

Photocatalyst-free formate-mediated aerobic C-O cleavage by EDA complex and SCS strategy for synthesis of diaryl 1,4- diketone

Molai Zhao, Yutong Liu, Min Peng, Yawen Wang, Xiangwei Liu, Hezhong Jiang, Rui Tan*,
Jiahong Li*

Email: jiahongljh@163.com

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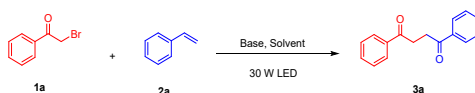
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1. General information

Unless otherwise stated, all chemicals were acquired from commercial suppliers and utilized without additional purification. NMR spectra were collected using a JNM-ECZ600R/S1 spectrometer, with ^1H spectra obtained at 400 MHz and 600 MHz, and ^{13}C spectra at 100 MHz and 150 MHz. Chemical shifts for ^1H NMR (at 400 MHz and 600 MHz) were referenced to CDCl_3 ($\delta = 7.26$). The chemical shifts for ^{13}C NMR (at 100 MHz and 150 MHz) were referenced to CDCl_3 ($\delta = 77.16$). Thin-layer chromatography (TLC) was carried out on glass-backed silica plates. Column chromatography was conducted using silica gel (300-400 mesh) and eluted with a mixture of petroleum ether and ethyl acetate (EA/PE = 10/90). Yields were calculated based on isolated compounds obtained after purification.

2. Optimization of the reaction conditions

Table S1. Optimization of the Reaction Conditions. ^a



Entry	Additive	Solvent	30 W LEDs	Yield (%) ^b
1	HCOOCs	DMSO	410nm	86
2	HCOOCs	DMSO	420nm	75
3	HCOOCs	DMSO	430nm	67
4	HCOOCs	DMSO	440nm	42
5	HCOOCs	DMSO	455nm	31
6	HCOOCs	MeCN	410nm	NR
7	HCOOCs	EtOH	410nm	NR
8	HCOOCs	Tol	410nm	NR
9	HCOOCs	Acetone	410nm	NR
10	HCOOCs	DMF	410nm	11
11	HCOOCs	DMA	410nm	17
12	Et_3N	DMSO	410nm	trace
13	DIPEA	DMSO	410nm	trace
14	DABCO	DMSO	410nm	10
15	Cs_2CO_3	DMSO	410nm	NR
16	NaOH	DMSO	410nm	NR
17	KOH	DMSO	410nm	NR
18	K_3PO_4	DMSO	410nm	NR
19	HCOOK	DMSO	410nm	trace

20	HCOONa	DMSO	410nm	trace
21	HCOONH ₄	DMSO	410nm	62
22	(HCOO) ₂ Ca	DMSO	410nm	43
23 ^c	HCOOCs	DMSO	410nm	trace
24 ^d	HCOOCs	DMSO	410nm	trace

^aReaction conditions: **1a** (1.0 mmol, 1.0 equiv), **2a** (1.0 mmol, 1.0 equiv), base (4.0 mmol, 4.0 equiv), solvent (4 mL), under air and room temperature conditions, illuminated by a 30 W LED lamp for 15 hours in air. NR: No reaction. ^bIsolated yield. ^cUnder dark condition. ^dUnder N₂.

Table S2 Effects of light^{a,b}

Entry	Conditions	Yield (%) ^b
1	410 nm	86
2	420 nm	75
3	430 nm	67
4	440 nm	42
5	455 nm	31

^aReaction conditions: **1a** (1.0 mmol, 1.0 equiv), **2a** (1.0 mmol, 1.0 equiv), base (4.0 mmol, 4.0 equiv), solvent (4 mL), under air and room temperature conditions, illuminated by a 30 W LED lamp for 15 hours. ^bIsolated yield.

Table S3 Effects of additive^{a,b}

Entry	Additive	Yield (%) ^b
1	HCOOCs	86
2	Et ₃ N	NR
3	DIPEA	trace
4	DABCO	10
5	Cs ₂ CO ₃	NR
6	NaOH	NR
7	KOH	NR
8	K ₃ PO ₄	NR

^aReaction conditions: **1a** (1.0 mmol, 1.0 equiv), **2a** (1.0 mmol, 1.0 equiv), base (4.0 mmol, 4.0 equiv), solvent (4 mL), under air and room temperature conditions, illuminated by a 30 W 410 nm LED lamp for 15 hours. ^bIsolated yield.

Table S4 Effects of solvent^{a,b}

Entry	conditions	Yield (%) ^b
1	DMSO	86
2	MeCN	NR ^c

3	EtOH	NR
4	Tol	NR
5	Acetone	NR
	M	11
7	DMA	17

^aReaction conditions: **1a** (1.0 mmol, 1.0 equiv), **2a** (1.0 mmol, 1.0 equiv), base (4.0 mmol, 4.0 equiv), solvent (4 mL), under air and room temperature conditions, illuminated by a 30 W 410 nm LED lamp for 15 hours. ^bIsolated yield.

Table S5 Effect of cesium formate dosage^{a,b}



Entry	Equiv of HCOOCs	Yield of 3a (%)	Yield of 4 (%)
1	1	0	96
2	2	55	27
3	4	86	0
4	6	87	0

^aReaction conditions: **1a** (1.0 mmol, 1.0 equiv), **2a** (1.0 mmol, 1.0 equiv), HCOOCs, 4 mL DMSO, under air and room temperature conditions, illuminated with a 30 W 410 nm LED lamp for 15 hours. ^bIsolated yield.

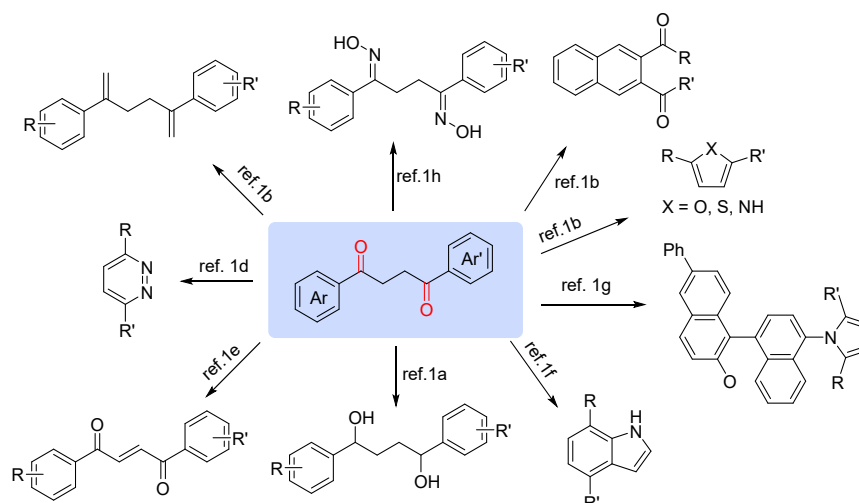
Table S6 Effect of formate types^{a,b}



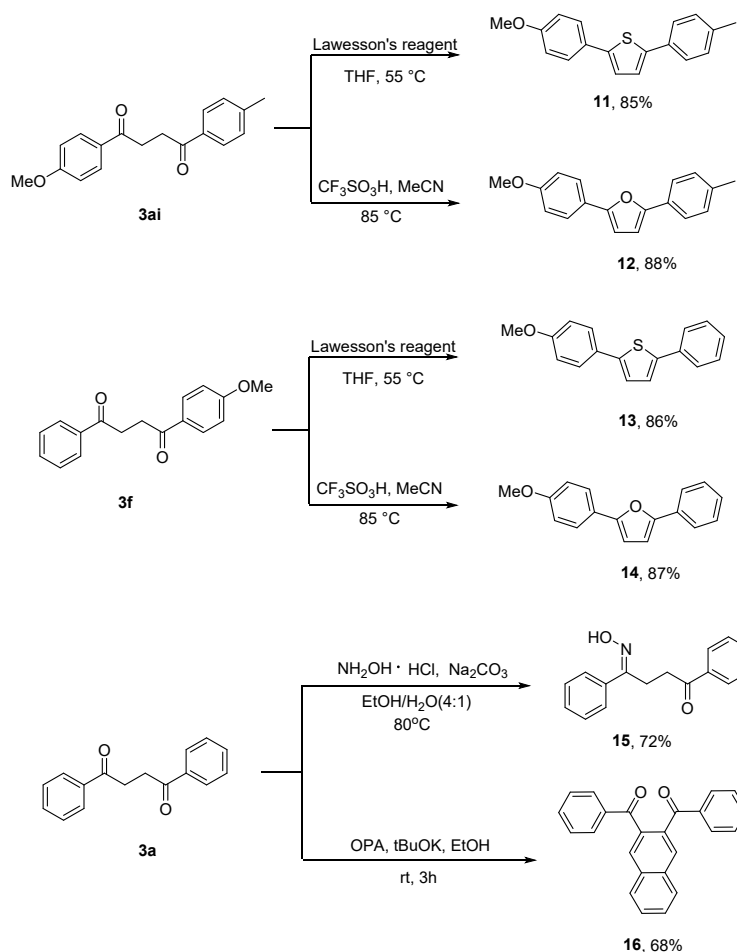
Entry	Formate	Yield of 3a (%)	Yield of 4 (%)
1	HCOOCs	86	trace
2	HCOOK	trace	96
3	HCOONa	trace	98
4	HCOONH ₄	62	trace
5	(HCOO) ₂ Ca	43	21

^aReaction conditions: **1a** (1.0 mmol, 1.0 equiv), **2a** (1.0 mmol, 1.0 equiv), Formate (1.0 mmol, 4.0 equiv), 4 mL DMSO, illuminated with 30W 410nm LED lamp for 15 hours under air and room temperature conditions. ^bIsolated yield.

