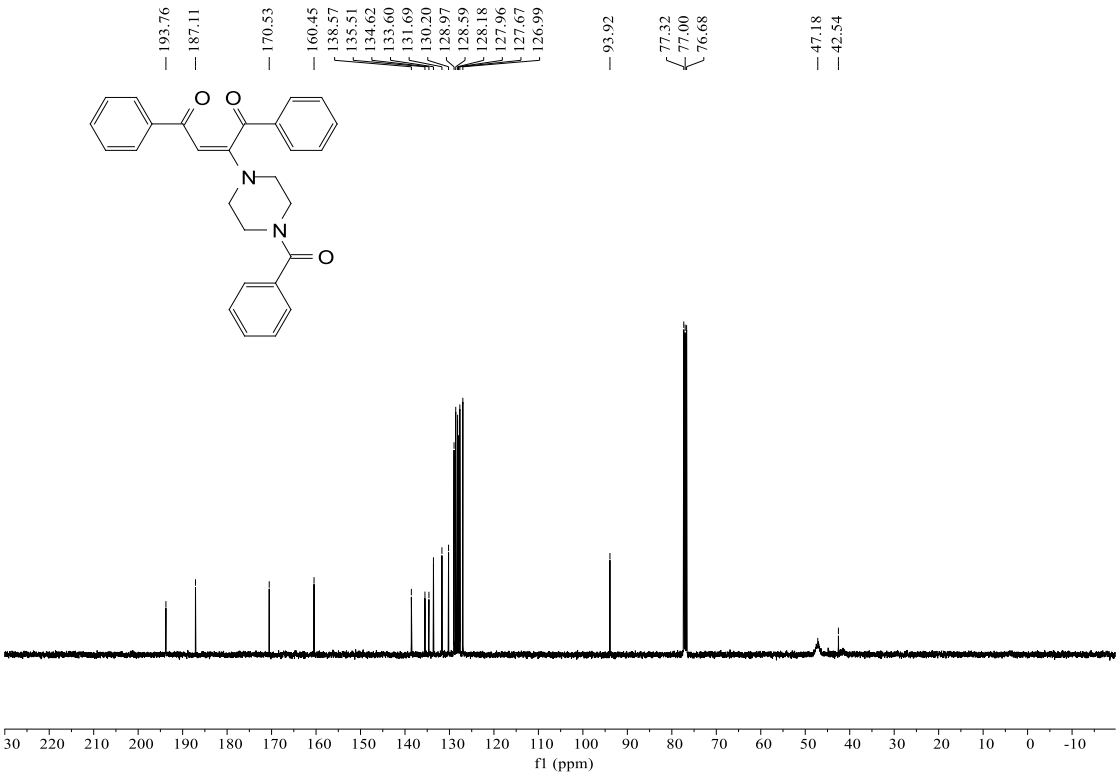
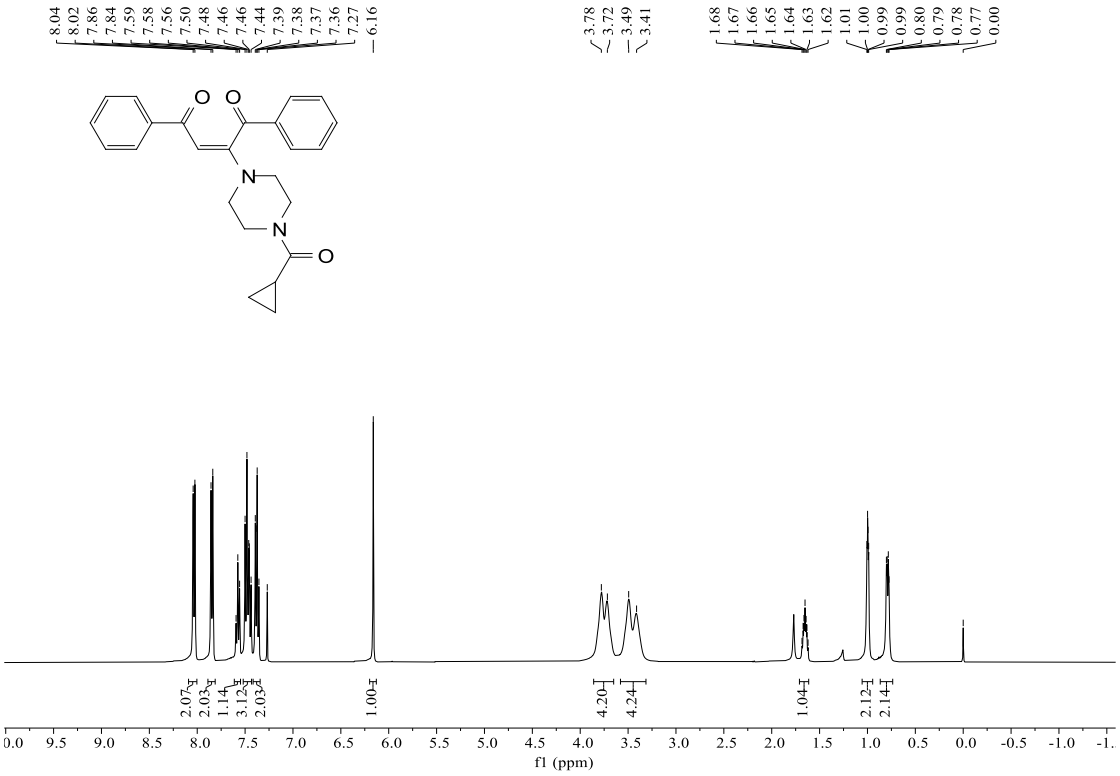
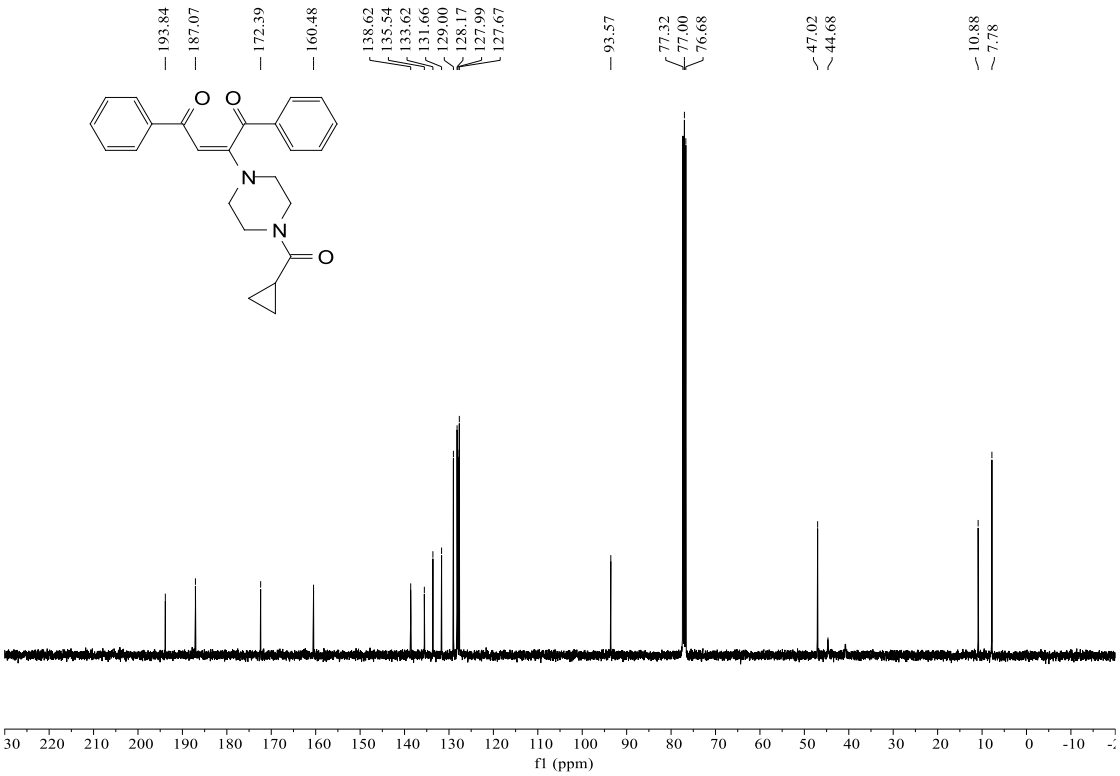


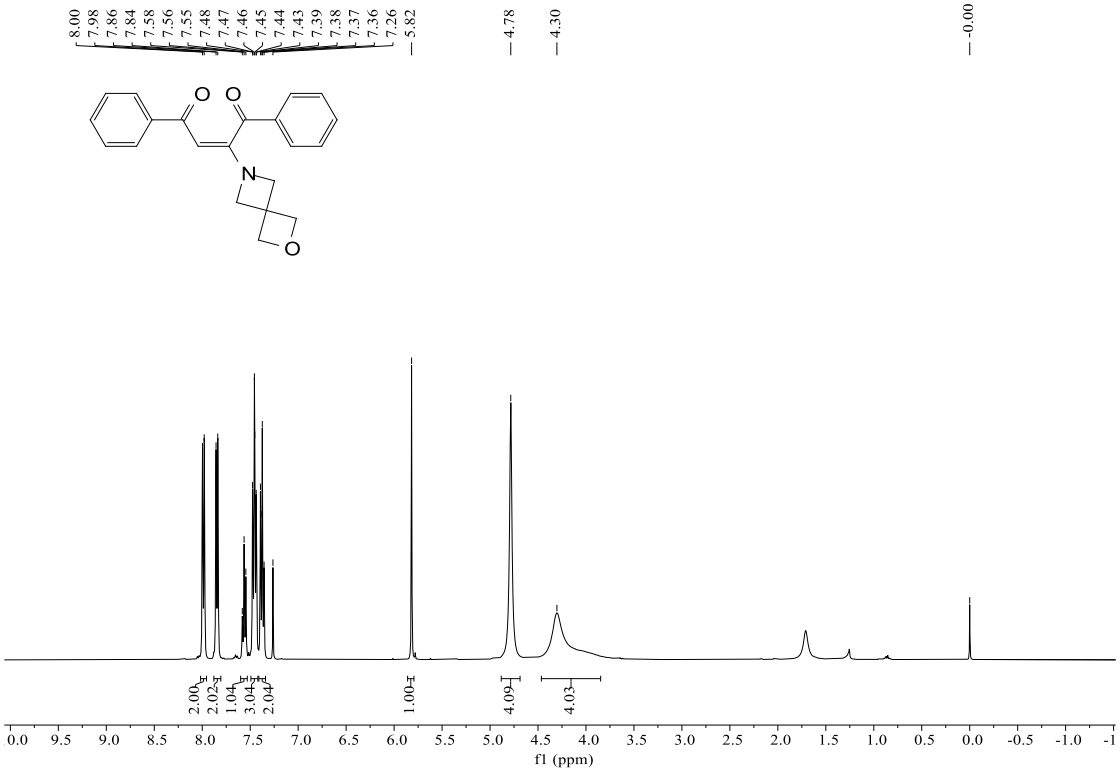
^{1}H NMR spectra of 3s (400 MHz, $CDCl_3$)



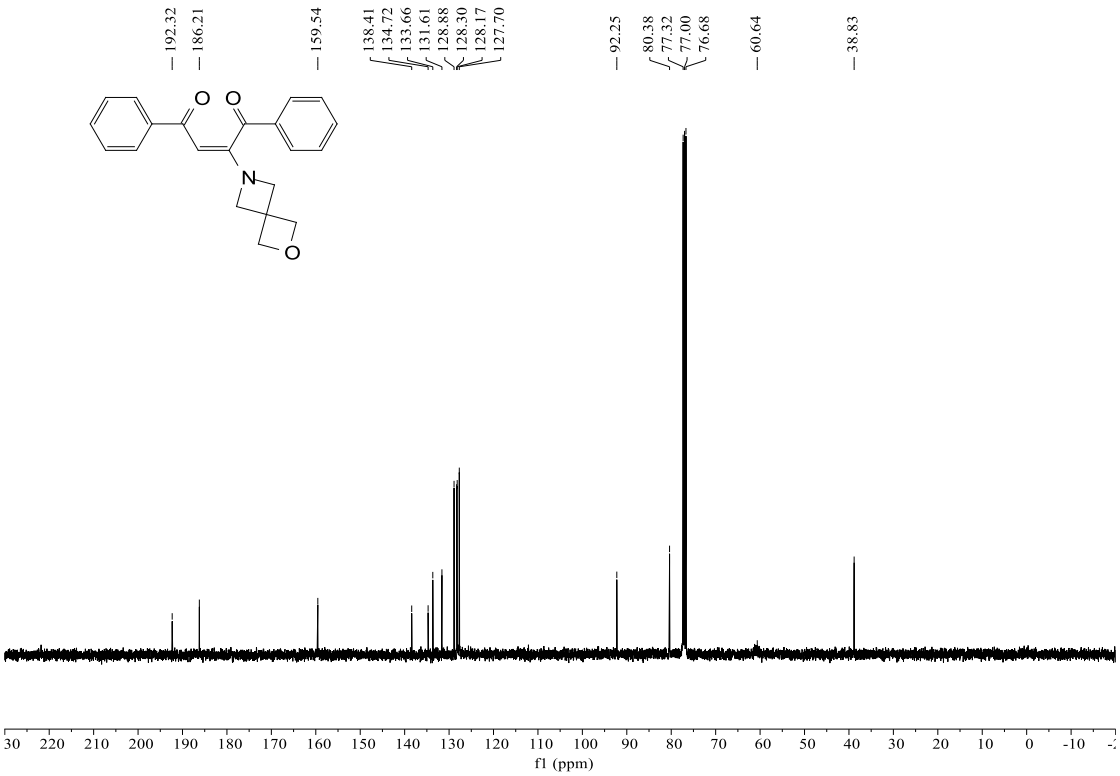
^{13}C NMR spectra of 3s (100 MHz, $CDCl_3$)



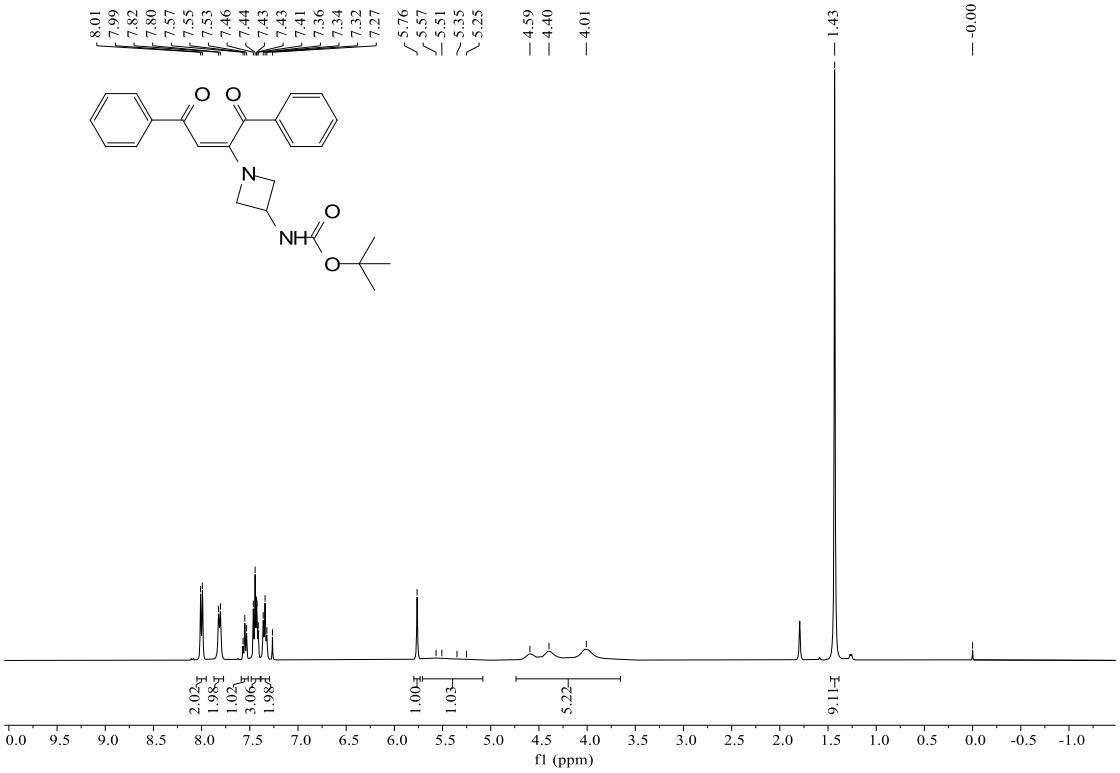
^{1}H NMR spectra of 3t (400 MHz, $CDCl_3$)