3. Application of diaryl 1,4-Diketone¹



Scheme S1. Application of diaryl 1,4-diketone reported by literatures



Scheme 5. The conversion of diaryl 1,4-diketone compounds by us

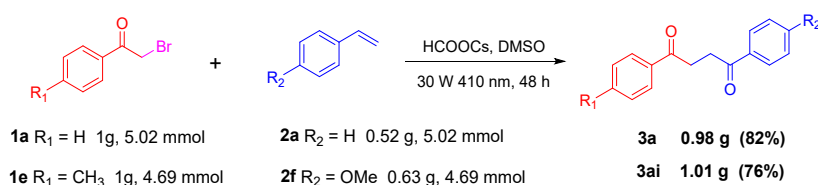
We explored the application of this method through corresponding studies, as demonstrated below in **Scheme 5**. The advantage of our method is that it can synthesize not only the symmetric 1,4-diketones, but also the asymmetric ones. So, We selected symmetric and asymmetric products

for conversion studies. First, we performed the corresponding transformation using an asymmetric substrate **3ai** under the action of Lawesson's reagent, the cyclization product **11** was obtained in high yield. Another cyclization product **12** was obtained in the presence of trifluoromethanesulfonic acid. Similarly, using **3f** as the starting material, products **13** and **14** were obtained respectively. Subsequently, we selected the symmetric substrate **3a** and added hydroxylamine hydrochloride to achieve the oxime etherification of one carbonyl group and get the compound **15**. Then, under the simultaneous action of *o*-phthalaldehyde and *t*-BuOK, we obtained compound **16**. The corresponding information has been included in the revised manuscript and Supporting Information.

4. General procedure for C–C coupling

In a reaction bottle with a magnetic stirrer, α -Bromophenone (0.25 mmol 50 mg), styrene (29.0 μ L) and cesium formate (1 mmol) was added into. Then add solvent DMSO (4 mL) and cap the bottle. Move the reaction bottle on a magnetic stirrer and irradiate it with 410 nm 30W LED at room temperature under an air atmosphere for 6 hours. Add pure water (5 mL) and EA (20 mL) for extraction, dry the combined organic phase with Na₂SO₄, filter and concentrate. The product 1,2-benzoyl ethane (**3a**) was isolated by flash chromatography on 300-400 mesh silica gel with PE/EA.

5. Scale up experiment



Scheme S3. Gram-scale synthesis

In a reaction bottle with a magnetic stirrer, α -Bromophenone (5 mmol 1 g), styrene (0.58 mL) and cesium formate (3.56 g) was added into. Then add solvent DMSO (20 mL) and cap the bottle. Move the reaction bottle on a magnetic stirrer and irradiate it with 410 nm 30W LED at room temperature under an air atmosphere for 48 hours. Add pure water (10 mL) and EA (60 mL) for extraction, dry the combined organic phase with Na₂SO₄, filter and concentrate. The product 1,2-benzoyl ethane (**3a**) was isolated by flash chromatography on 300-400 mesh silica gel with PE/EA (12.5/1(v/v)). The same applies to the synthesis of 1-(4-methoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione.

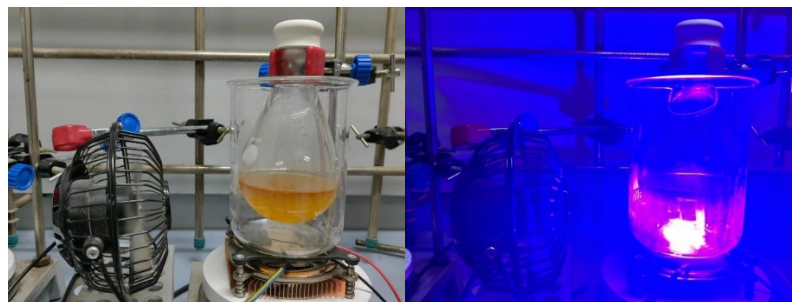


Figure S1 Reaction apparatus

6. Mechanistic experiments

6.1 NMR experiment

NMR experiment : Solutions containing equal molar concentrations of the **4** (0.02 M in DMSO- d_6) and the HCOOCs(0.08 M in DMSO- d_6) were prepared and mixed to cover acceptor/donor ratio from 0.2:0.8, 0.5:0.5 to 0.8:0.2.

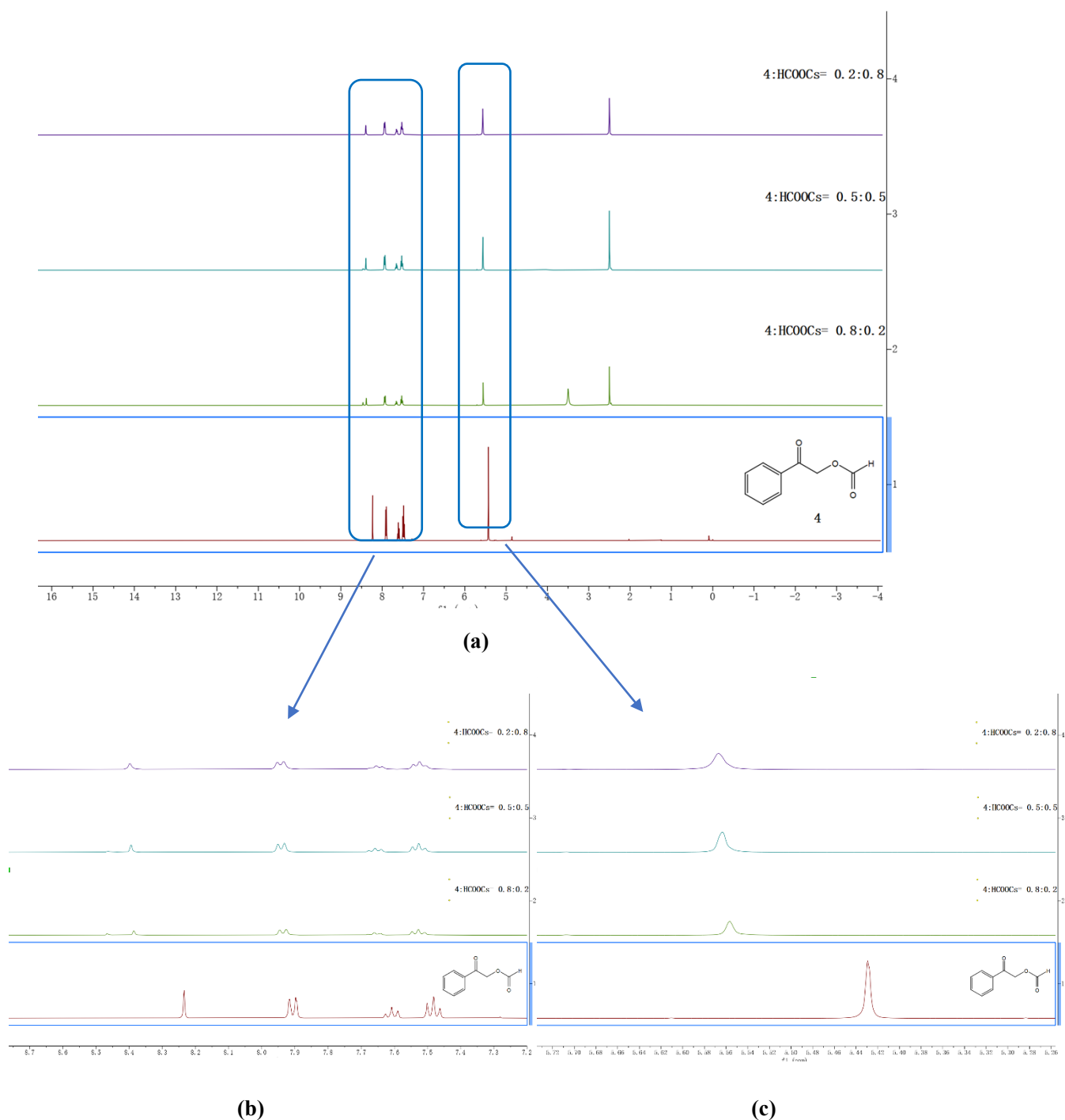
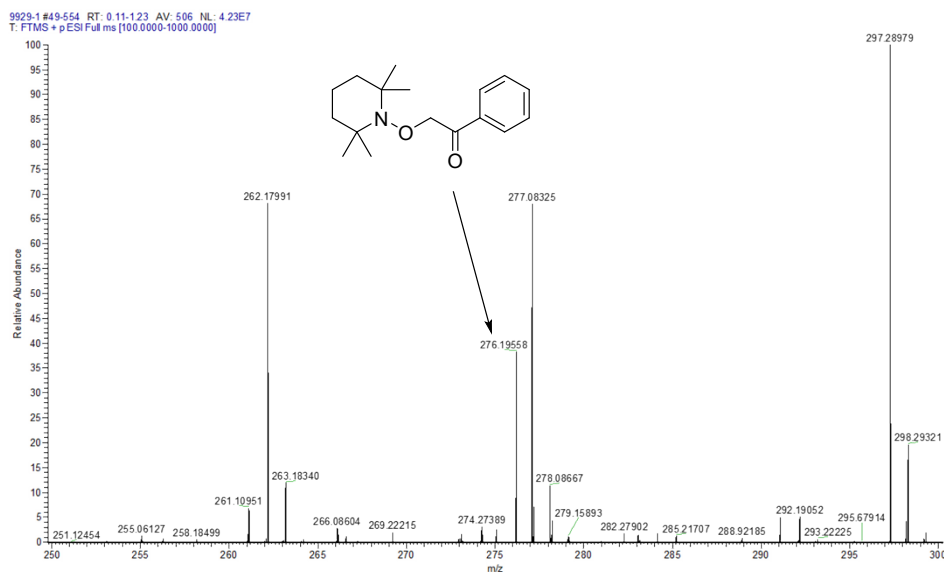


Figure S2 NMR study of mixture intermediate **4** and HCOOCs in DMSO- d_6

6.2 Radical trapping experiments

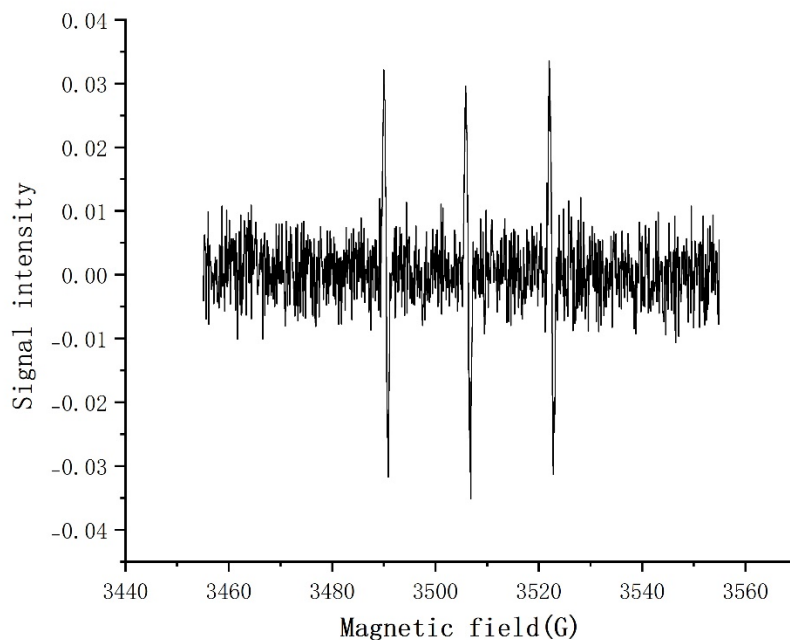
We conducted radical trapping experiments by adding TEMPO and BHT separately. HRMS tests confirmed the presence of the TEMPO radical adducts.