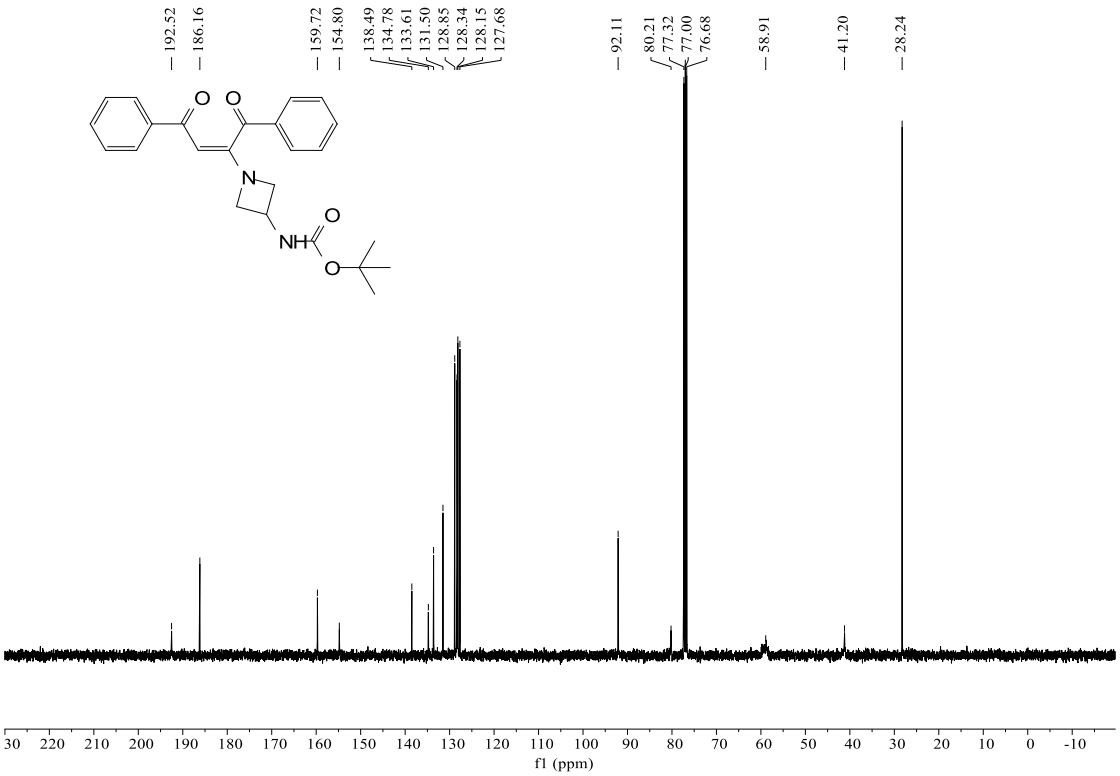
^{13}C NMR spectra of 3t (100 MHz, $CDCl_3$)



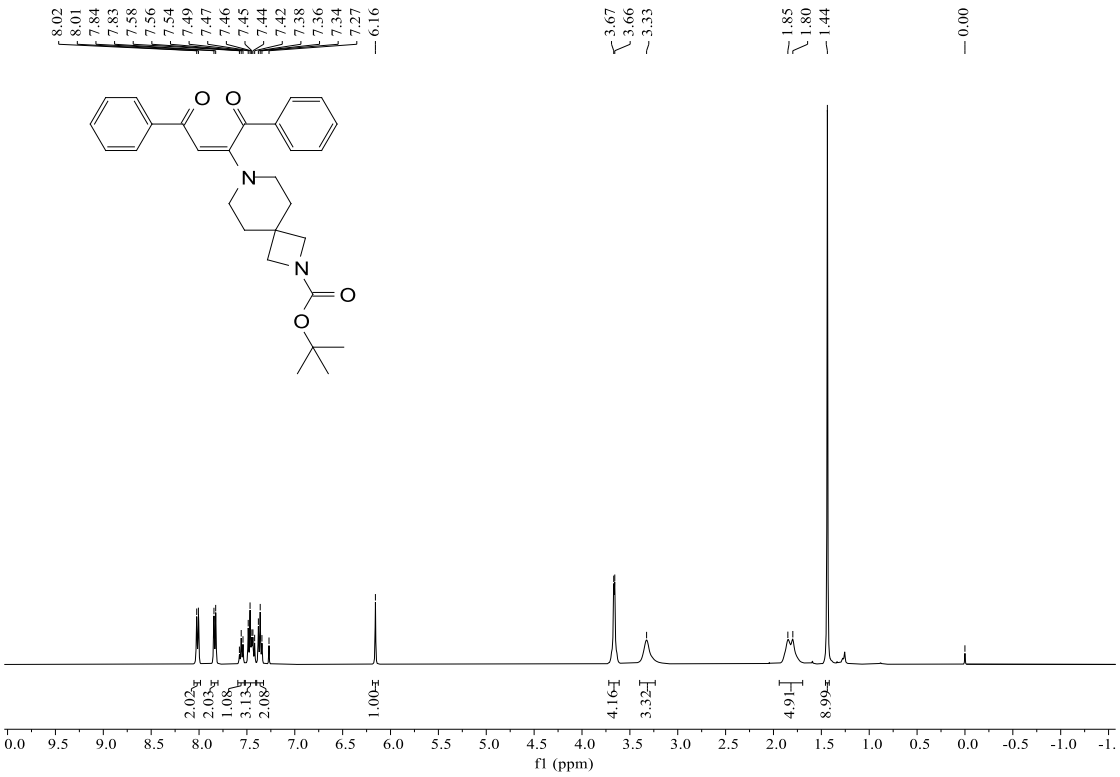
^{1}H NMR spectra of 3u (400 MHz, $CDCl_3$)



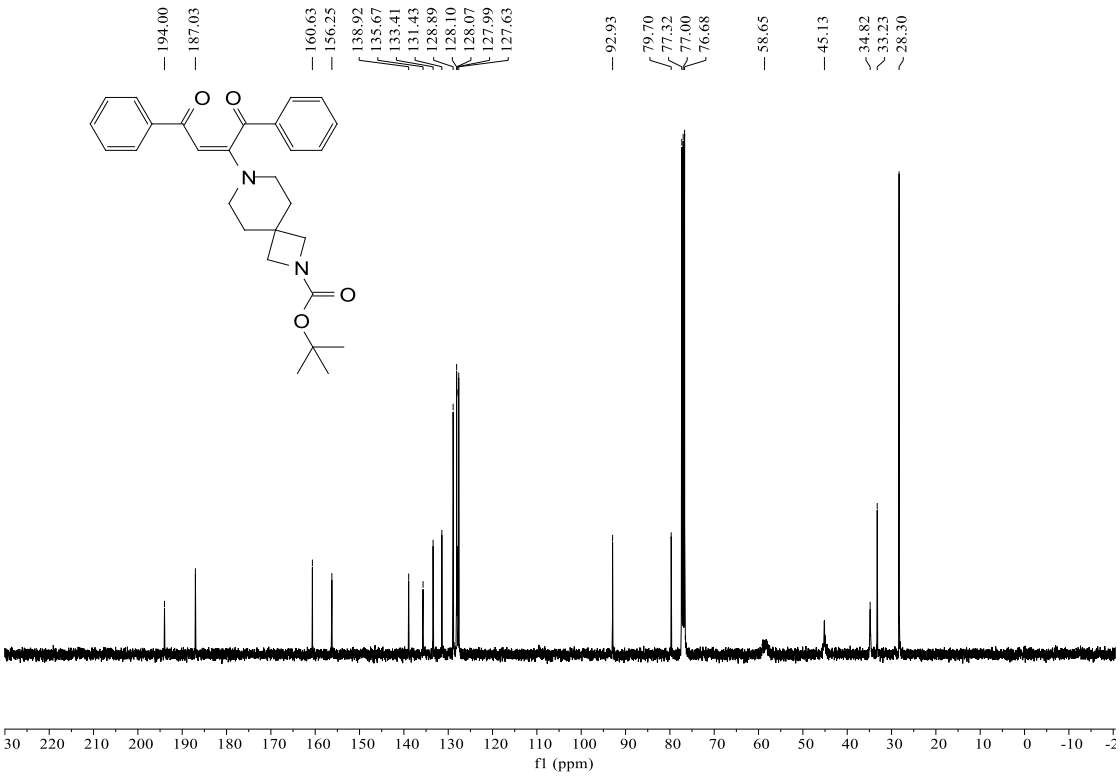
^{13}C NMR spectra of 3u (100 MHz, $CDCl_3$)



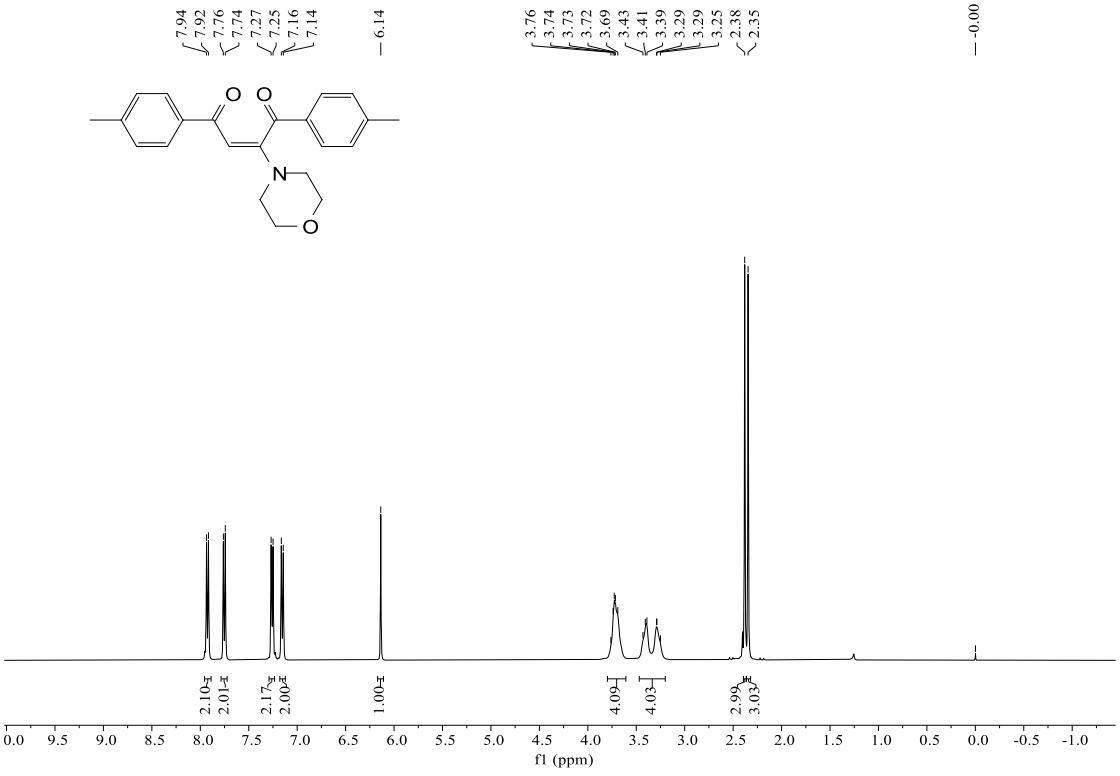
^{1}H NMR spectra of 3v (400 MHz, $CDCl_3$)



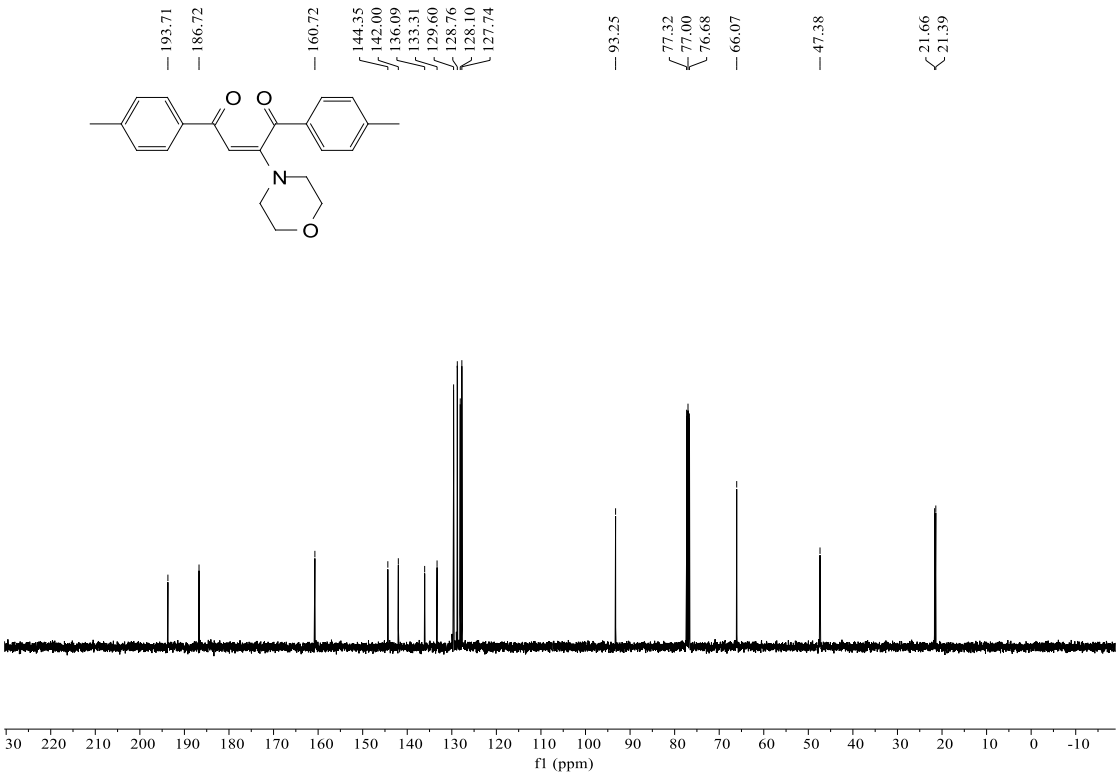
^{13}C NMR spectra of 3v (100 MHz, $CDCl_3$)



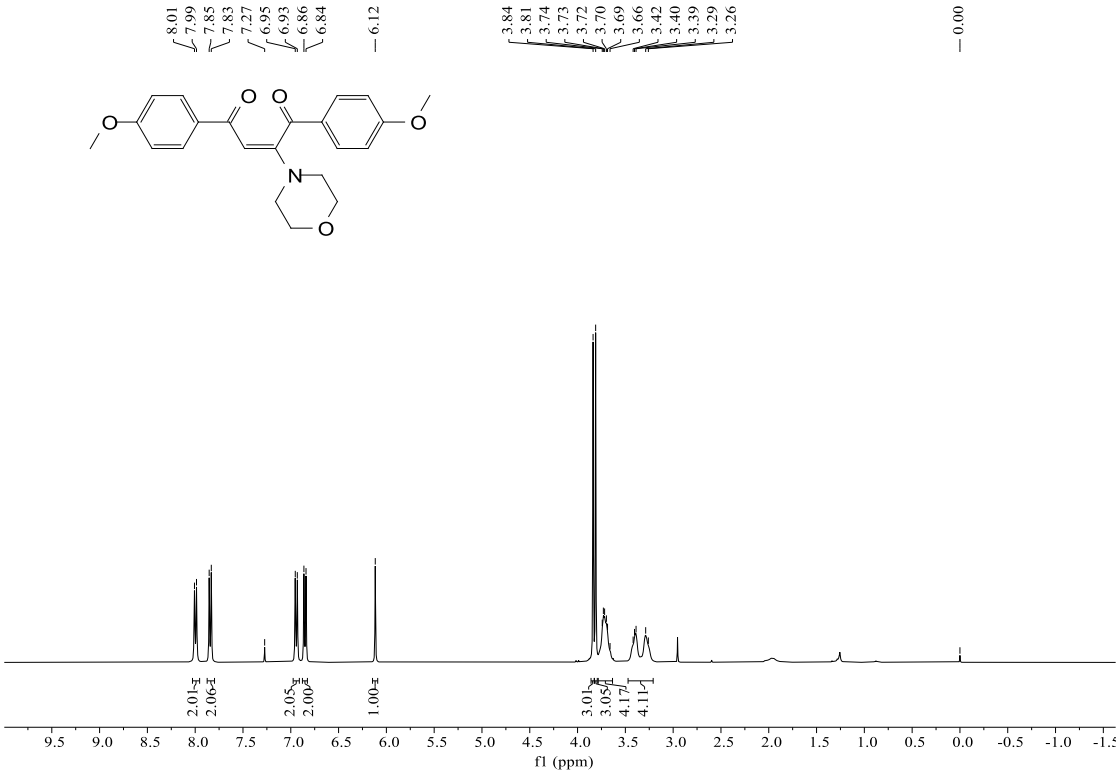
^{1}H NMR spectra of 3w (400 MHz, $CDCl_3$)



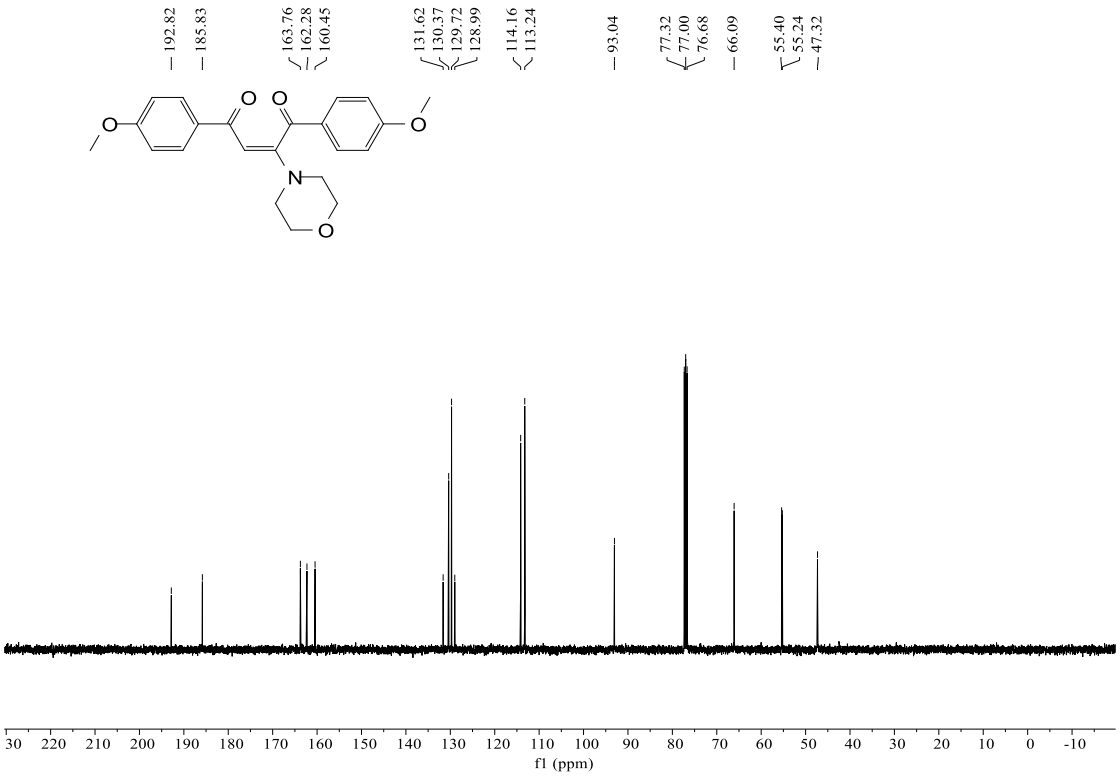
^{13}C NMR spectra of 3w (100 MHz, $CDCl_3$)