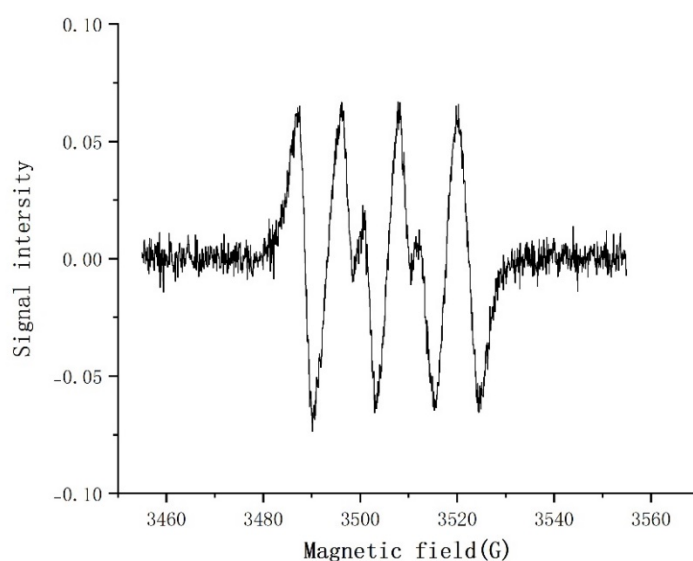
TEMPO-adduct: HRMS (ESI) calcd for $[M + H]^+$: $C_{17}H_{15}NO_2$ 276.1963, found 276.1956.

Figure S3 High-resolution mass spectrometry of radical trapping experiment

a) ESR tests



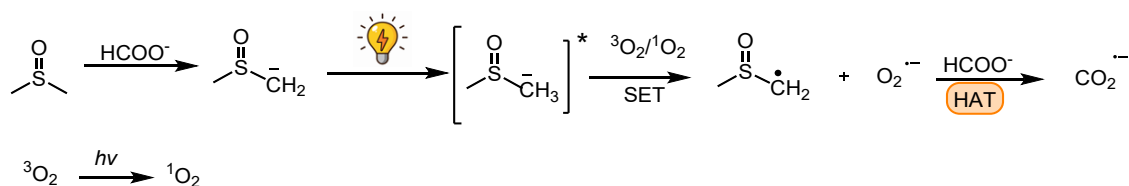
(a) In the presence of TEMP (singlet oxygen²)



(b) In the presence of DMPO (superoxide anion³)

Figure S4 ESR experiments

To understand the role of oxygen in this reaction, we conducted electron spin resonance (ESR) experiments. TEMP was added to a mixture of intermediate **4**, styrene, and HCOOCs, and the mixture was irradiated by Blue Led for 10 s for ESR testing. The results indicate the presence of $^1\text{O}_2$. In another experiment, DMPO was added as a radical scavenger to a mixture of intermediate **4**, styrene, and HCOOCs, and the $\text{O}_2^{\cdot-}$ signal was observed. The possible mechanism for the generation of reactive oxygen species and carbon dioxide radical anions from DMSO-HCOOM- O_2 under illumination conditions has been reported by Li (Scheme S3).⁴



Scheme S4 The possible mechanism for the generation of reactive oxygen species and carbon dioxide radical anions from DMSO-HCOOM- O_2 under illumination conditions.⁴

6.3 UV-visible wavelength scanning

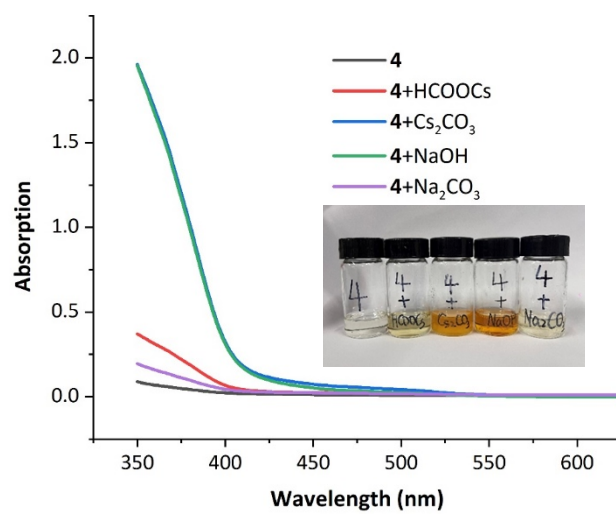
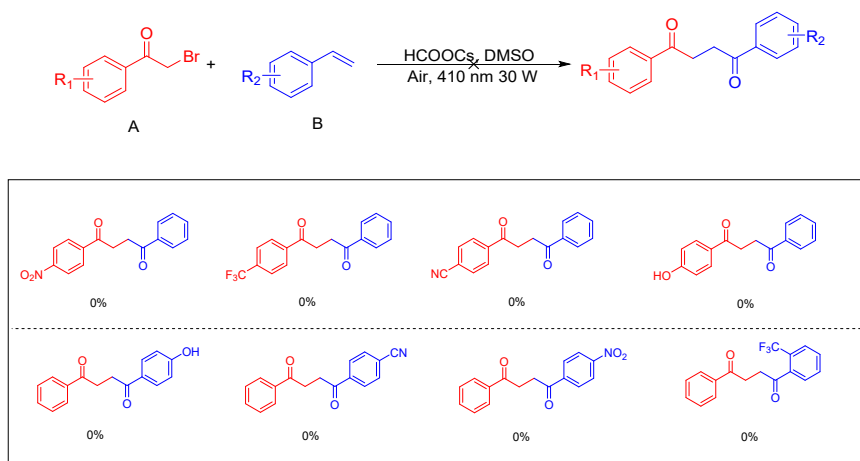


Figure S5 UV-visible wavelength scanning-different base

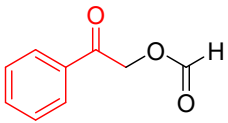
7. Unsuccessful substrates

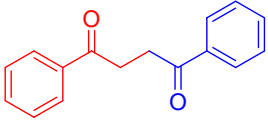


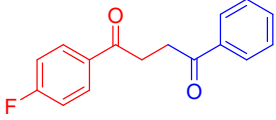
Unsuccessful examples

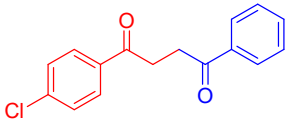
8. Characterization data

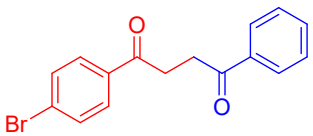
The known compounds are as follows **3a-3g**^{1f, 5}, **3i-3j**, **3l-3q**^{5a, 6}, **3r-3ab**^{1f, 5a}, **3ad**⁷, **11-14**⁸, **16**⁹ and the spectroscopic datas matched literature values.

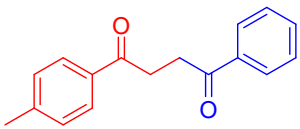
**2-oxo-2-phenylethyl formate(4)** Yellow oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.21 (s, 1H), 7.89 (d, *J* = 6.8 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 5.41 (d, *J* = 0.9 Hz, 2H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 191.3, 160.2, 134.2, 129.1, 127.9, 127.8, 65.5.

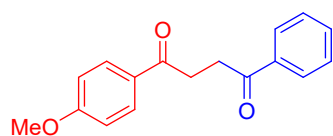
**1,4-diphenylbutane-1,4-dione(3a)** 204 mg, 86% yield, white solid. M.p.: 140–142 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 7.2 Hz, 4H), 7.58 (t, *J* = 7.3 Hz, 2H), 7.48 (t, *J* = 7.5 Hz, 4H), 3.47 (s, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 198.8, 136.9, 133.3, 128.7, 128.3, 32.7.

**1-(4-fluorophenyl)-4-phenylbutane-1,4-dione (3b, 215 mg, 84% yield; 3v 198 mg, 79% yield)**, white solid. M.p.: 96–98 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.12 – 8.00 (m, 4H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.15 (t, *J* = 8.6 Hz, 2H), 3.50 – 3.41 (m, 4H). ¹³C NMR (150 MHz, Chloroform-*d*) δ 198.7, 197.3, 166.0 (d, *J* = 253.0 Hz), 136.9, 133.4, 133.4 (d, *J* = 3.0 Hz), 130.9 (d, *J* = 9.3 Hz), 128.8, 128.3, 115.8 (d, *J* = 21.6 Hz), 32.7, 32.6.

**1-(4-chlorophenyl)-4-phenylbutane-1,4-dione (3c, 220 mg, 81% yield; 3w 210 mg, 80% yield)**, white solid. M.p.: 42–44 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 7.0 Hz, 2H), 7.98 (d, *J* = 8.6 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.48 (dd, *J* = 14.1, 8.3 Hz, 4H), 3.44 (dd, *J* = 12.7, 5.9 Hz, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 198.7, 197.7, 139.8, 136.8, 135.3, 133.4, 129.7, 129.1, 128.8, 128.3, 32.7, 32.7.

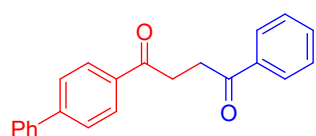
**1-(4-bromophenyl)-4-phenylbutane-1,4-dione (3d, 273 mg, 86% yield; 3x 200 mg, 77% yield)**, white solid. M.p.: 113–115 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 7.1 Hz, 2H), 7.91 (d, *J* = 8.5 Hz, 2H), 7.63 (d, *J* = 8.6 Hz, 2H), 7.58 (d, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 2H), 3.46 (d, *J* = 5.1 Hz, 2H), 3.42 (d, *J* = 5.1 Hz, 2H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 198.7, 197.9, 136.8, 135.7, 133.4, 132.1, 129.8, 128.8, 128.5, 128.3, 32.7, 32.7.