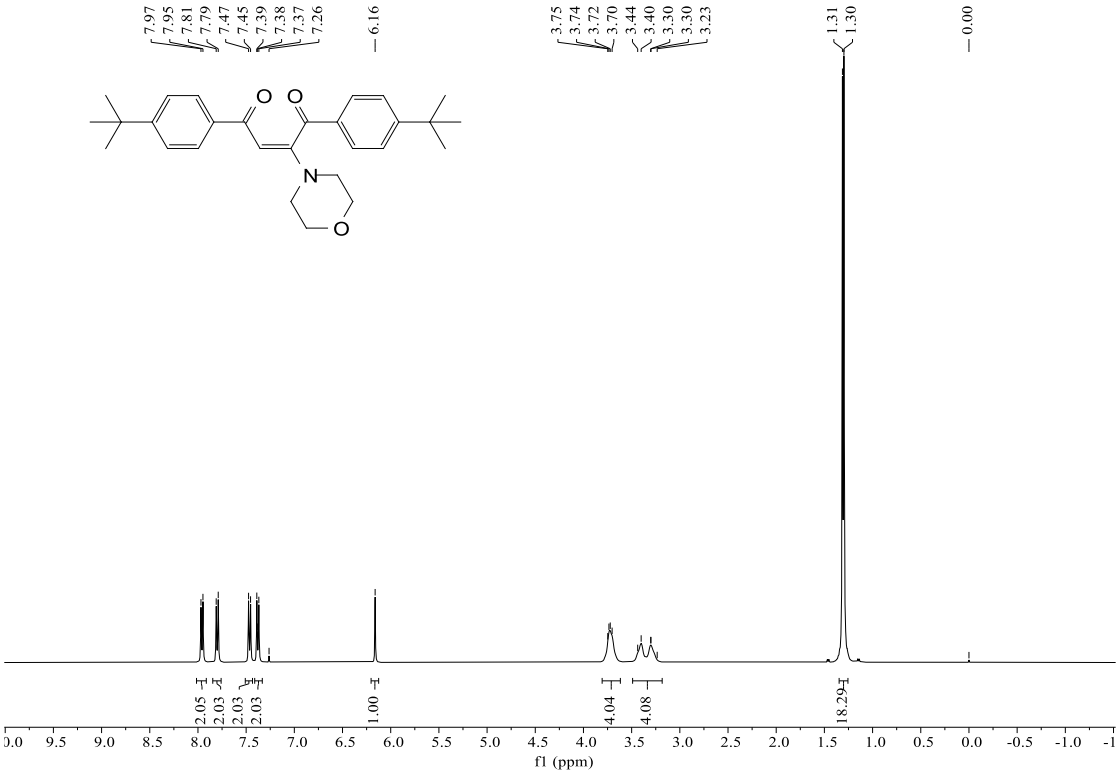
^{1}H NMR spectra of 3x (400 MHz, $CDCl_3$)



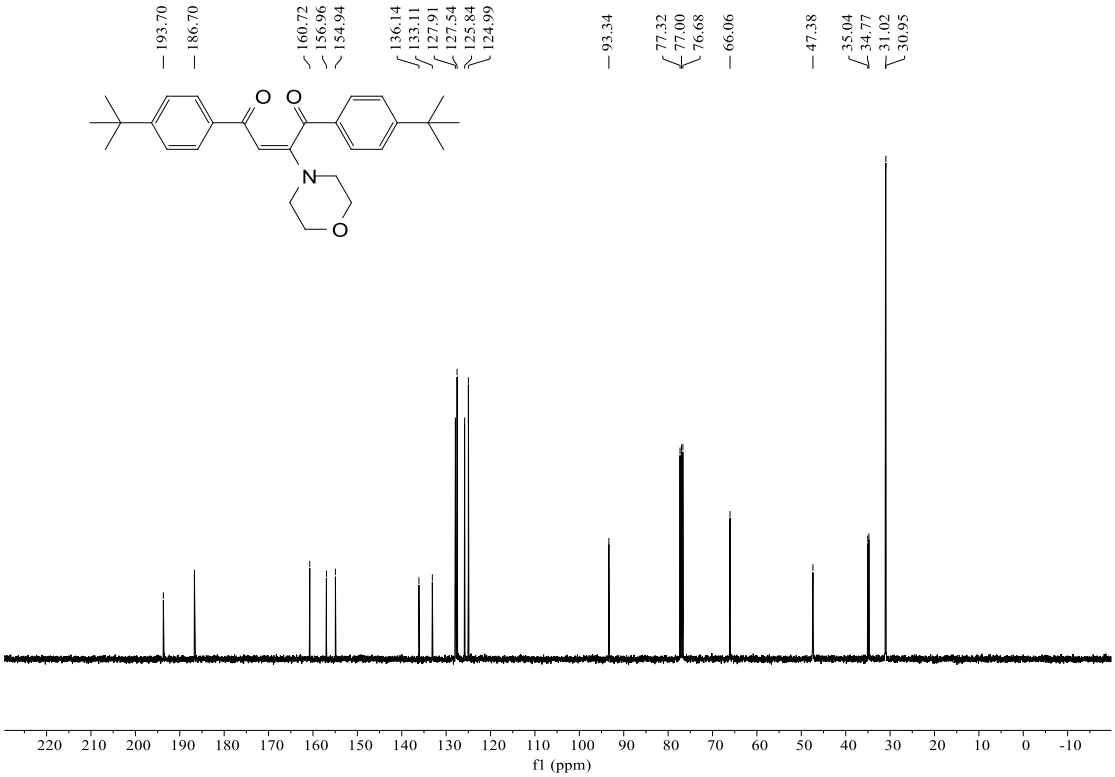
^{13}C NMR spectra of 3x (100 MHz, $CDCl_3$)



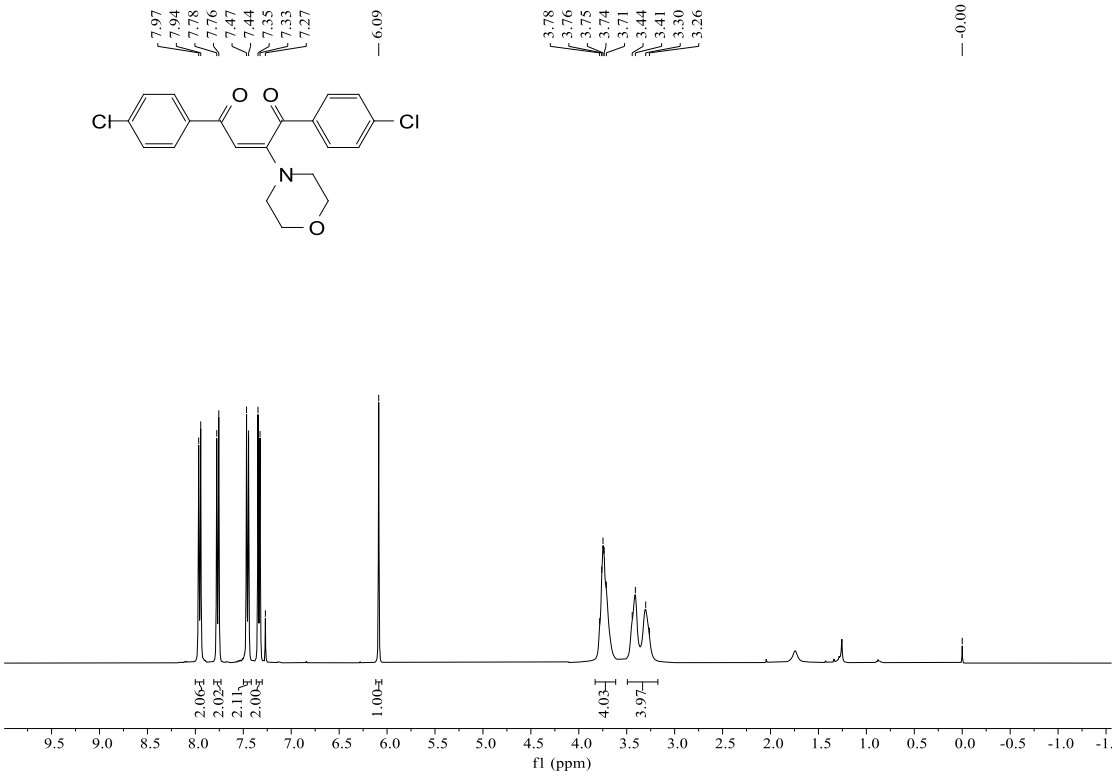
^{1}H NMR spectra of 3y (400 MHz, $CDCl_3$)



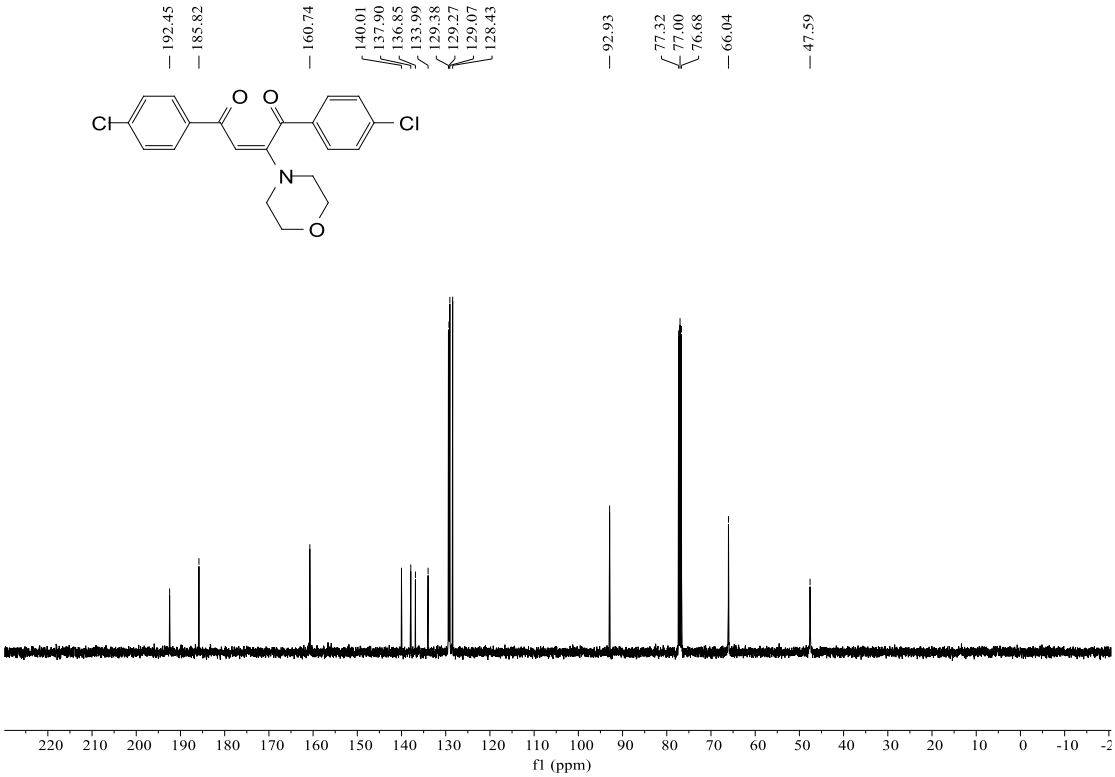
^{13}C NMR spectra of 3y (100 MHz, $CDCl_3$)



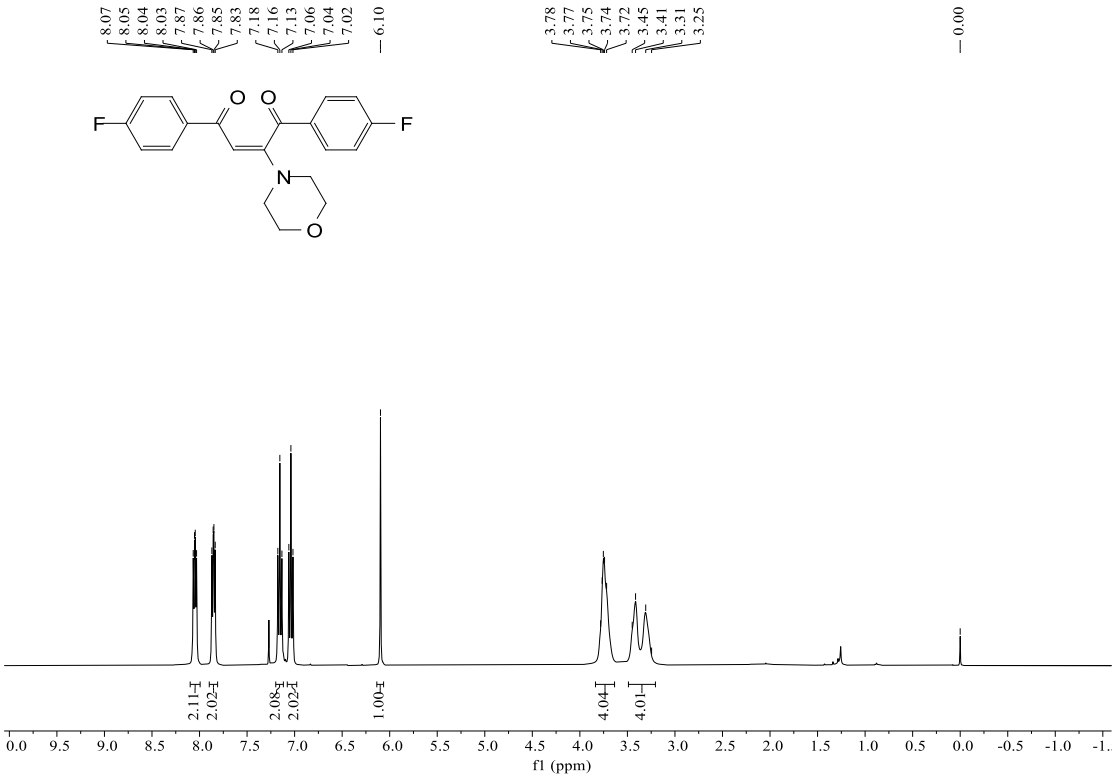
^{1}H NMR spectra of 3z (400 MHz, $CDCl_3$)



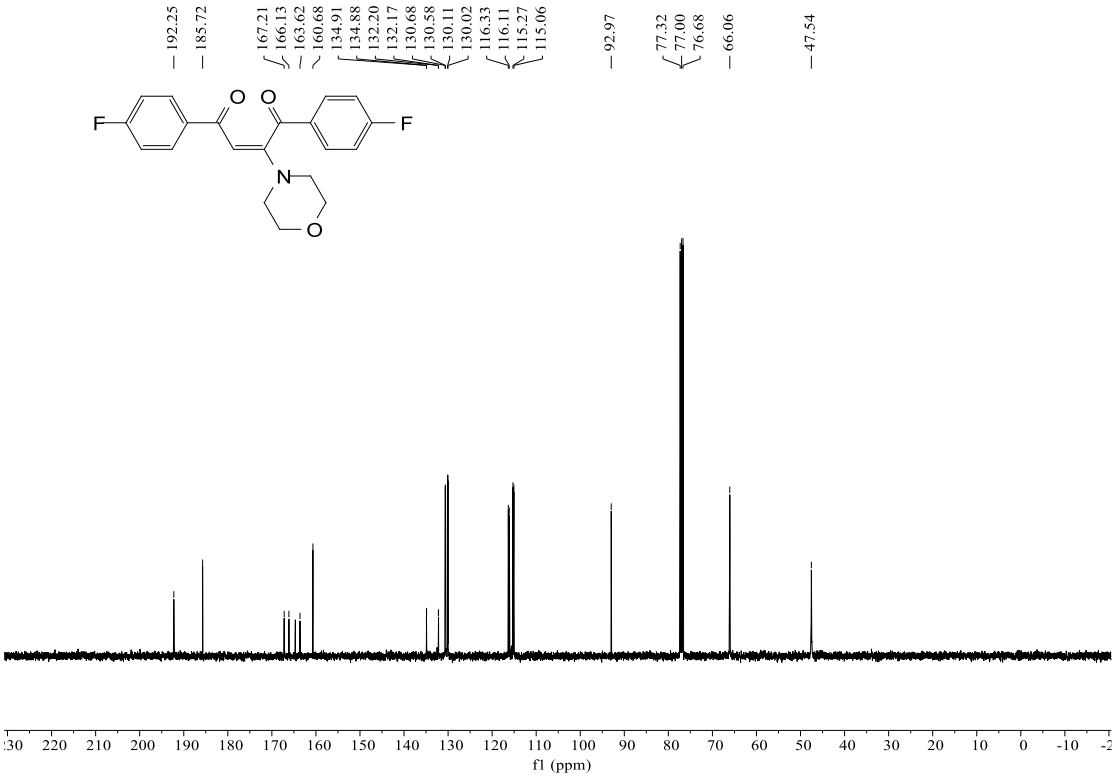
^{13}C NMR spectra of 3z (100 MHz, $CDCl_3$)