**1-phenyl-4-(p-tolyl)butane-1,4-dione (3e, 224 mg, 89% yield; 3y 236 mg, 84% yield)**, white solid. M.p.: 113–115 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 7.0 Hz, 2H), 7.94 (d, *J* = 8.2 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 7.9 Hz, 2H), 3.45 (d, *J* = 1.8 Hz, 4H), 2.42 (s, 3H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 199.0, 198.5, 144.1, 137.0, 134.5, 133.3, 129.4, 128.7, 128.4, 128.3, 32.8, 32.6, 21.8.



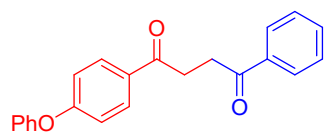
1-(4-methoxyphenyl)-4-phenylbutane-1,4-dione

(**3f**, 246 mg, 92% yield; **3s** 227 mg, 81% yield), white solid. M.p.: 98–100 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.08 – 7.98 (m, 4H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 6.95 (d, *J* = 8.9 Hz, 2H), 3.88 (s, 3H), 3.49 – 3.37 (m, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 199.0, 197.4, 163.7, 137.0, 133.2, 130.5, 130.0, 128.7, 128.3, 113.9, 55.6, 32.8, 32.4.



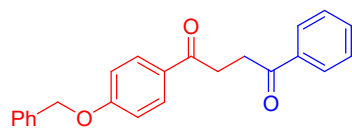
1-([1,1'-biphenyl]-4-yl)-4-phenylbutane-1,4-dione

(**3g**, 251 mg, 80% yield; **3z** 200 mg, 79% yield), white solid. M.p.: 99-101 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.12 (d, *J* = 8.4 Hz, 2H), 8.06 (d, *J* = 7.1 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 2H), 7.66 – 7.62 (m, 2H), 7.58 (t, *J* = 7.1 Hz, 1H), 7.51 – 7.45 (m, 4H), 7.42 – 7.40 (m, 1H), 3.50 (s, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 198.9, 146.0, 140.1, 135.7, 133.3, 129.1, 128.9, 128.8, 128.4, 128.3, 127.5, 127.4, 32.8, 32.8.



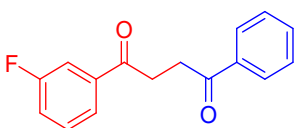
1-(4-phenoxyphenyl)-4-phenylbutane-1,4-dione(**3h**)

(**3h**, 270 mg, 82% yield; **3aa** 160 mg, 80% yield), white solid. M.p.: 113-115 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.07 – 7.98 (m, 4H), 7.52 – 7.35 (m, 4H), 7.11 – 6.98 (m, 4H), 3.44 (tt, *J* = 10.3, 5.0 Hz, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 198.9, 197.4, 162.2, 155.7, 136.9, 133.3, 131.6, 130.5, 130.2, 128.7, 128.3, 124.7, 120.3, 117.5, 32.8, 32.5. HRMS (ESI) calcd for [M + H⁺]: C₂₂H₁₉O₃ 331.1328, found 331.1330



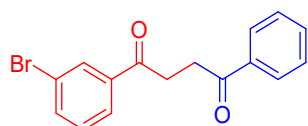
1-(4-iodine)- 4-(phenylmethoxy)phenyl-1,4-dione(**3i**)

230 mg, 82% yield, white solid. M.p.: 80-82 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.03 (t, *J* = 7.7 Hz, 4H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.51 – 7.34 (m, 7H), 7.03 (d, *J* = 8.9 Hz, 2H), 5.15 (s, 2H), 3.48 – 3.38 (m, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 199.2, 197.5, 160.1, 133.3, 130.6, 130.1, 128.9, 128.7, 128.4, 128.3, 127.6, 114.8, 70.3, 32.9, 32.4.



1-(3-fluorophenyl)-4-phenylbutane-1,4-dione

(**3j**, 220 mg, 86% yield; **3ae** 208 mg, 83% yield), white solid. M.p.: 100-102 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.69 (d, *J* = 7.8 Hz, 2H), 7.63 (dt, *J* = 9.0, 2.1 Hz, 2H), 7.50 (td, *J* = 8.0, 5.4 Hz, 2H), 7.38 – 7.33 (m, 3H), 4.86 (s, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 198.6, 197.6, 163.0 (d, *J* = 246.4 Hz), 139.0 (d, *J* = 6.1 Hz), 136.8, 133.4, 130.4 (d, *J* = 7.6 Hz), 128.8, 128.3, 124.0 (d, *J* = 3.0 Hz), 120.3 (d, *J* = 21.4 Hz), 115.0 (d, *J* = 22.1 Hz), 32.9, 32.7.

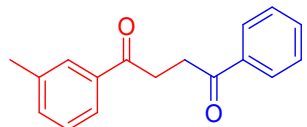


1-(3- bromophenyl)-4-phenylbutane-1,4-dione(**3k**)

225 mg, 68% yield, white solid. M.p.: 82-84 °C. ¹H NMR (400 MHz, Chloroform-

d) δ 8.16 (t, J = 1.9 Hz, 1H), 8.03 (d, J = 7.1 Hz, 2H), 7.96 (dt, J = 7.8, 1.4 Hz, 1H), 7.70 (ddd, J = 8.0, 2.1, 1.0 Hz, 1H), 7.58 (t, J = 7.4 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 7.36 (t, J = 7.9 Hz, 1H), 3.44 (ddd, J = 15.6, 5.1, 1.0 Hz, 4H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 198.5, 197.5, 138.7, 136.8, 136.1, 133.4, 131.4, 130.4, 128.8, 128.3, 126.8, 123.1, 32.8, 32.7. HRMS (ESI) calcd for $[\text{M} + \text{H}^+]$: $\text{C}_{16}\text{H}_{14}\text{BrO}_2$ 317.0172, found 317.0169

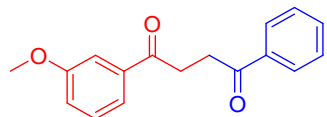
1-(3-methyl)-4-phenylbutane-1,4-dione



(**3l**, 230 mg, 83% yield; **3af** 190 mg, 76% yield), white solid.

M.p.: 76-78 °C. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.07 – 8.01 (m, 2H), 7.84 (d, J = 6.4 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.48 (t, J = 7.7 Hz, 2H), 7.37 (d, J = 7.9 Hz, 2H), 3.46 (d, J = 1.4 Hz, 4H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 199.1, 198.9, 138.5, 137.0, 134.1, 133.3, 128.8, 128.7, 128.6, 128.3, 125.5, 32.8, 21.5.

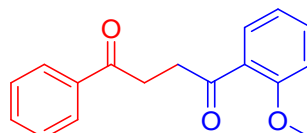
1-(3-methoxyphenyl)-4-phenylbutane-1,4-dione(3m) 246 mg, 92%



yield, white solid. M.p.: 76-78 °C. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, J = 7.0 Hz, 2H), 7.64 (d, J = 7.7 Hz, 1H), 7.59 (d, J =

7.4 Hz, 1H), 7.57 – 7.53 (m, 1H), 7.48 (t, J = 7.6 Hz, 2H), 7.39 (t, J = 7.9 Hz, 1H), 7.13 (ddd, J = 8.2, 2.7, 1.0 Hz, 1H), 3.86 (s, 3H), 3.46 (s, 4H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 198.8, 198.7, 160.0, 138.3, 136.9, 133.3, 129.8, 128.8, 128.3, 121.0, 119.9, 112.4, 55.6, 32.9, 32.8.

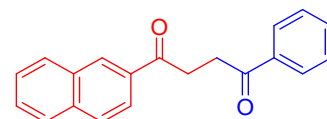
1-(2-methoxyphenyl)-4-phenylbutane-1,4-dione



(**3n**, 200 mg, 69% yield; **3ab** 216 mg, 77% yield), colorless

liquid. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.03 (d, J = 7.2 Hz, 2H), 7.77 (dd, J = 7.7, 1.9 Hz, 1H), 7.57 (t, J = 7.3 Hz, 1H), 7.47 (t, J = 7.6 Hz, 3H), 7.08 – 6.94 (m, 2H), 3.93 (s, 3H), 3.44 (dd, J = 16.3, 5.4 Hz, 4H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 200.8, 199.2, 159.0, 137.2, 133.7, 133.1, 130.7, 128.7, 128.3, 128.1, 120.8, 111.8, 55.7, 38.1, 33.2.

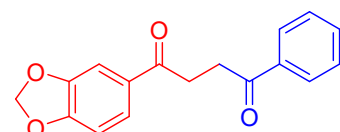
1-(naphthalen-2-yl)-4-phenylbutane-1,4-dione



(**3o**, 216 mg, 75% yield; **3ai** 218 mg, 84% yield), white solid. M.p.:

146-148 °C. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.59 (d, J = 1.7 Hz, 1H), 8.16 – 8.03 (m, 3H), 8.02 – 7.95 (m, 1H), 7.90 (dd, J = 9.9, 7.8 Hz, 2H), 7.66 – 7.53 (m, 3H), 7.49 (t, J = 7.6 Hz, 2H), 3.61 (dd, J = 7.4, 6.0 Hz, 2H), 3.53 (dd, J = 6.8, 5.3 Hz, 2H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 198.9, 198.8, 137.0, 135.8, 134.3, 133.3, 132.7, 130.0, 129.8, 128.8, 128.6, 128.3, 127.9, 126.9, 124.0, 32.9, 32.8.

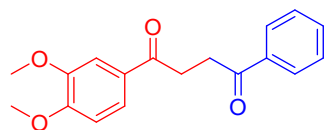
1-(benzo[d][1,3]dioxol-5-yl)-4-phenylbutane-1,4-dione(3p) 231



mg, 82% yield, white solid. M.p.: 132-134 °C. ^1H NMR (400 MHz,

Chloroform-*d*) δ 8.04 (d, J = 6.9 Hz, 2H), 7.67 (dd, J = 8.2, 1.7 Hz, 1H), 7.58 (t, J = 7.4 Hz, 1H), 7.52 – 7.38 (m, 3H), 6.87 (d, J = 8.2

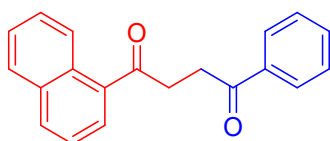
Hz, 1H), 6.05 (s, 2H), 3.49 – 3.35 (m, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 198.9, 196.9, 152.0, 148.3, 137.0, 133.3, 131.8, 128.7, 128.3, 124.5, 108.1, 108.1, 102.0, 32.9, 32.5.



1-(3,4-dimethoxyphenyl)-4-phenylbutane-1,4-dione(3q) 247

mg, 83% yield, white solid. M.p.: 79-81 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 7.0 Hz, 2H), 7.70 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.56 (d, *J* = 2.1 Hz, 2H), 7.47 (t, *J* = 7.7 Hz, 2H), 6.91 (d, *J* =

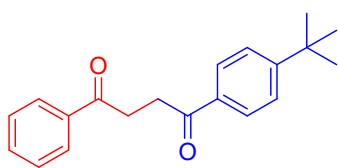
8.4 Hz, 1H), 3.94 (d, *J* = 8.5 Hz, 6H), 3.43 (s, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 199.1, 197.4, 153.5, 149.1, 137.0, 133.3, 130.2, 128.7, 128.3, 122.9, 110.3, 110.2, 56.2, 56.1, 32.9, 32.3.



1-(naphthalen-1-yl)-4-phenylbutane-1,4-dione(3r) 227 mg, 79%

yield, white solid. M.p.: 77-78 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.60 (dd, *J* = 8.5, 1.2 Hz, 1H), 8.07 (ddd, *J* = 7.2, 3.0, 1.2 Hz, 3H), 8.00 (d, *J* = 8.2 Hz, 1H), 7.88 (dd, *J* = 8.1, 1.6 Hz,

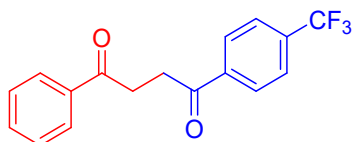
1H), 7.62 – 7.46 (m, 6H), 3.60 – 3.48 (m, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 203.0, 198.8, 137.0, 136.2, 134.1, 133.3, 132.7, 130.3, 128.8, 128.5, 128.3, 128.0, 127.8, 126.6, 126.0, 124.6, 36.1, 33.2.



1-(4-(tert-butyl)phenyl)-4-phenylbutane-1,4-dione(3t) 223 mg,

76% yield, white solid. M.p.: 60-62 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 7.0 Hz, 2H), 7.98 (d, *J* = 8.6 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.51 – 7.45 (m, 4H), 3.45 (s, 4H), 1.35 (s,

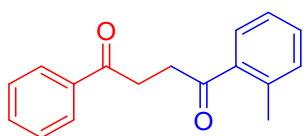
9H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 199.0, 198.5, 157.0, 137.0, 134.4, 133.3, 128.7, 128.3, 128.3, 125.7, 35.3, 32.8, 32.7, 31.3.



1-phenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione(3u)

254 mg, 83% yield, white solid. M.p.: 72-74 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.15 (d, *J* = 8.1 Hz, 2H), 8.04 (d, *J* = 7.1 Hz, 2H), 7.76 (d, *J* = 8.2 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.49

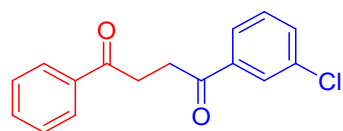
(dd, *J* = 8.4, 7.0 Hz, 2H), 3.55 – 3.42 (m, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 200.1, 197.8, 138.8, 136.7, 133.5, 128.8, 128.6, 128.3, 126.8, 125.9, 125.9, 77.5, 77.2, 76.8, 33.0, 32.7.



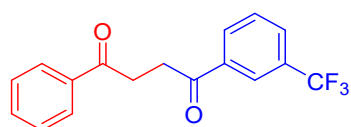
1-(2-methyl)-4-phenylbutane-1,4-dione (3ac) 220 mg, 76% yield,

white solid. M.p.: 60-62 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 7.2 Hz, 2H), 7.81 (d, *J* = 7.8 Hz, 1H), 7.58 (d, *J* = 7.7 Hz,

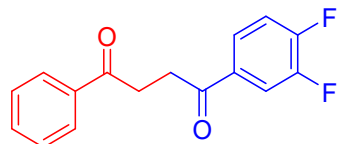
1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.41 – 7.35 (m, 1H), 7.31 – 7.25 (m, 2H), 3.48 – 3.33 (m, 4H), 2.50 (s, 3H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 202.8, 198.8, 138.2, 138.1, 137.0, 133.3, 132.0, 131.4, 128.7, 128.7, 128.3, 125.9, 35.5, 33.0, 21.4. HRMS (ESI) calcd for [M + H⁺]: C₁₇H₁₇O₂ 253.1223, found 253.1229.



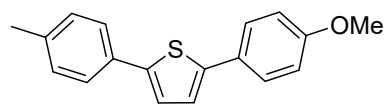
1-(3-bromophenyl)-4-phenylbutane-1,4-dione (3ad) 192 mg, 71% yield, white solid. M.p.: 75-77 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.10 – 7.99 (m, 3H), 7.92 (d, *J* = 7.8 Hz, 1H), 7.61 – 7.53 (m, 2H), 7.46 (dt, *J* = 22.8, 7.9 Hz, 3H), 3.52 – 3.38 (m, 4H). ¹³C NMR (100MHz, Chloroform-*d*) δ 198.6, 197.6, 138.5, 136.8, 135.1, 133.4, 133.2, 130.1, 128.8, 128.4, 128.3, 126.4, 32.8, 32.7.



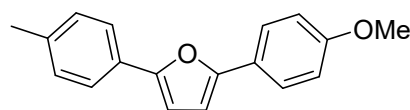
1-(3-trifluoromethyl)-4-phenylbutane-1,4-dione (3ag) 235 mg, 85% yield, white solid. M.p.: 71-73 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.29 (s, 1H), 8.23 (d, *J* = 7.9 Hz, 1H), 8.04 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.84 (d, *J* = 7.7 Hz, 1H), 7.72 – 7.55 (m, 2H), 7.49 (t, *J* = 7.6 Hz, 2H), 3.49 (h, *J* = 5.4 Hz, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 199.1, 198.9, 138.5, 137.0, 134.1, 133.3, 128.8, 128.8, 128.6, 128.3, 125.5, 32.7 (d, *J* = 11.6 Hz). HRMS (ESI) calcd for [M + H⁺]: C₁₇H₁₄F₃O₂ 307.0940, found 307.0942.



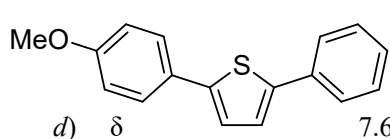
1-(3,4-difluorine)-4-phenylbutane-1,4-dione(3ah) 220 mg, 82% yield, white solid. M.p.: 89-91 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.12 – 7.99 (m, 2H), 7.91 – 7.79 (m, 2H), 7.64 – 7.55 (m, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.35 – 7.28 (m, 1H), 3.47 (t, *J* = 5.8 Hz, 2H), 3.40 (dd, *J* = 6.5, 5.0 Hz, 2H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 198.5, 196.4, 136.8, 133.5, 128.5 (d, *J* = 53.6 Hz), 125.4, 117.7 (d, *J* = 18 Hz), 32.7, 32.6. HRMS (ESI) calcd for [M + H⁺]: C₁₆H₁₃F₂O₂ 275.0878, found 275.0879.



2-(4-methoxyphenyl)-5-(p-tolyl)thiophene(11) 103 mg, 85% yield, white solid. M.p.: 158-160 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.55 (dd, *J* = 14.1, 8.3 Hz, 4H), 7.25 – 7.14 (m, 4H), 6.94 (d, *J* = 8.7 Hz, 2H), 3.85 (s, 3H), 2.39 (s, 3H). ¹³C NMR (150 MHz, Chloroform-*d*) δ 159.3, 143.2, 142.9, 137.3, 131.8, 129.7, 127.5, 127.0, 125.6, 123.6, 123.0, 114.5, 55.5, 21.3.

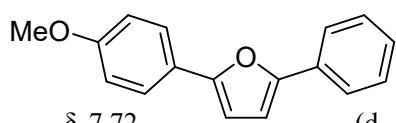


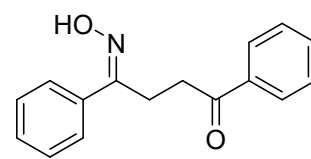
2-(4-methoxyphenyl)-5-(p-tolyl)furan(12) 159 mg, 88% yield, white solid. M.p.: 166-168 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.72 – 7.67 (m, 2H), 7.64 (d, *J* = 8.0 Hz, 2H), 7.25 – 7.19 (m, 2H), 6.96 (d, *J* = 8.9 Hz, 2H), 6.67 (d, *J* = 3.4 Hz, 1H), 6.60 (d, *J* = 3.4 Hz, 1H), 3.85 (s, 3H), 2.39 (s, 3H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 159.2, 153.2, 153.1, 137.1, 129.5, 128.4, 125.3, 124.2, 123.7, 114.3, 106.6, 105.7, 55.5, 21.4.

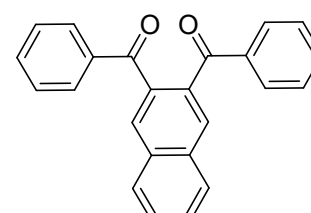


2-(4-methoxyphenyl)-5-phenylthiophene(13) 172 mg, 86% yield, yellow solid. M.p.: 146-148 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.61 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 8.8 Hz, 2H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.27 – 7.23 (m, 2H), 7.17 (d, *J* = 3.8 Hz, 1H), 6.92 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H). ¹³C NMR

(100 MHz, Chloroform-*d*) δ 159.5, 143.8, 142.8, 134.6, 129.0, 127.5, 127.1, 125.7, 124.1, 123.1, 114.5, 55.5.