^{1}H NMR spectra of 3aa (400 MHz, $CDCl_3$)

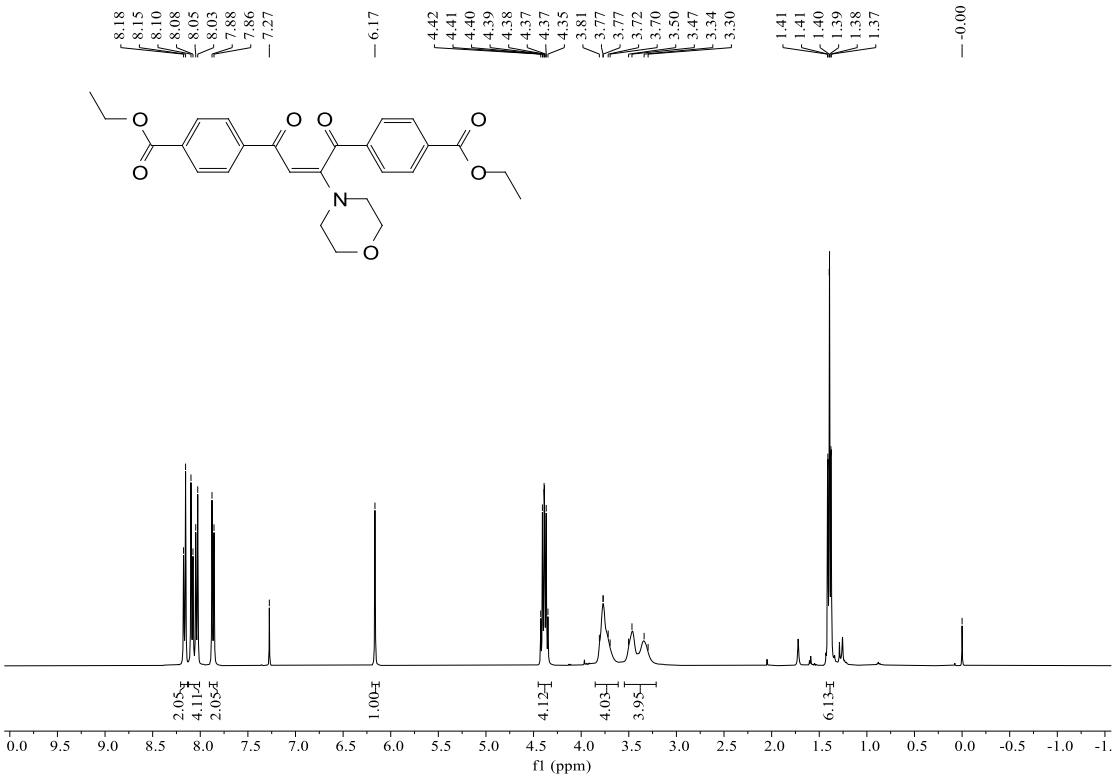


^{13}C NMR spectra of 3aa (100 MHz, $CDCl_3$)

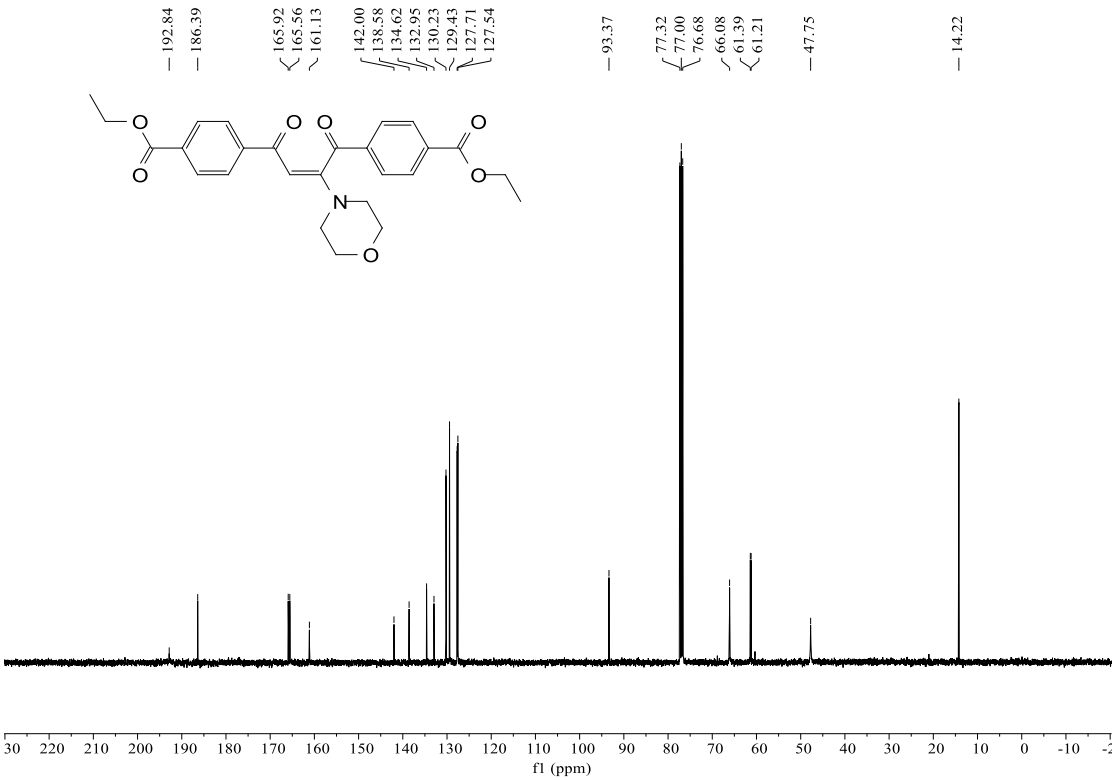


^{19}F NMR spectra of 3aa (376 MHz, $CDCl_3$)

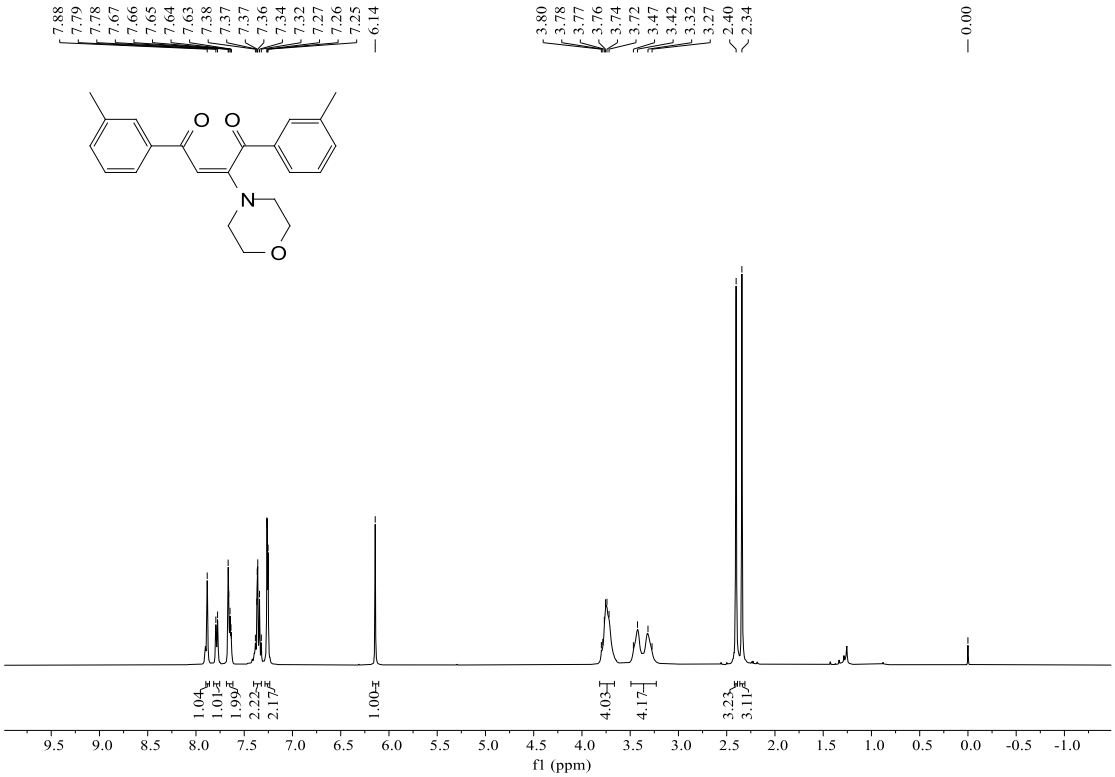
^{1}H NMR spectra of 3ab (400 MHz, $CDCl_3$)



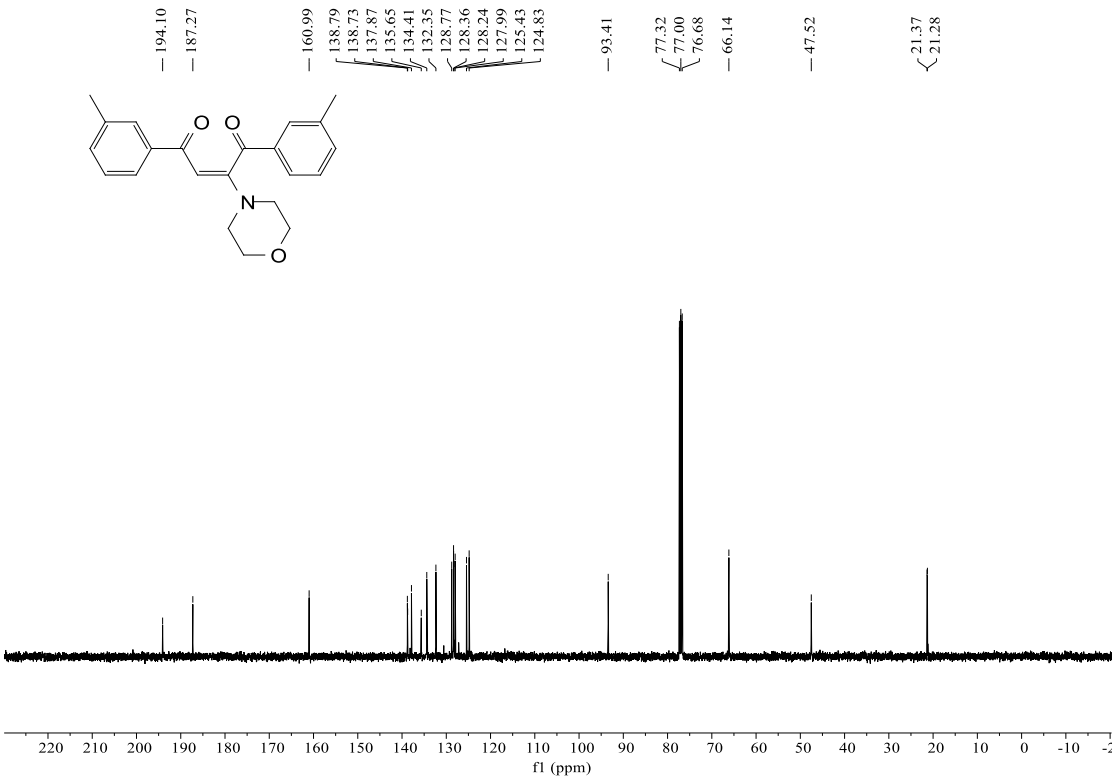
^{13}C NMR spectra of 3ab (100 MHz, $CDCl_3$)



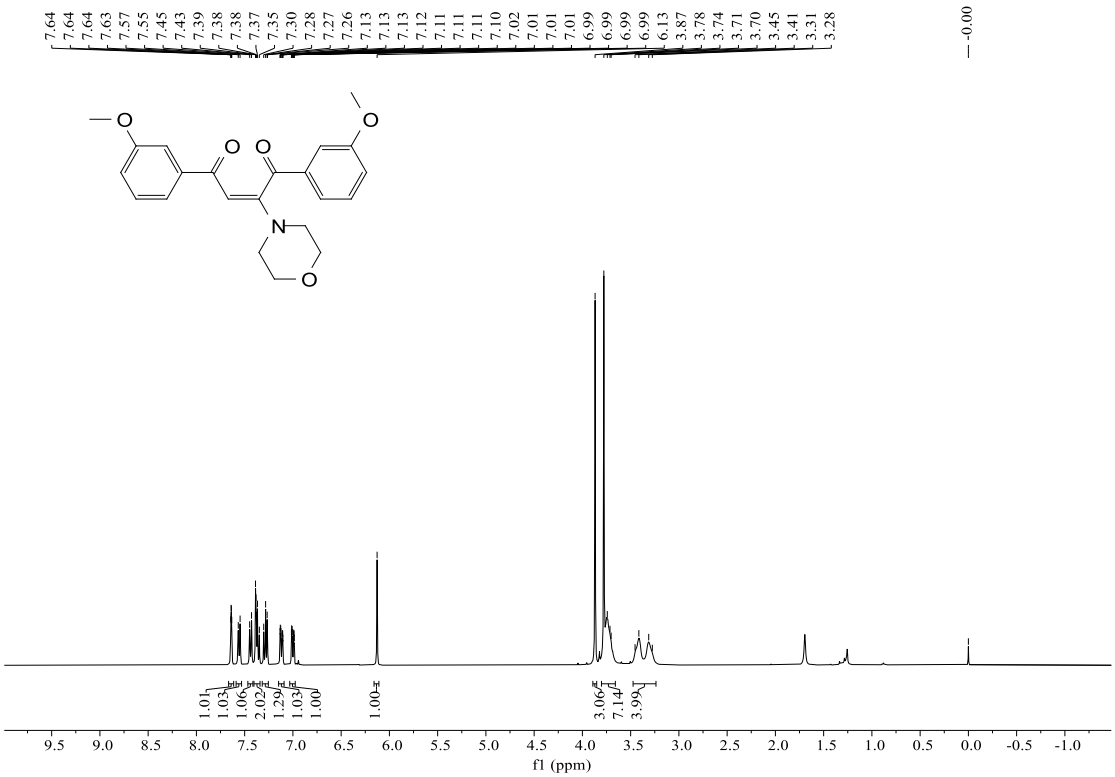
^{1}H NMR spectra of 3ac (400 MHz, $CDCl_3$)



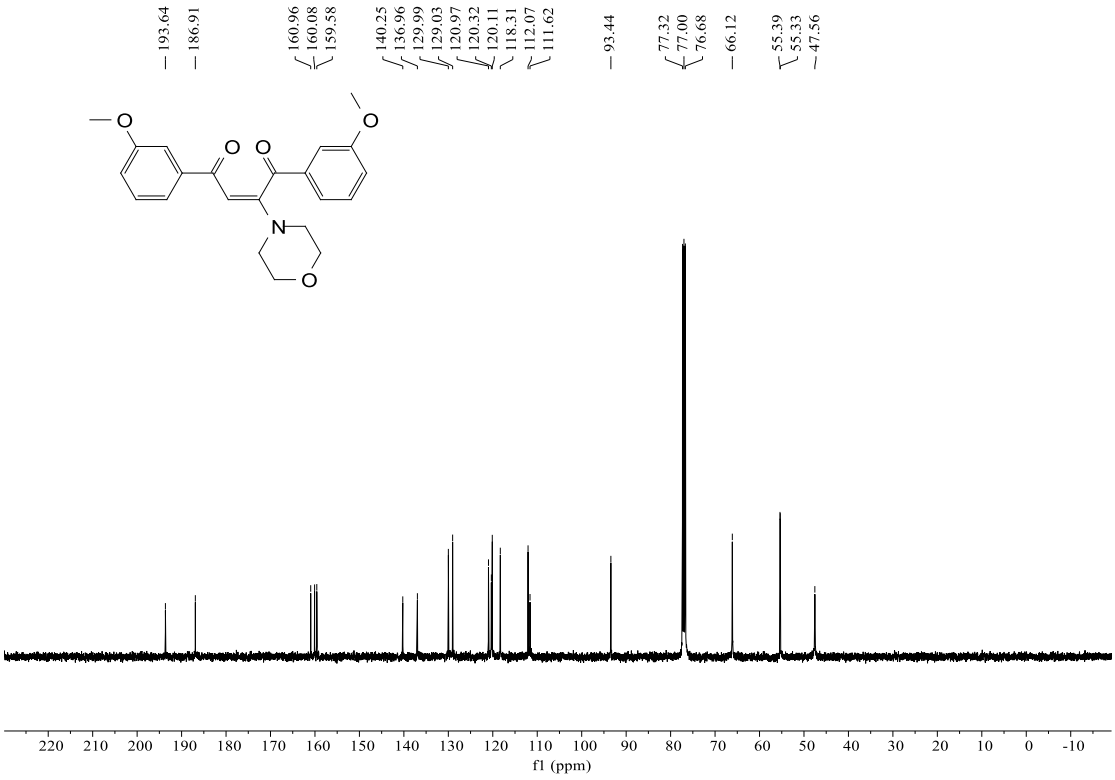
^{13}C NMR spectra of 3ac (100 MHz, $CDCl_3$)