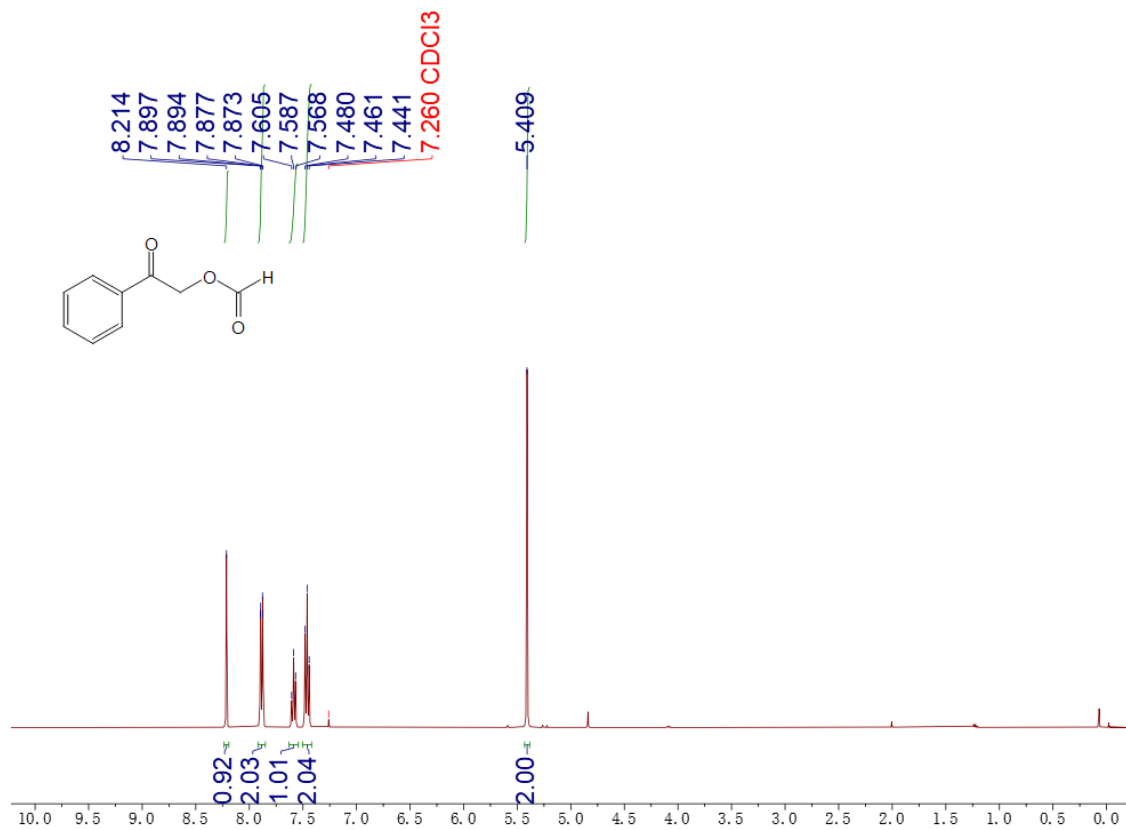
 **2-(4-methoxyphenyl)-5-phenylfuran(14)** 191 mg, 87% yield, white solid. M.p.: 148-150 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.72 (d, *J* = 7.0 Hz, 2H), 7.67 (d, *J* = 8.9 Hz, 2H), 7.39 (dd, *J* = 8.4, 7.1 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 8.9 Hz, 2H), 6.71 (d, *J* = 3.4 Hz, 1H), 6.59 (d, *J* = 3.4 Hz, 1H), 3.84 (s, 3H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 159.3, 153.7, 152.9, 131.1, 128.8, 127.2, 125.4, 124.1, 123.7, 114.4, 107.3, 105.8, 55.5.

 **(Z)-4-(hydroxyimino)-1,4-diphenylbutan-1-one(15)** 166 mg, 72% yield, white solid. M.p.: 90-92 °C. ¹H NMR (600 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.69 – 7.63 (m, 2H), 7.55 (td, *J* = 7.5, 1.3 Hz, 1H), 7.47 – 7.42 (m, 2H), 7.42 – 7.37 (m, 3H), 3.33 – 3.20 (m, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 199.0, 159.1, 136.7, 135.4, 133.3, 129.6, 128.8, 128.7, 128.3, 126.4, 35.1, 21.5. HRMS (ESI) calcd for [M + H⁺]: C₁₆H₁₅NO₂ 254.1183, found 254.1189.

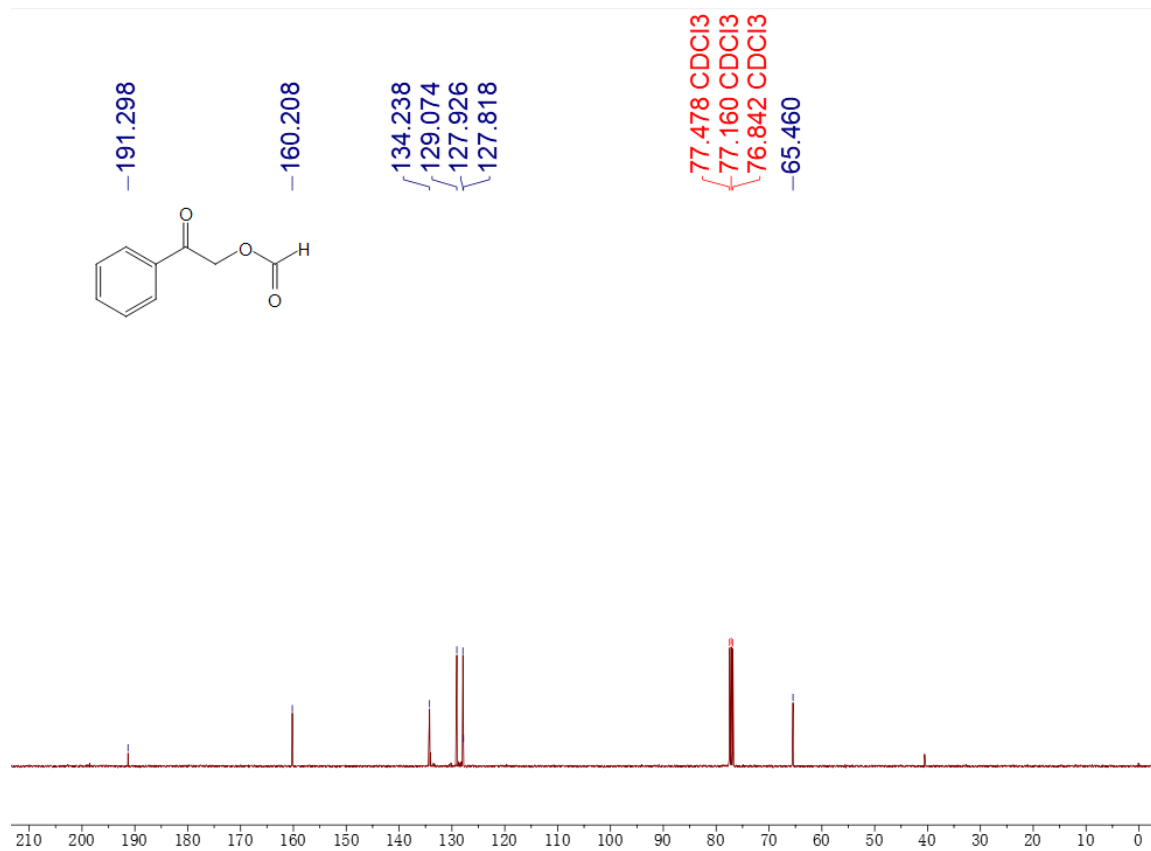
 **naphthalene-2,3-diylbis(phenylmethanone)(16)** 133 mg, 68% yield, white solid. M.p.: 144-146 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.10 (s, 2H), 7.94 (dd, *J* = 6.2, 3.3 Hz, 2H), 7.87 – 7.79 (m, 4H), 7.66 (dd, *J* = 6.2, 3.2 Hz, 2H), 7.53 (d, *J* = 7.4 Hz, 2H), 7.41 (t, *J* = 7.7 Hz, 4H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 196.4, 137.6, 137.1, 133.1, 133.0, 130.9, 130.0, 128.9, 128.8, 128.5.

9. Copies of ¹H NMR and ¹³C NMR

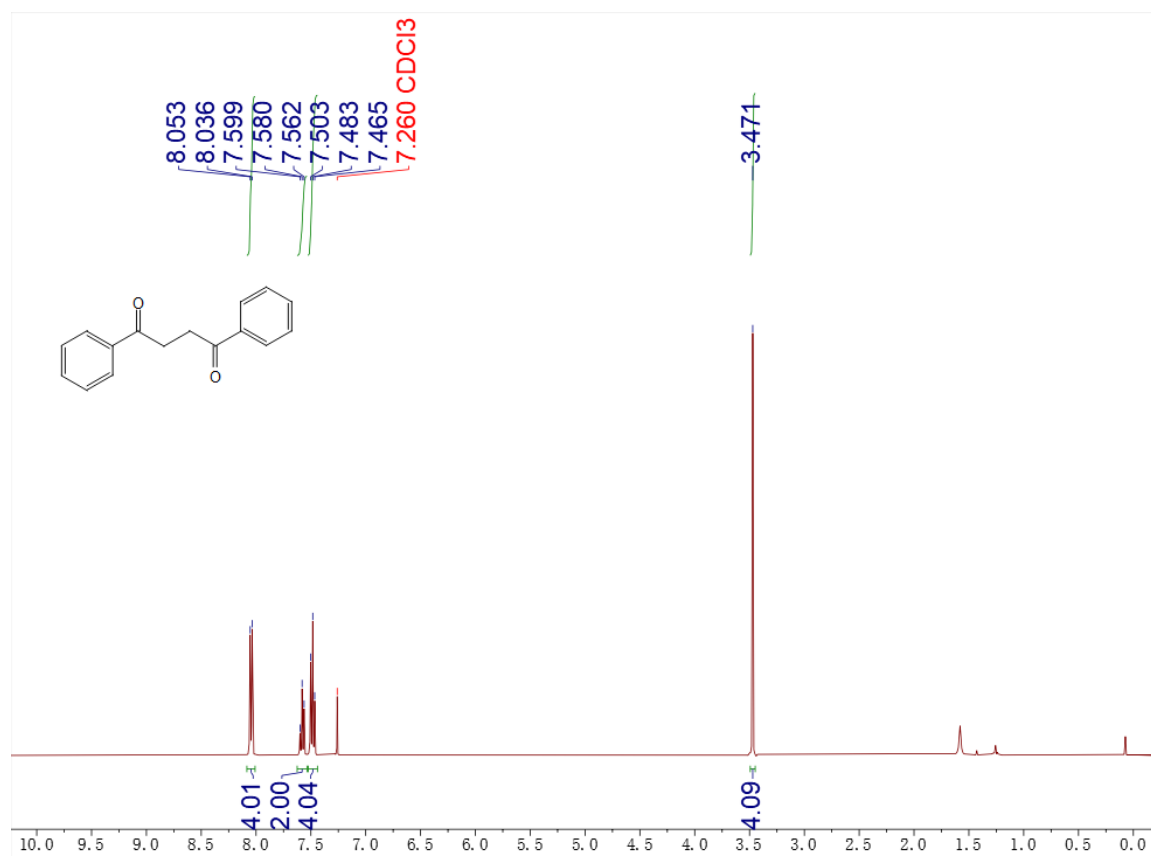
¹H NMR spectra (400 MHz) in CDCl₃ of compound 4