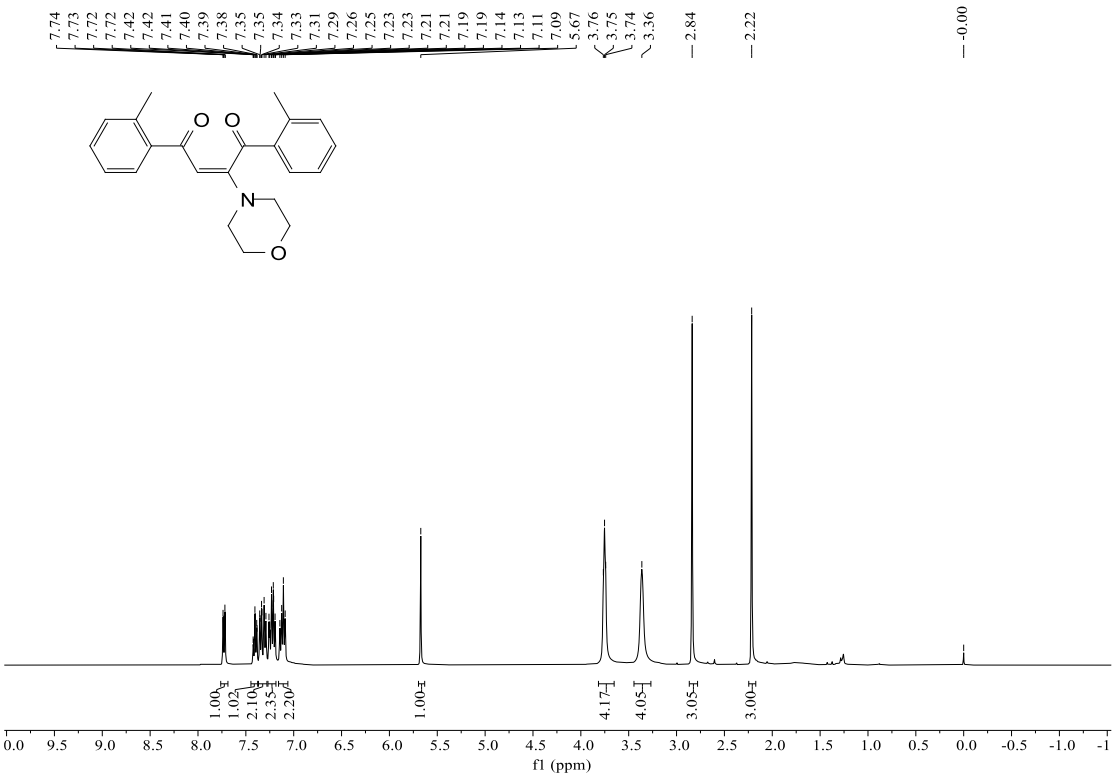
^{1}H NMR spectra of 3ad (400 MHz, $CDCl_3$)



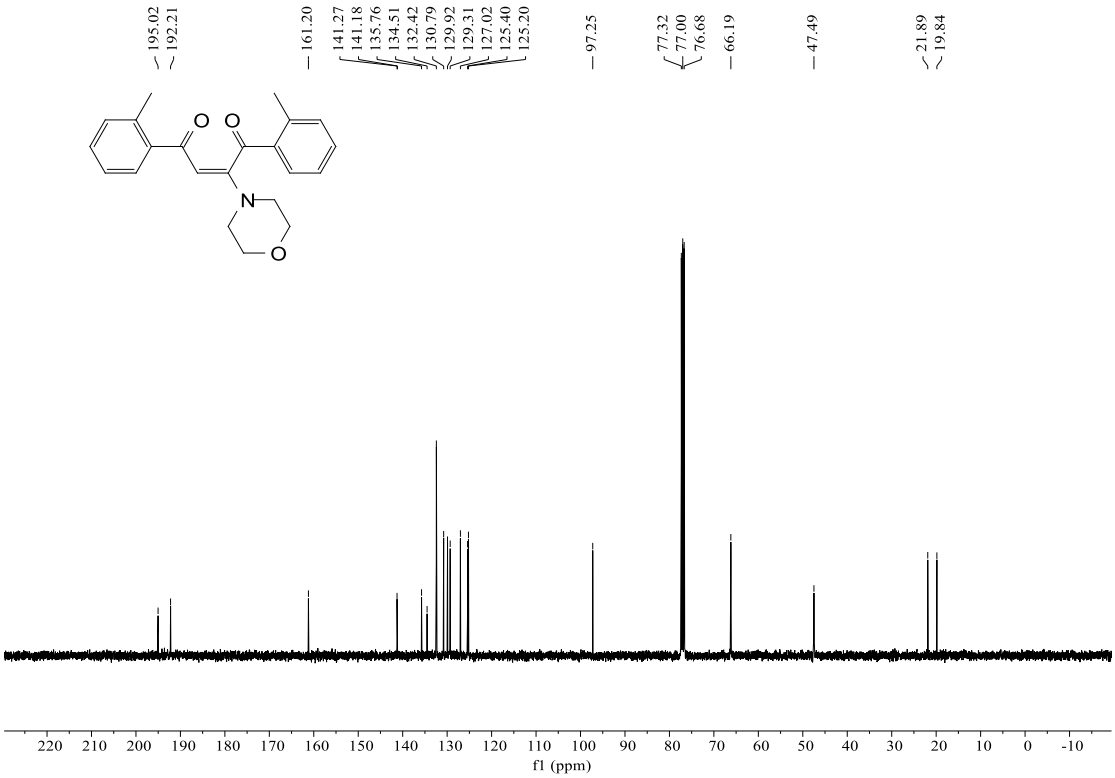
^{13}C NMR spectra of 3ad (100 MHz, $CDCl_3$)



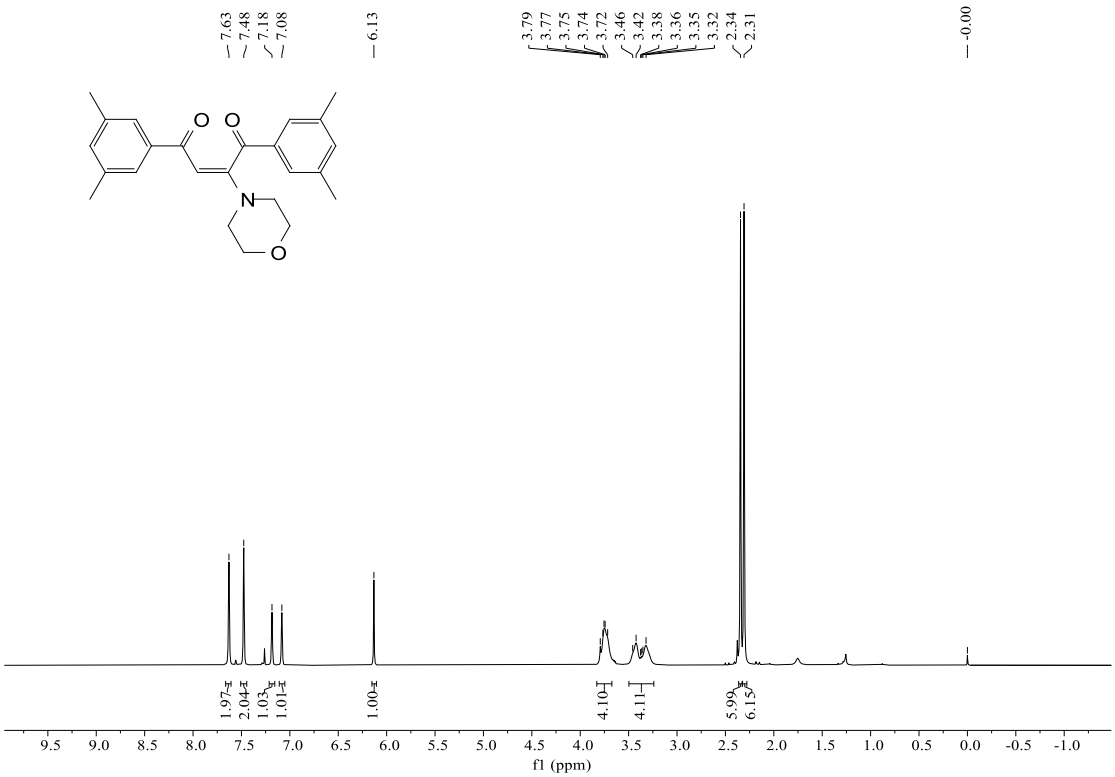
^{1}H NMR spectra of 3ae (400 MHz, $CDCl_3$)



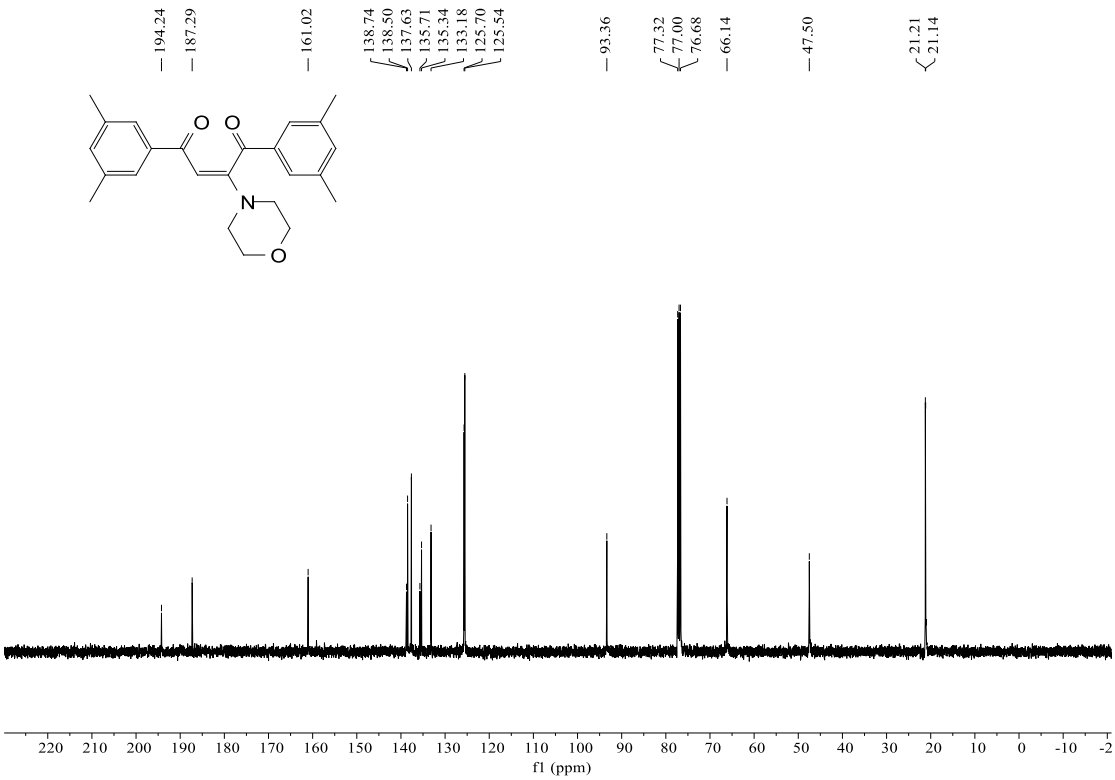
^{13}C NMR spectra of 3ae(100 MHz, $CDCl_3$)



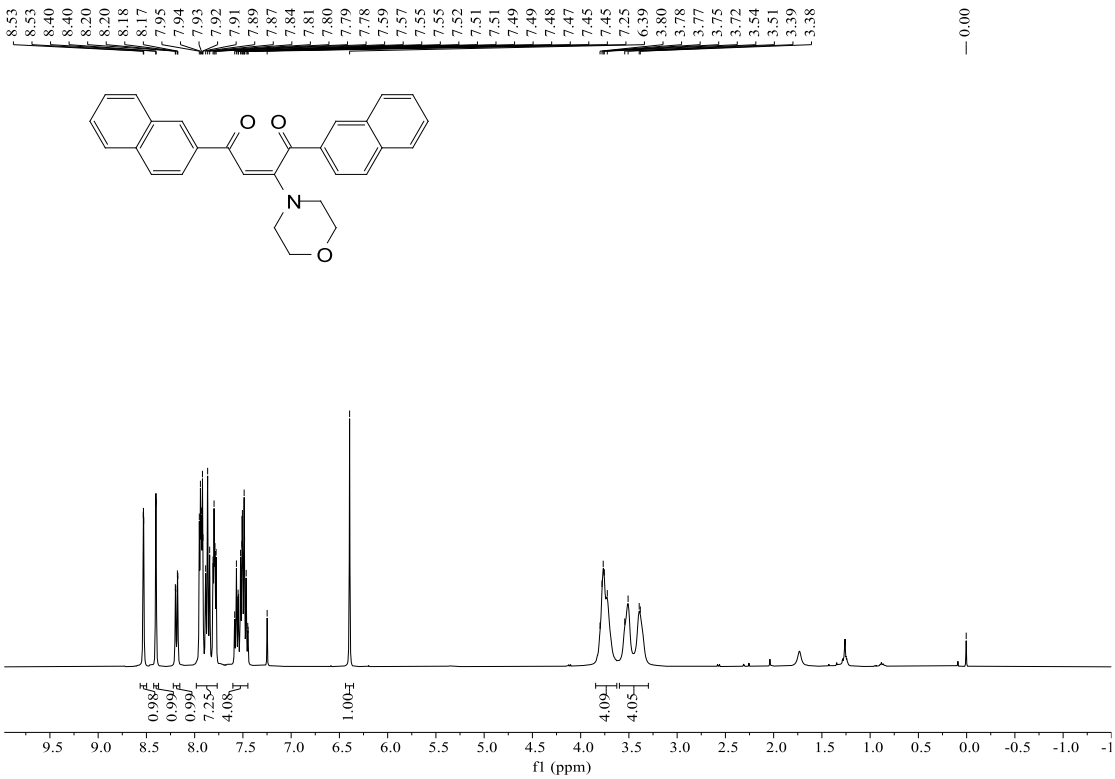
^{1}H NMR spectra of 3af (400 MHz, $CDCl_3$)



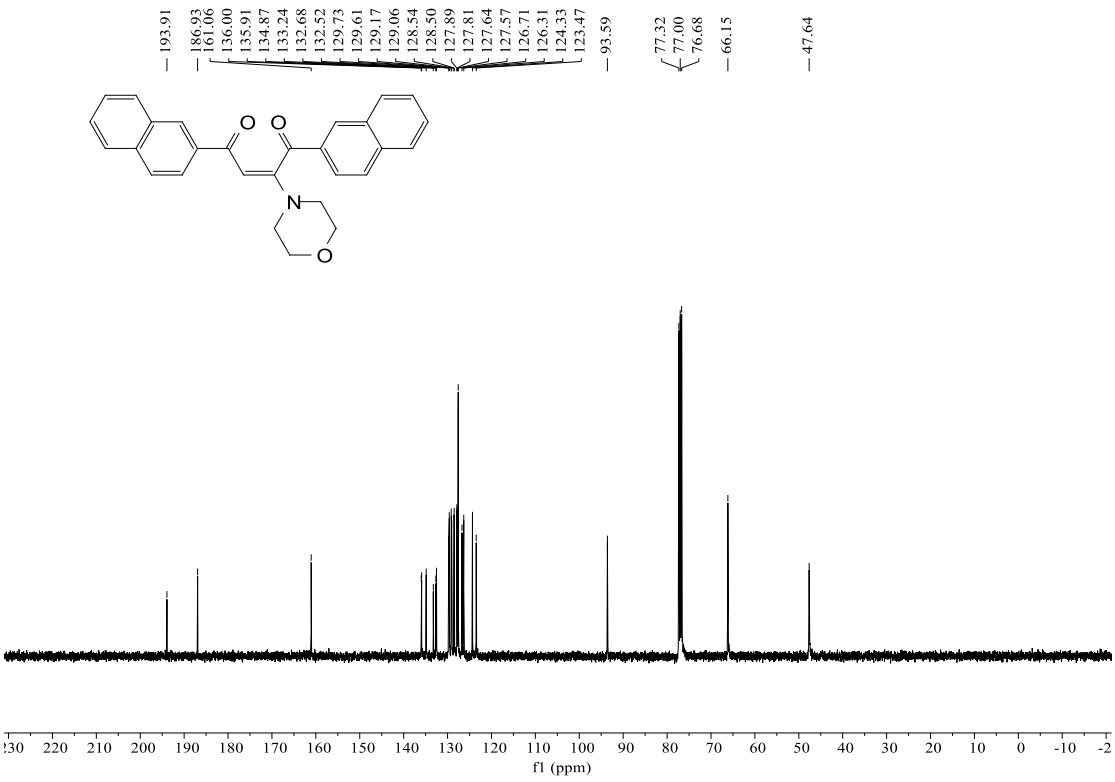
^{13}C NMR spectra of 3af (100 MHz, $CDCl_3$)



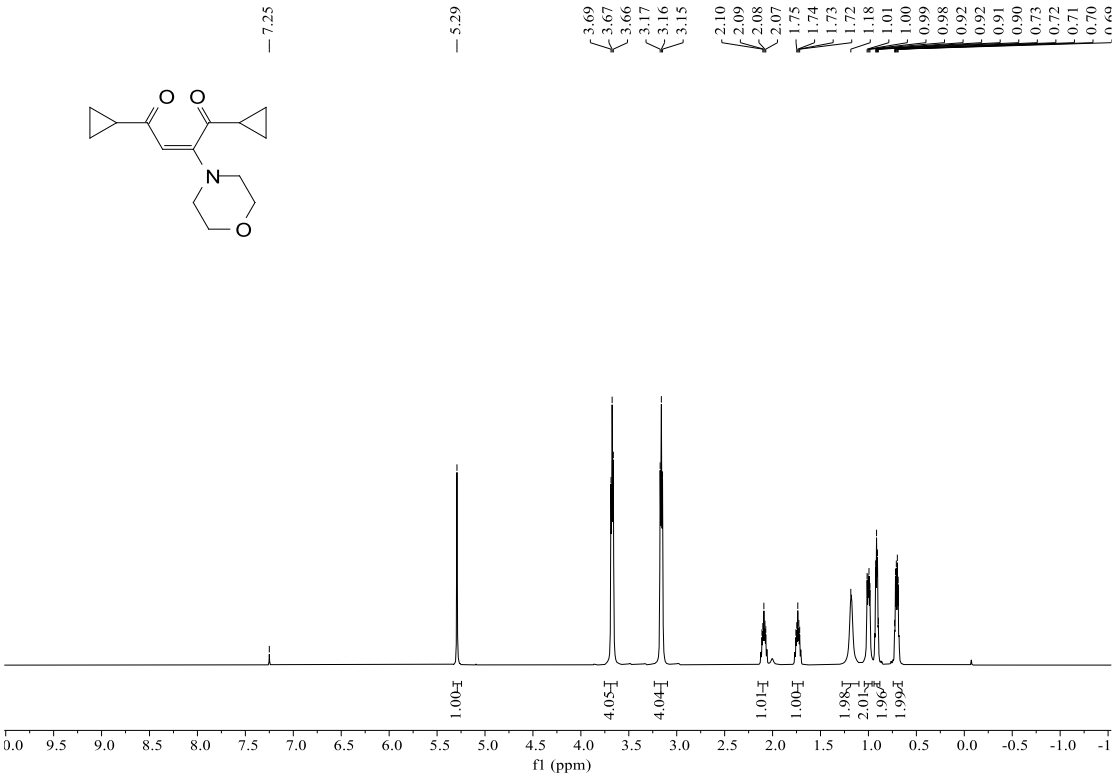
^{1}H NMR spectra of 3ag (400 MHz, $CDCl_3$)



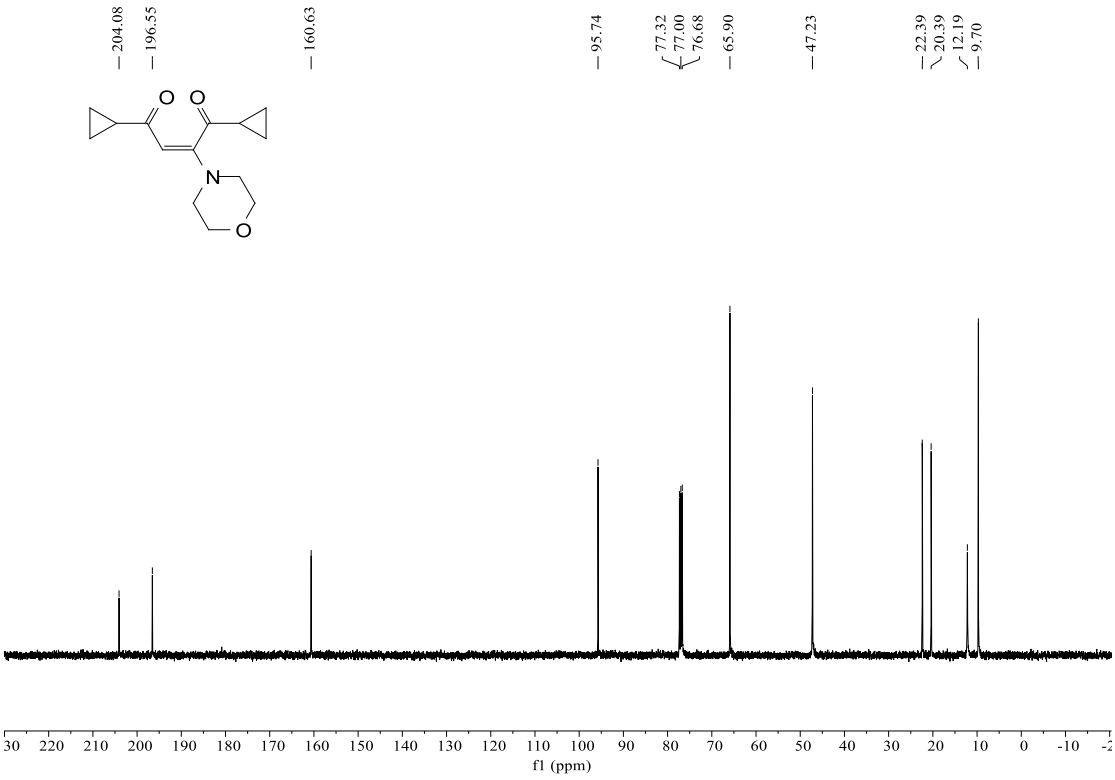
^{13}C NMR spectra of 3ag (100 MHz, $CDCl_3$)



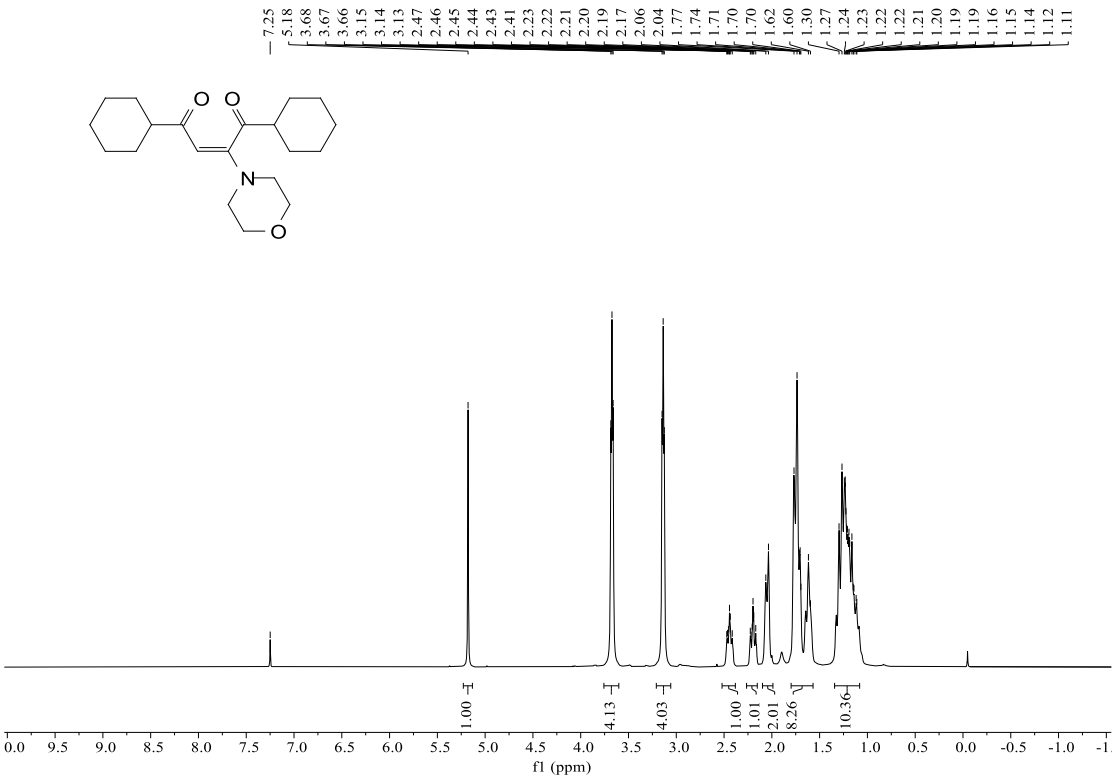
^{1}H NMR spectra of 3ah (400 MHz, $CDCl_3$)



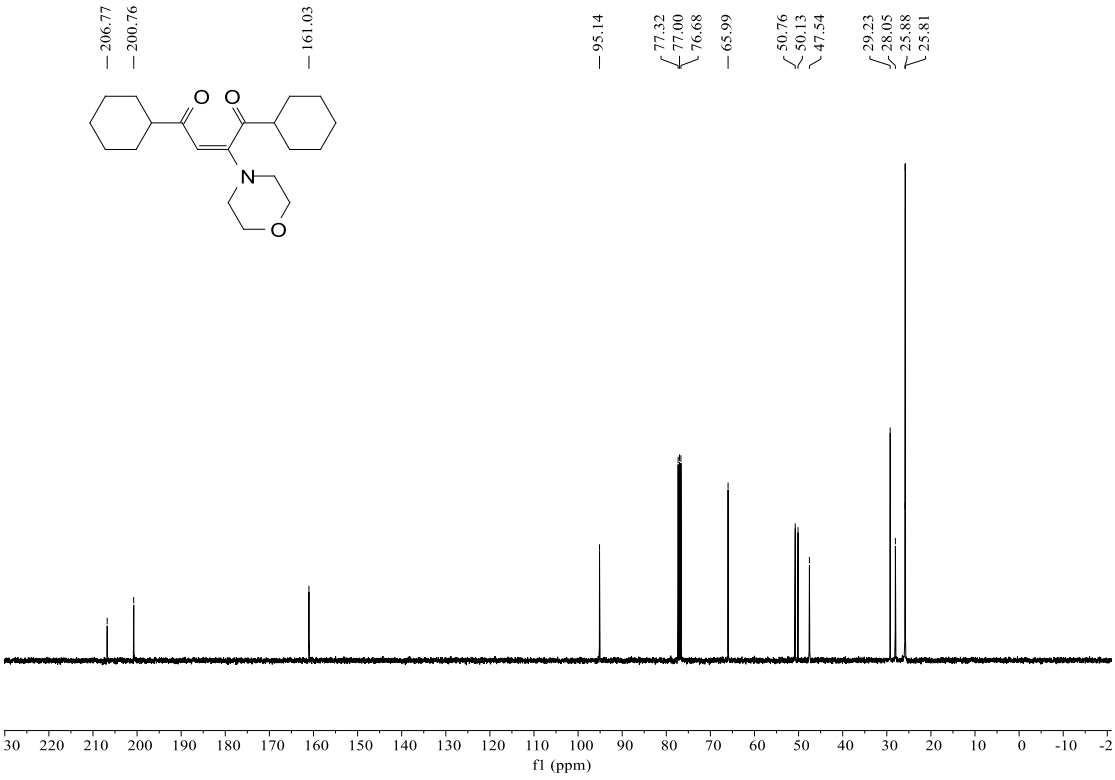
^{13}C NMR spectra of 3ah (100 MHz, $CDCl_3$)



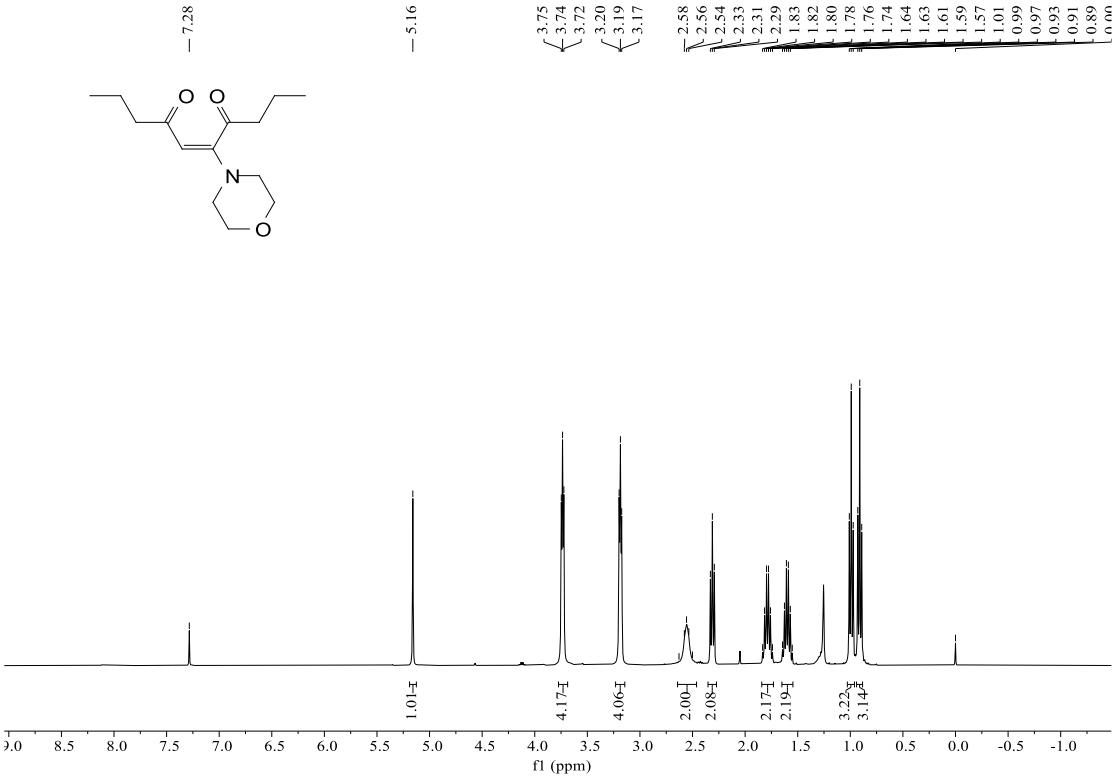
^{1}H NMR spectra of 3ai (400 MHz, $CDCl_3$)



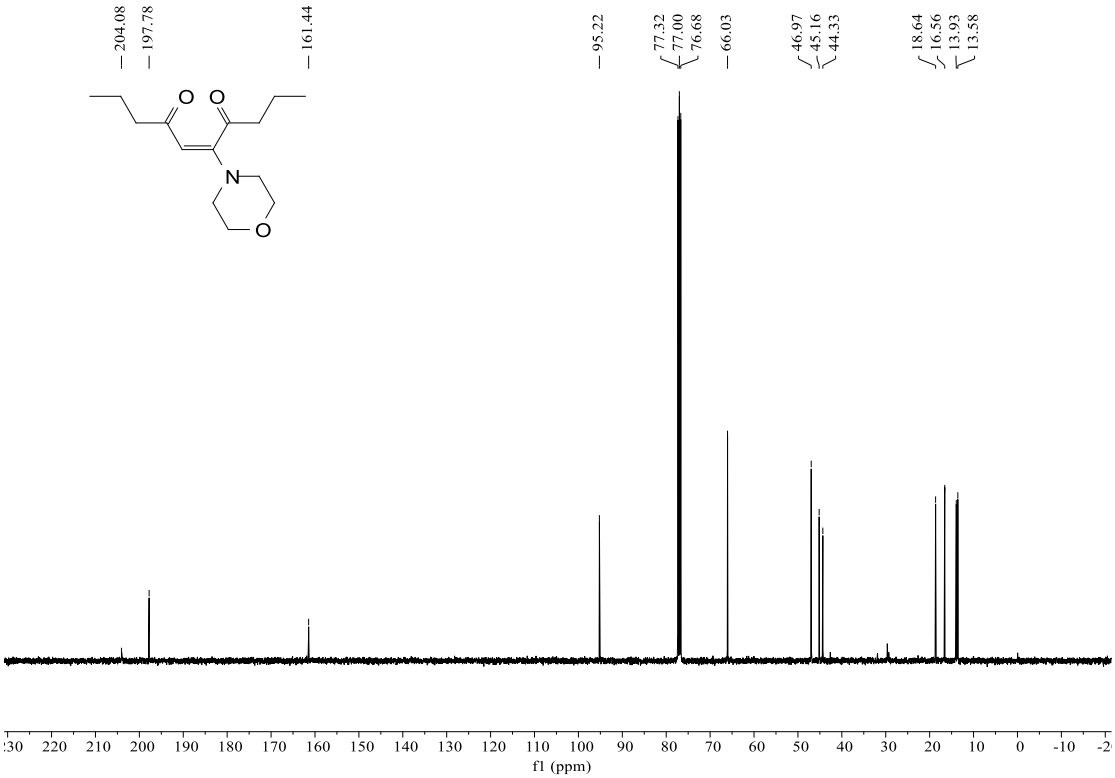
^{13}C NMR spectra of 3ai (100 MHz, $CDCl_3$)



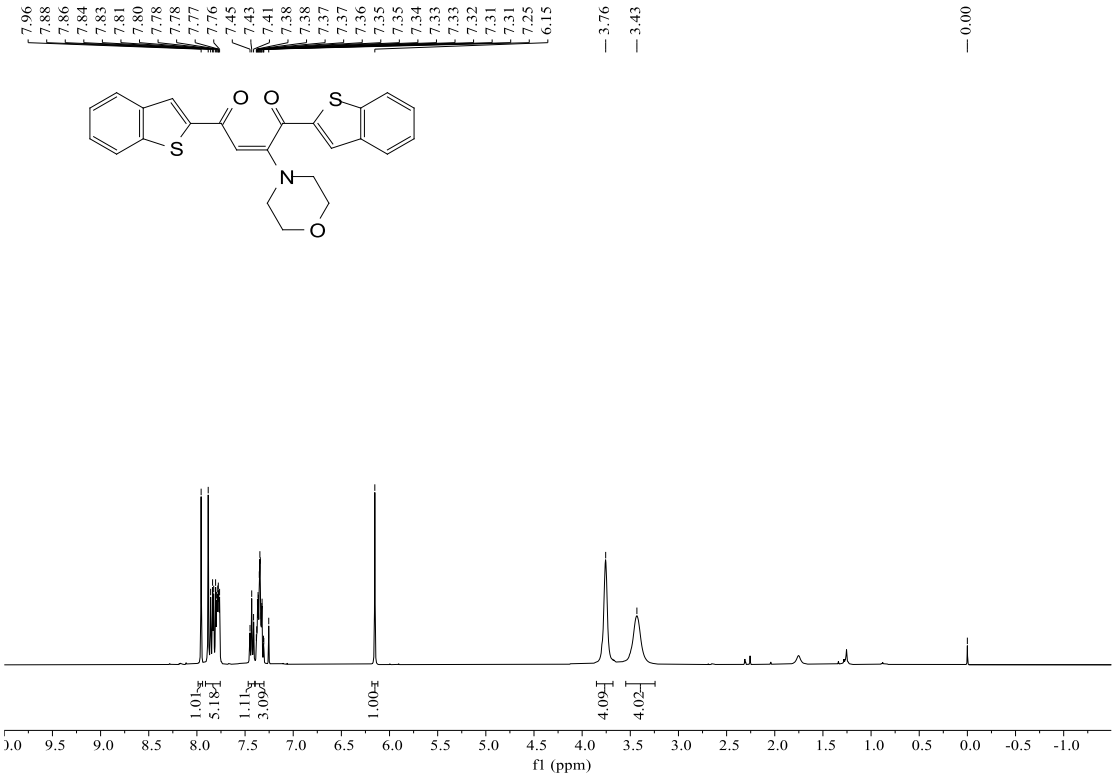
^{1}H NMR spectra of 3aj (400 MHz, $CDCl_3$)