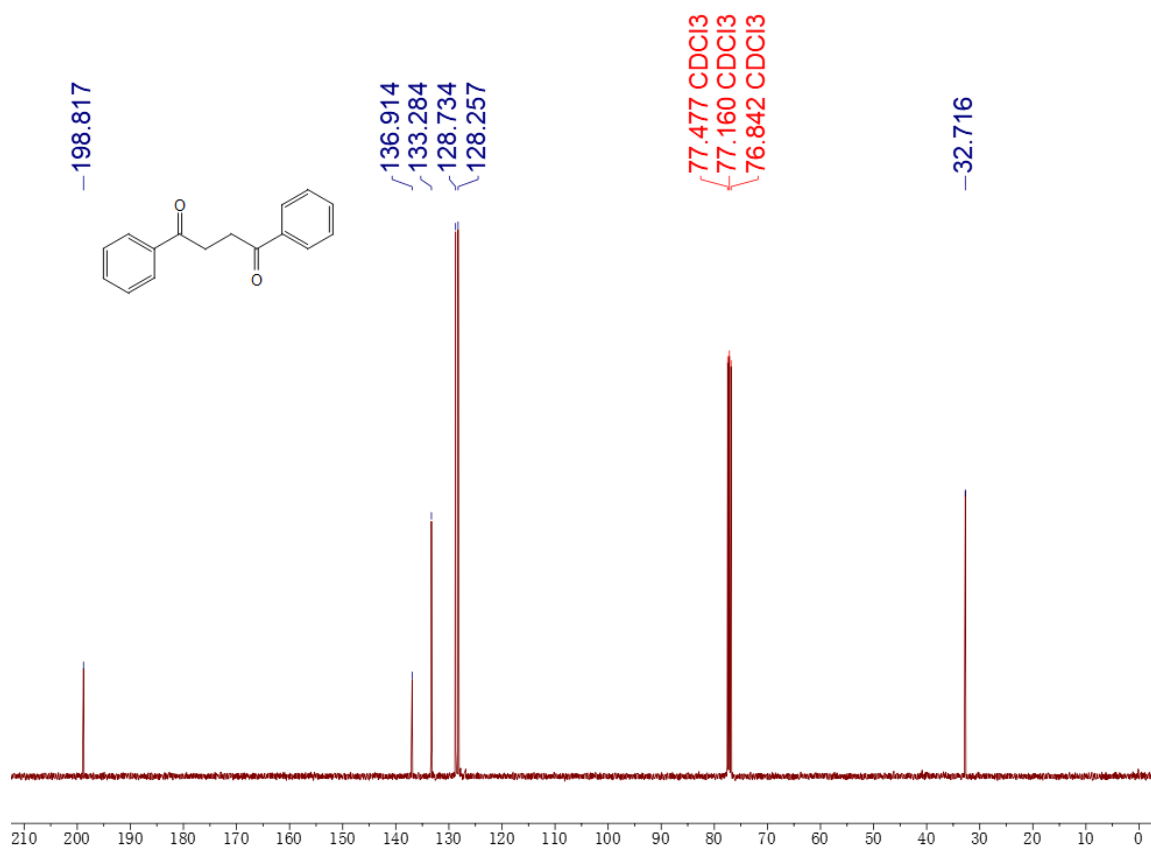
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **4**



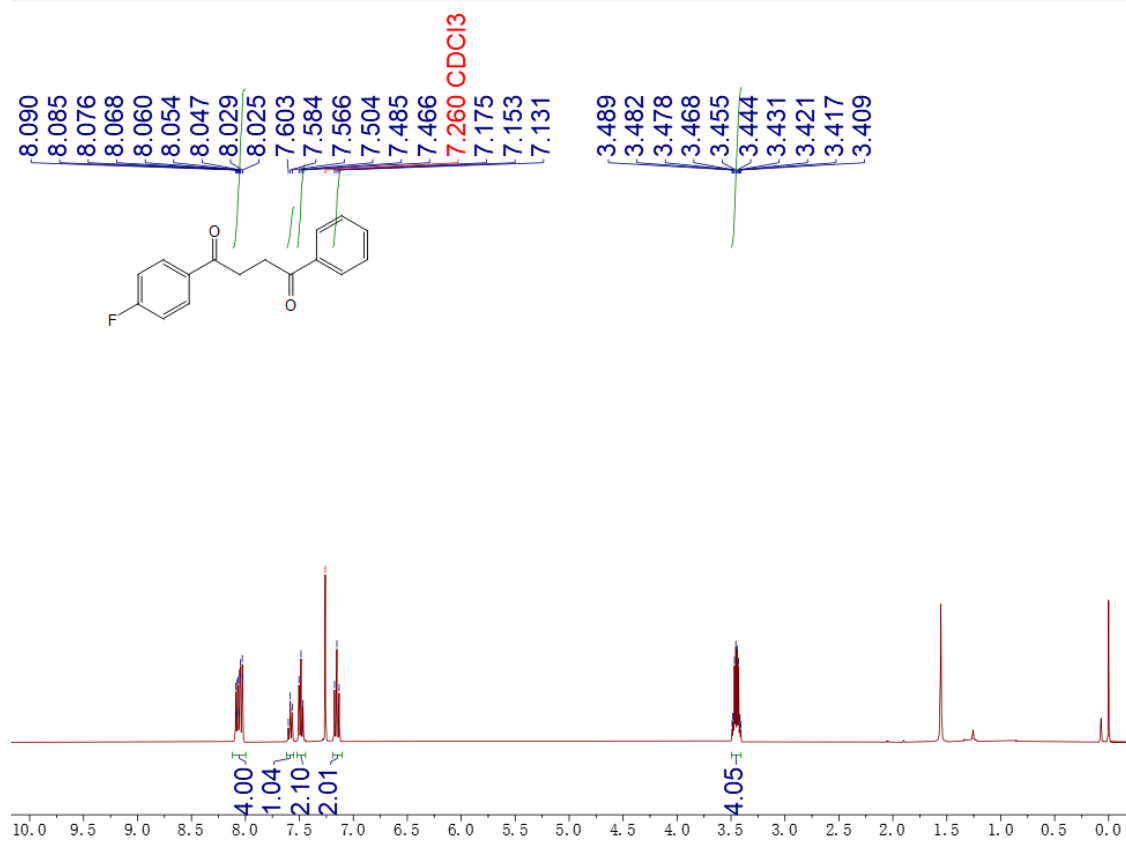
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3a**



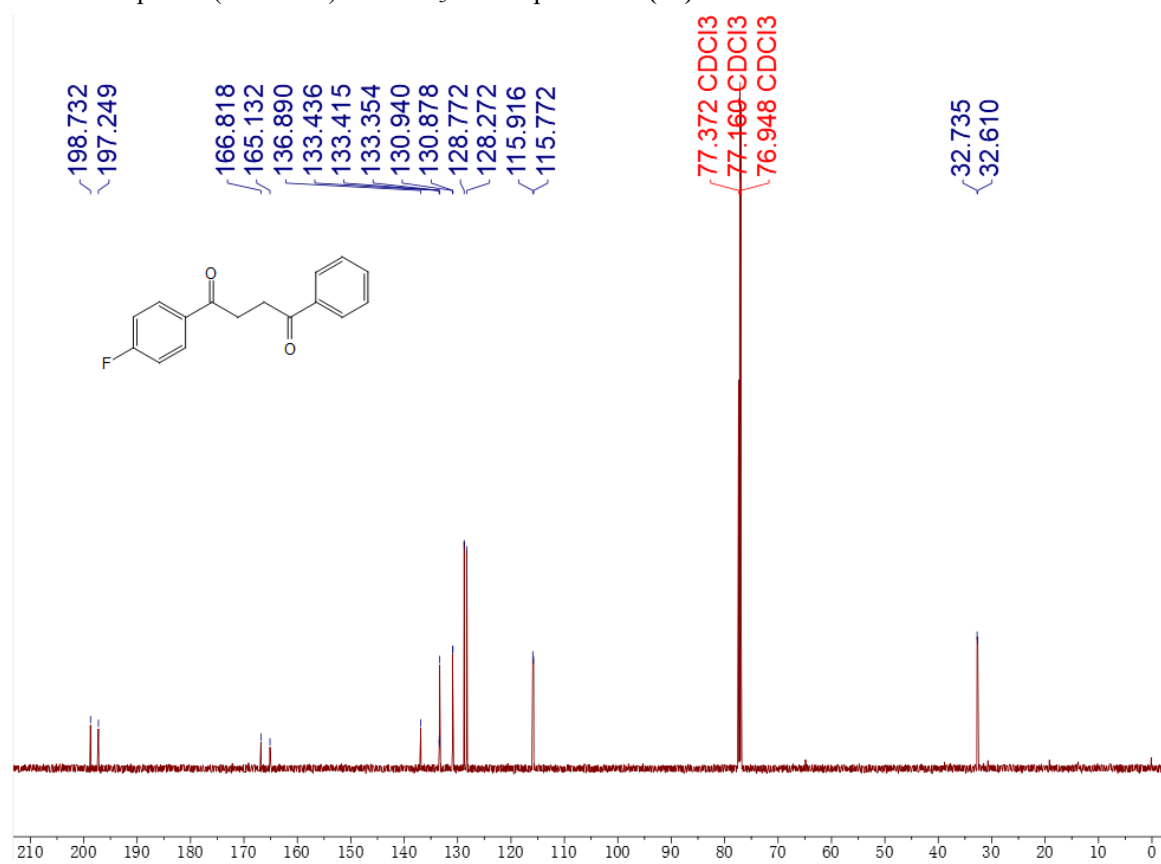
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3a**