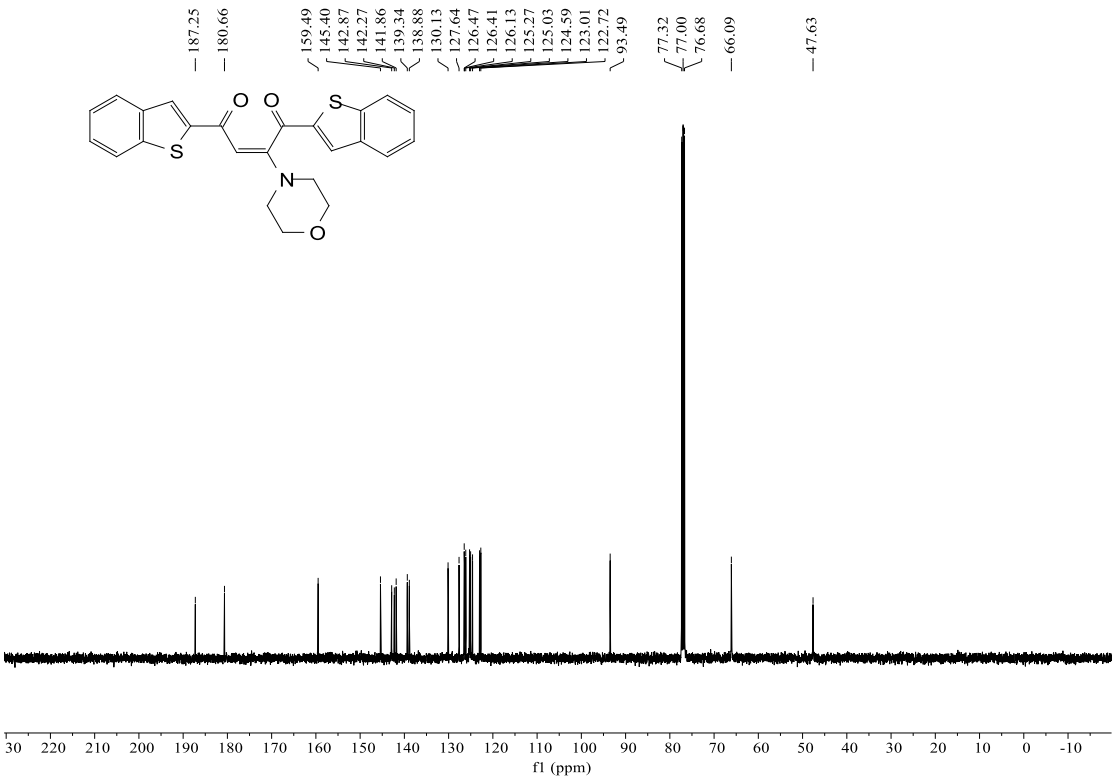
^{13}C NMR spectra of 3aj (100 MHz, $CDCl_3$)



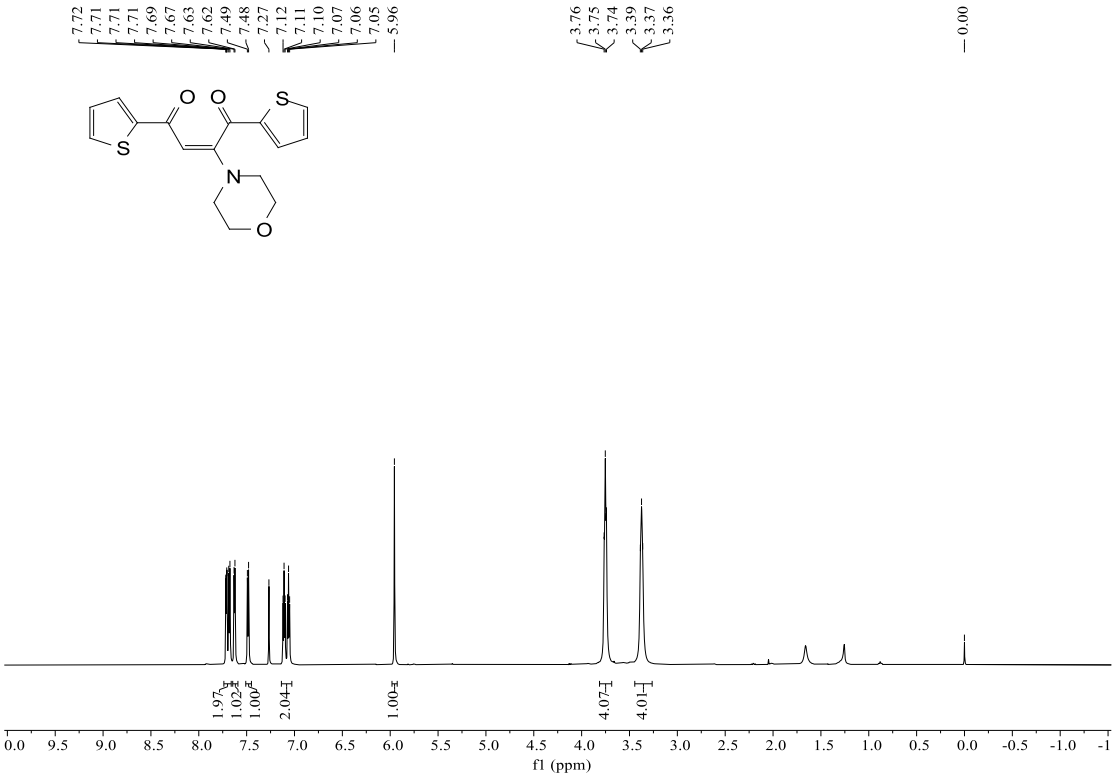
^{1}H NMR spectra of 3ak (400 MHz, $CDCl_3$)



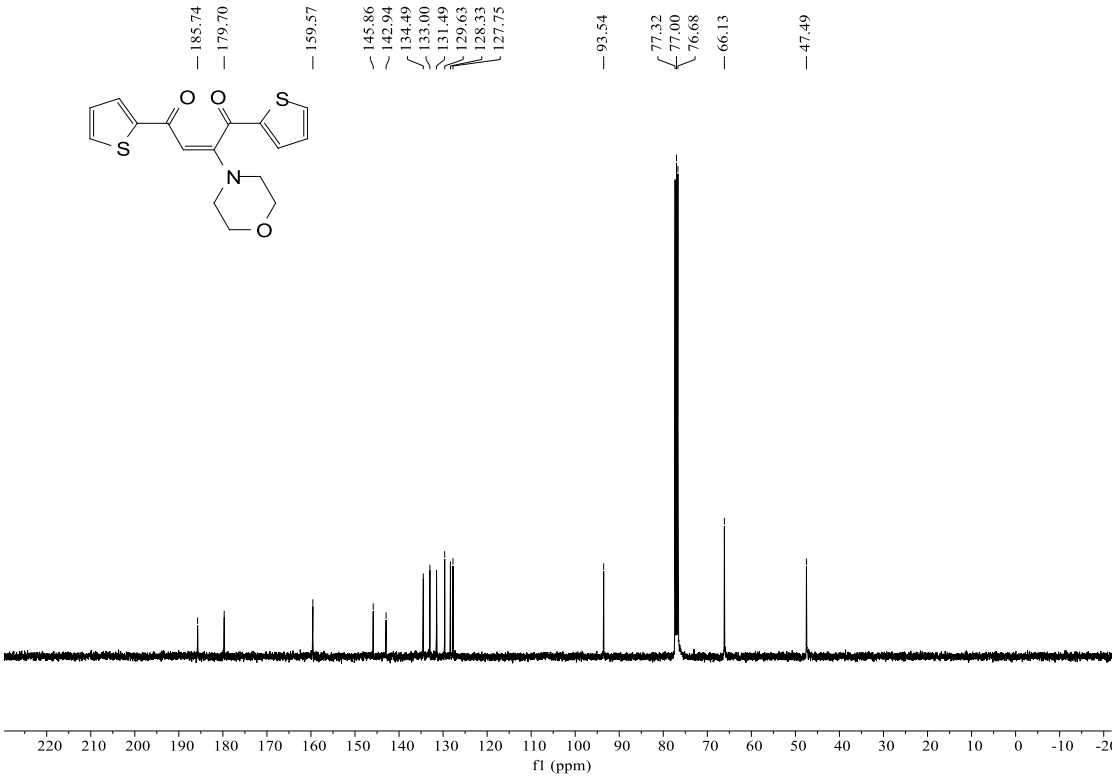
^{13}C NMR spectra of 3ak (100 MHz, $CDCl_3$)



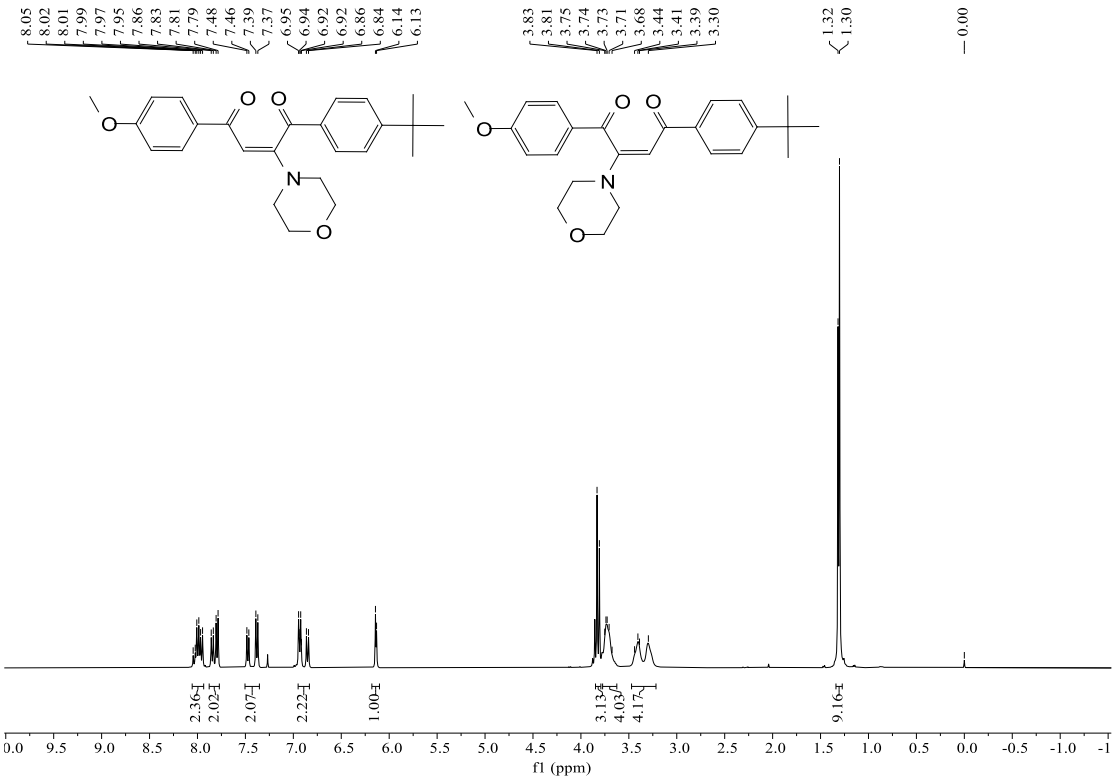
^{1}H NMR spectra of 3al (400 MHz, $CDCl_3$)



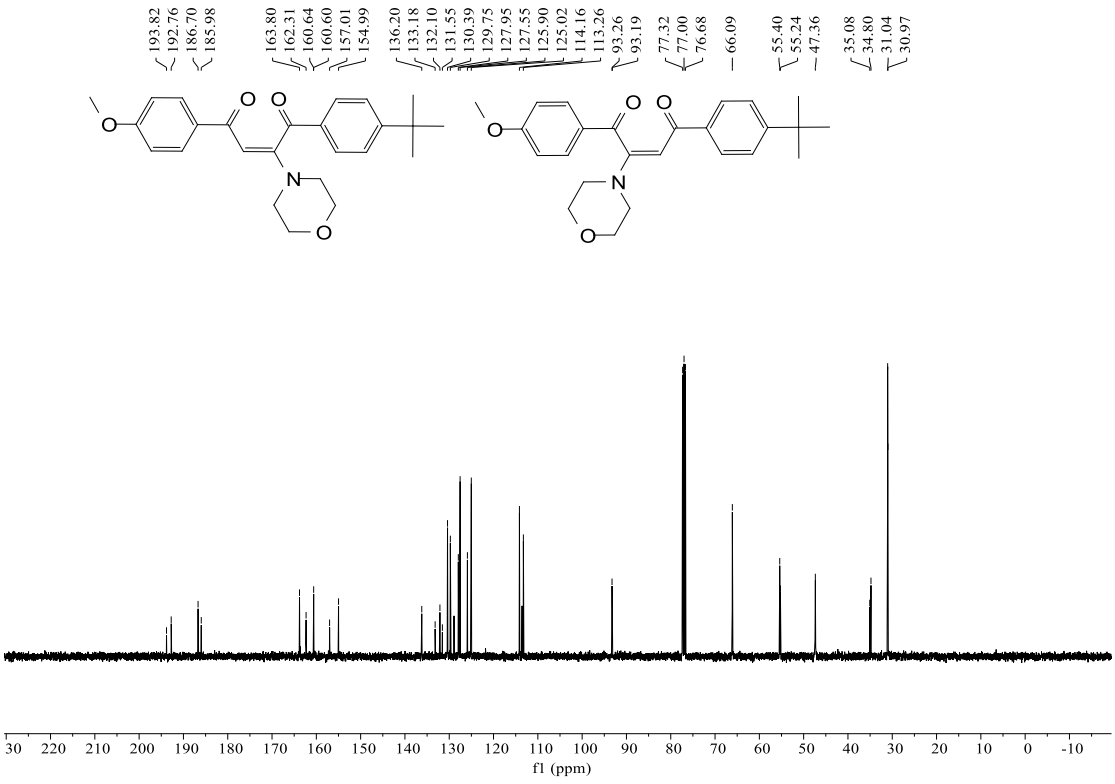
^{13}C NMR spectra of 3al (100 MHz, $CDCl_3$)



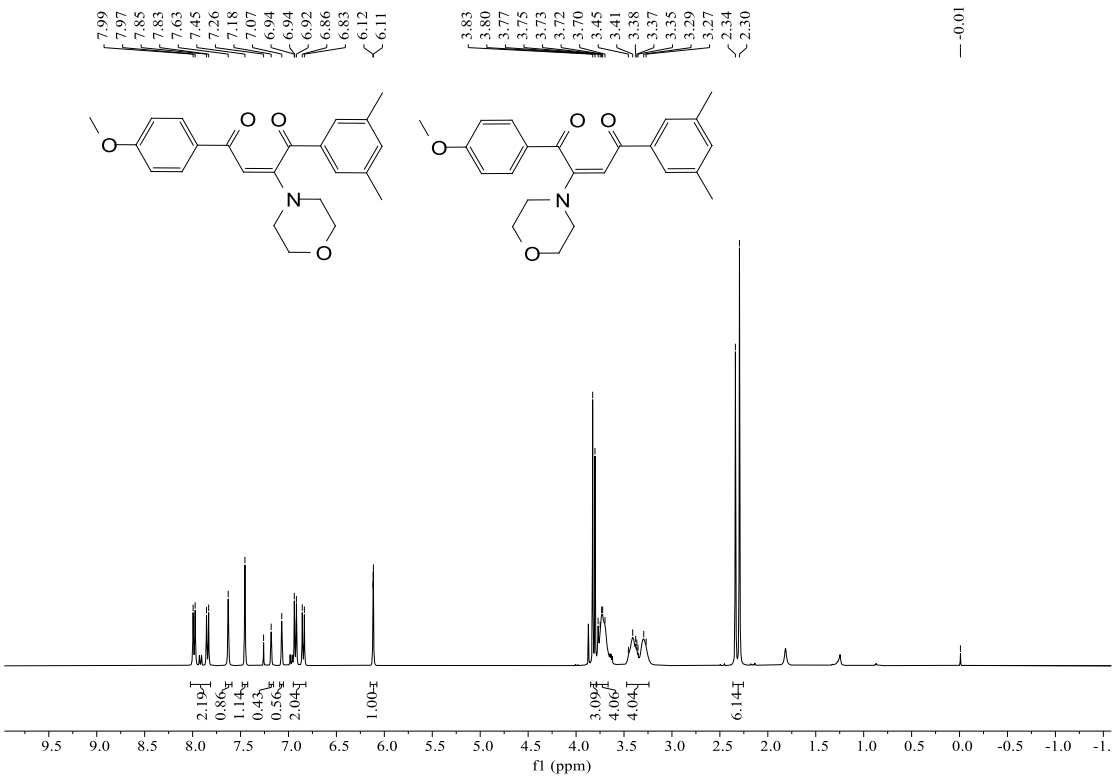
^{1}H NMR spectra of 3am (400 MHz, $CDCl_3$)



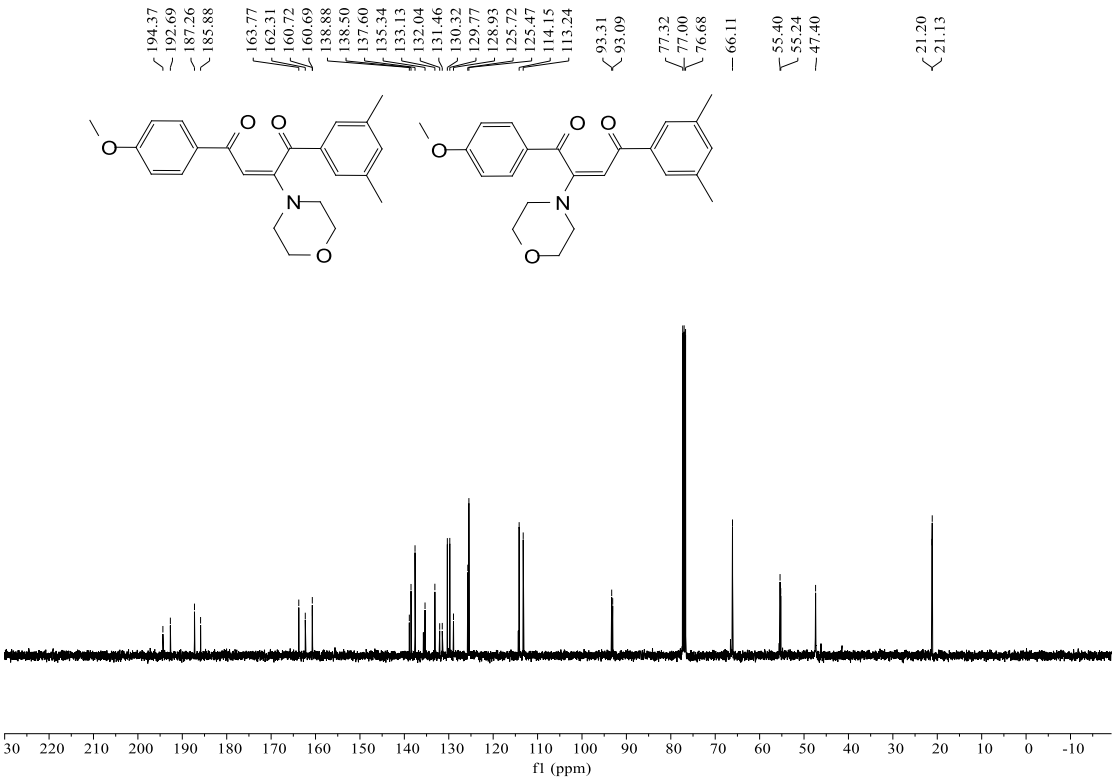
^{13}C NMR spectra of 3am (100 MHz, $CDCl_3$)



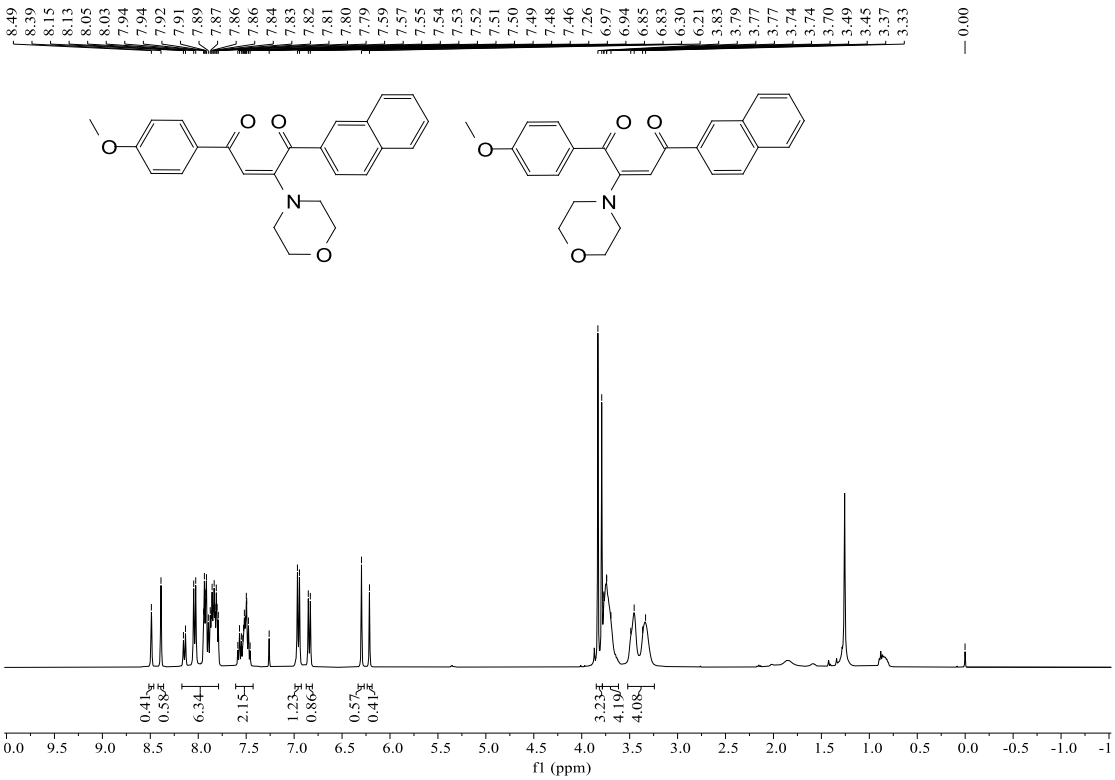
^{1}H NMR spectra of 3an (400 MHz, $CDCl_3$)



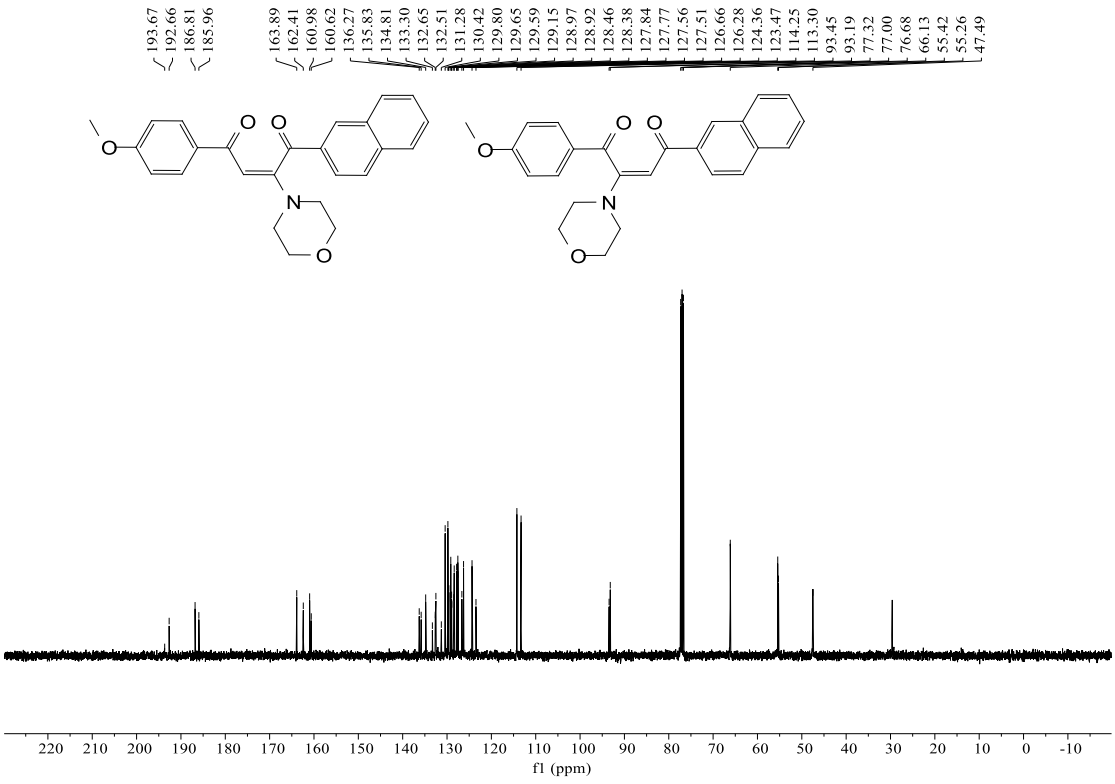
^{13}C NMR spectra of 3an (100 MHz, $CDCl_3$)



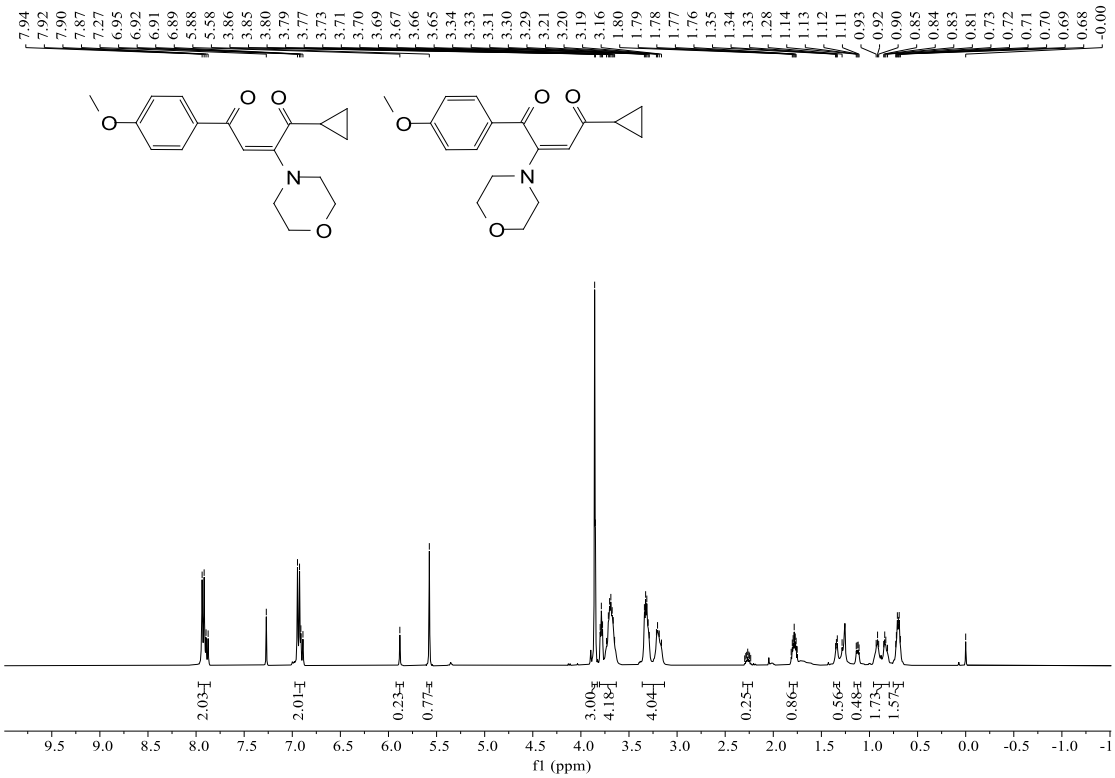
^{1}H NMR spectra of 3ao (400 MHz, $CDCl_3$)



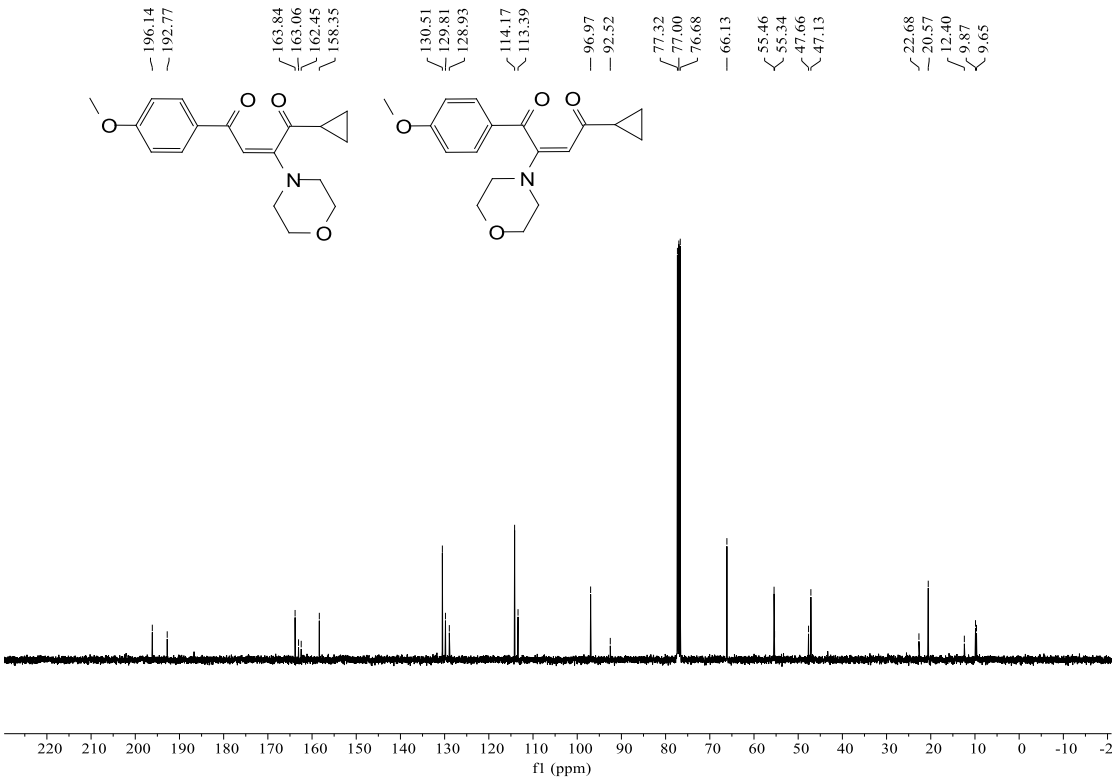
^{13}C NMR spectra of 3ao (100 MHz, $CDCl_3$)



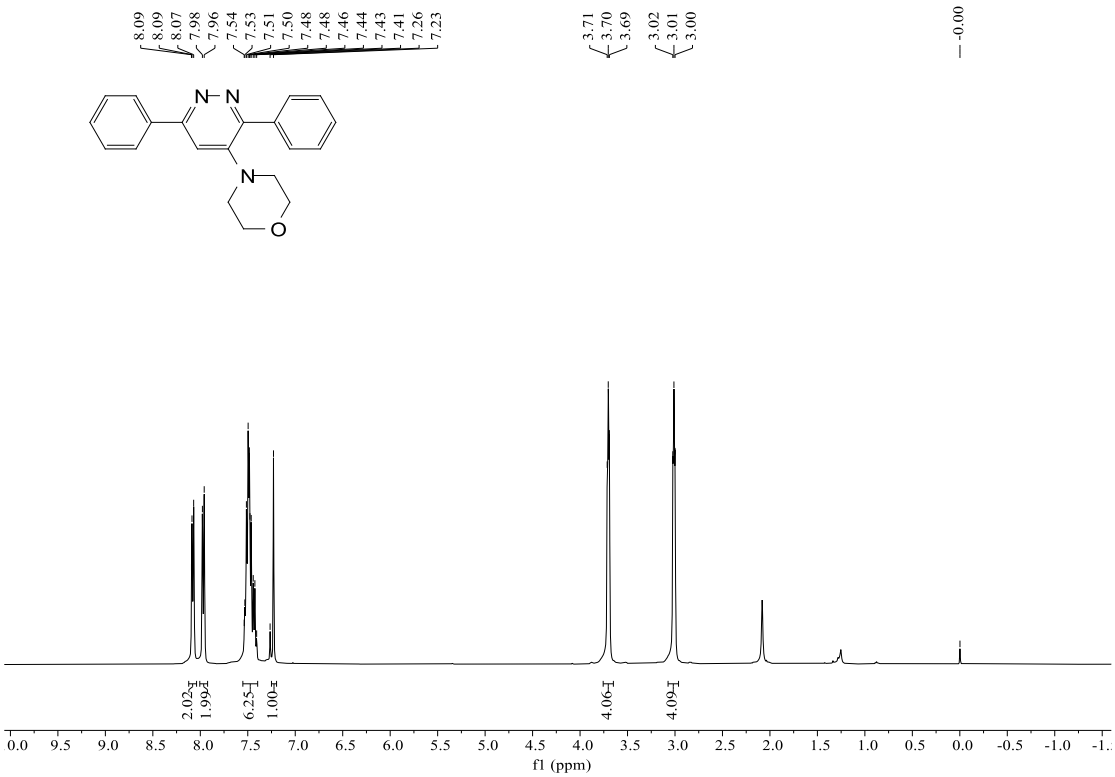
^{1}H NMR spectra of 3ap (400 MHz, $CDCl_3$)



^{13}C NMR spectra of 3ap (100 MHz, $CDCl_3$)



^{1}H NMR spectra of 4a (400 MHz, $CDCl_3$)



^{13}C NMR spectra of 4a (100 MHz, $CDCl_3$)