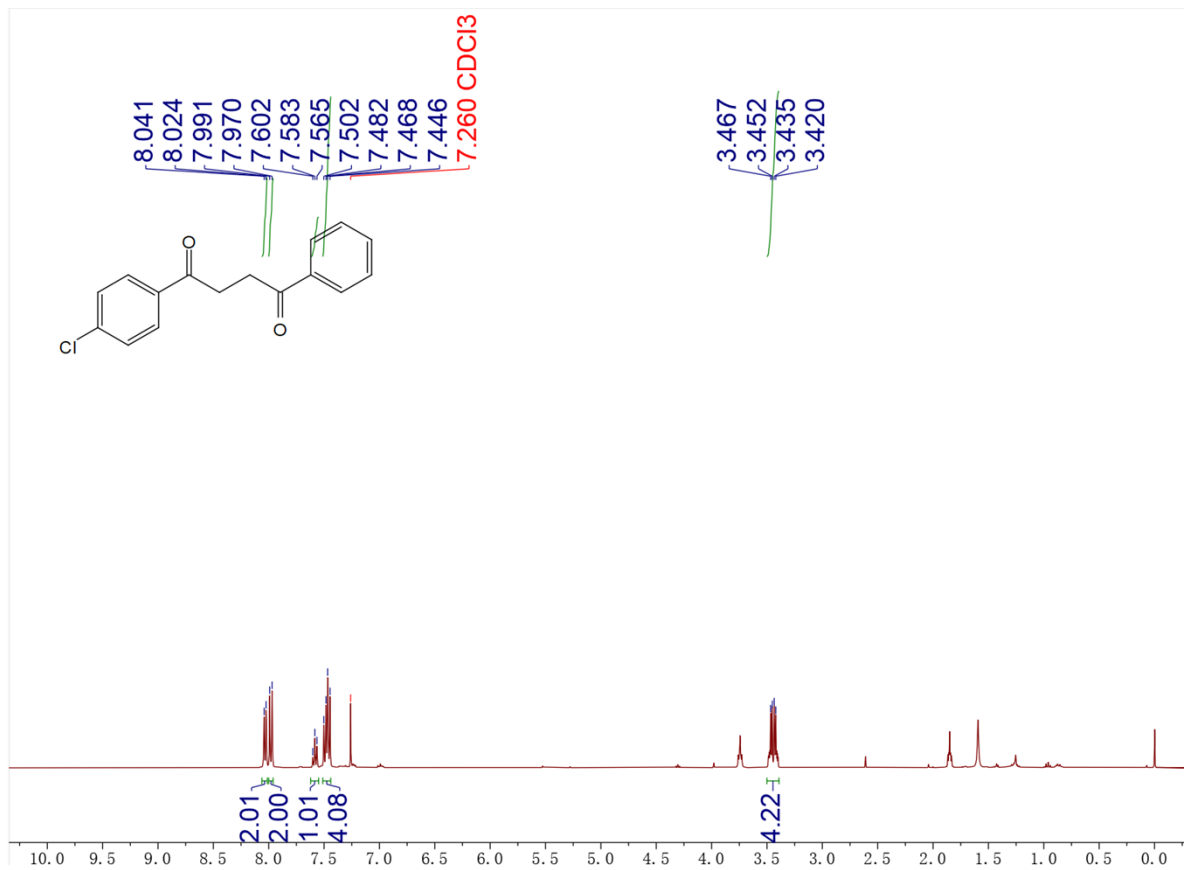
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3b (3v)**



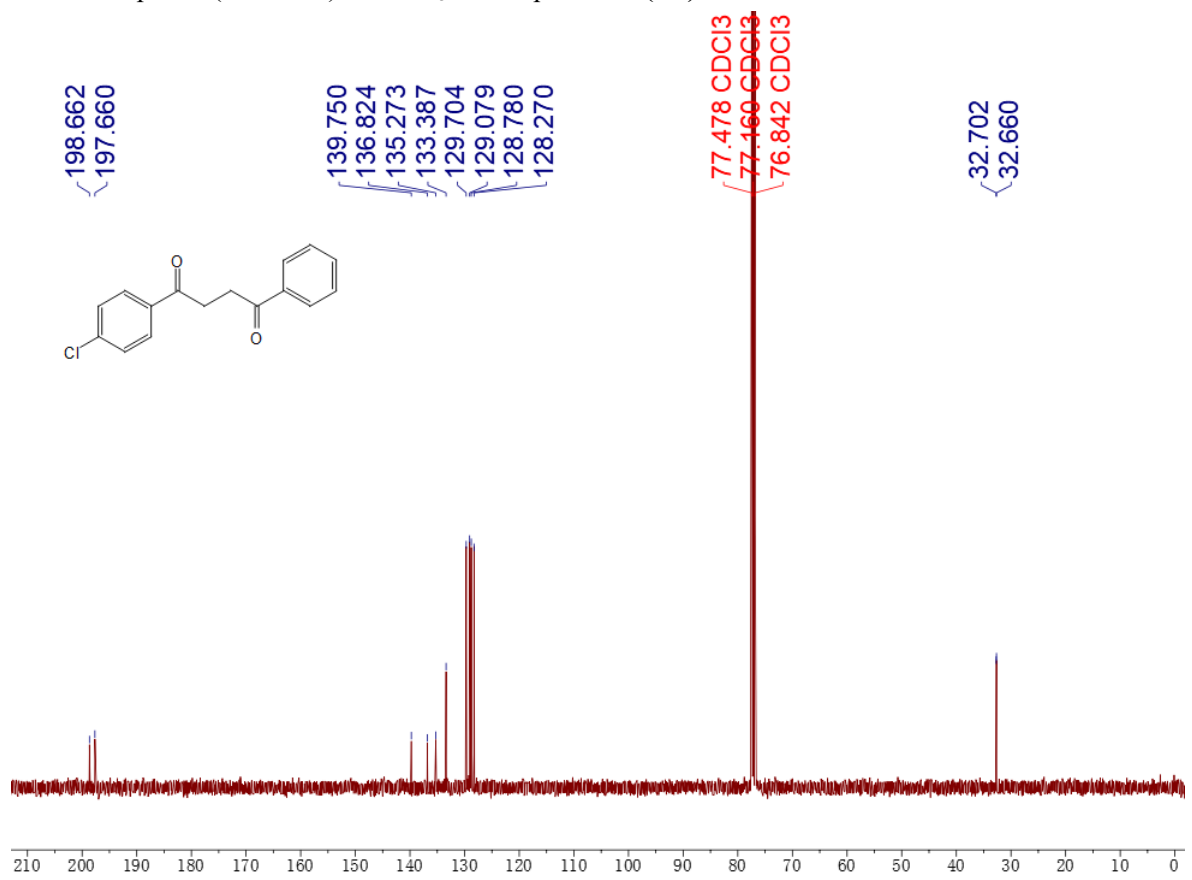
¹³C NMR spectra (600 MHz) in CDCl₃ of compound **3b (3v)**



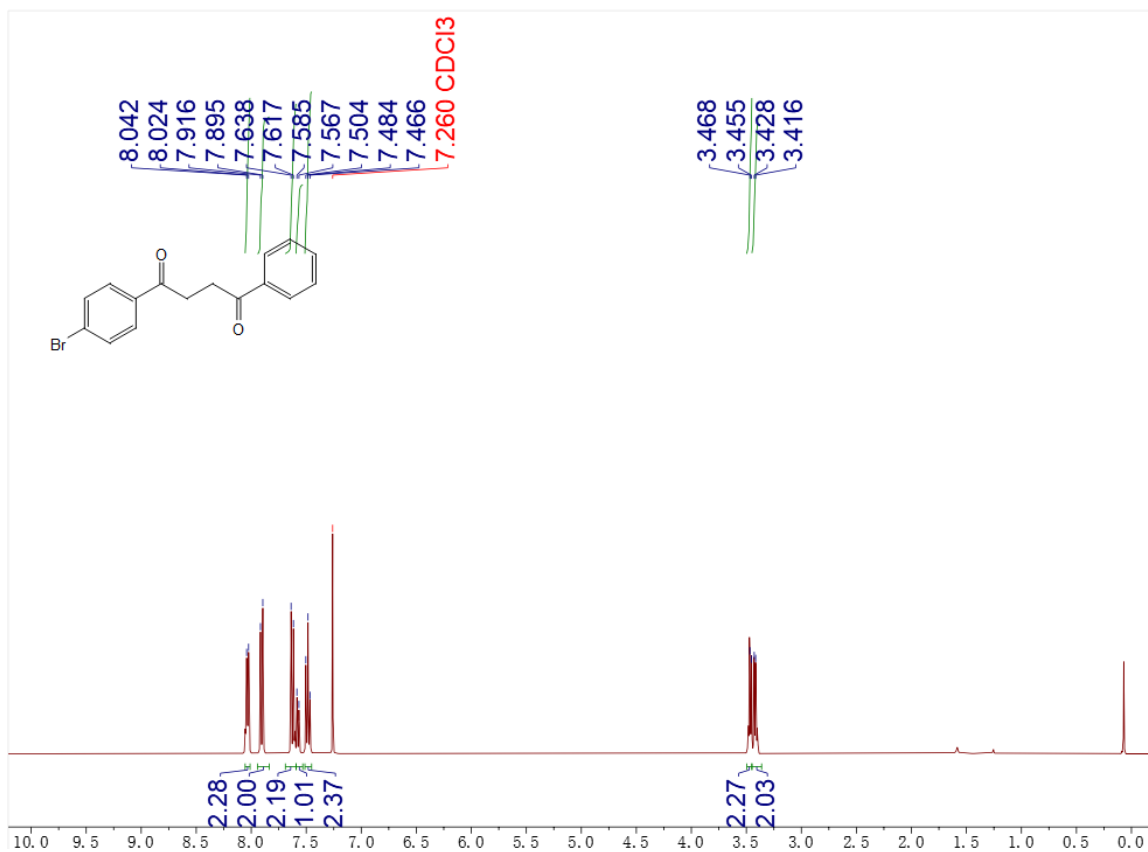
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3c (3w)**



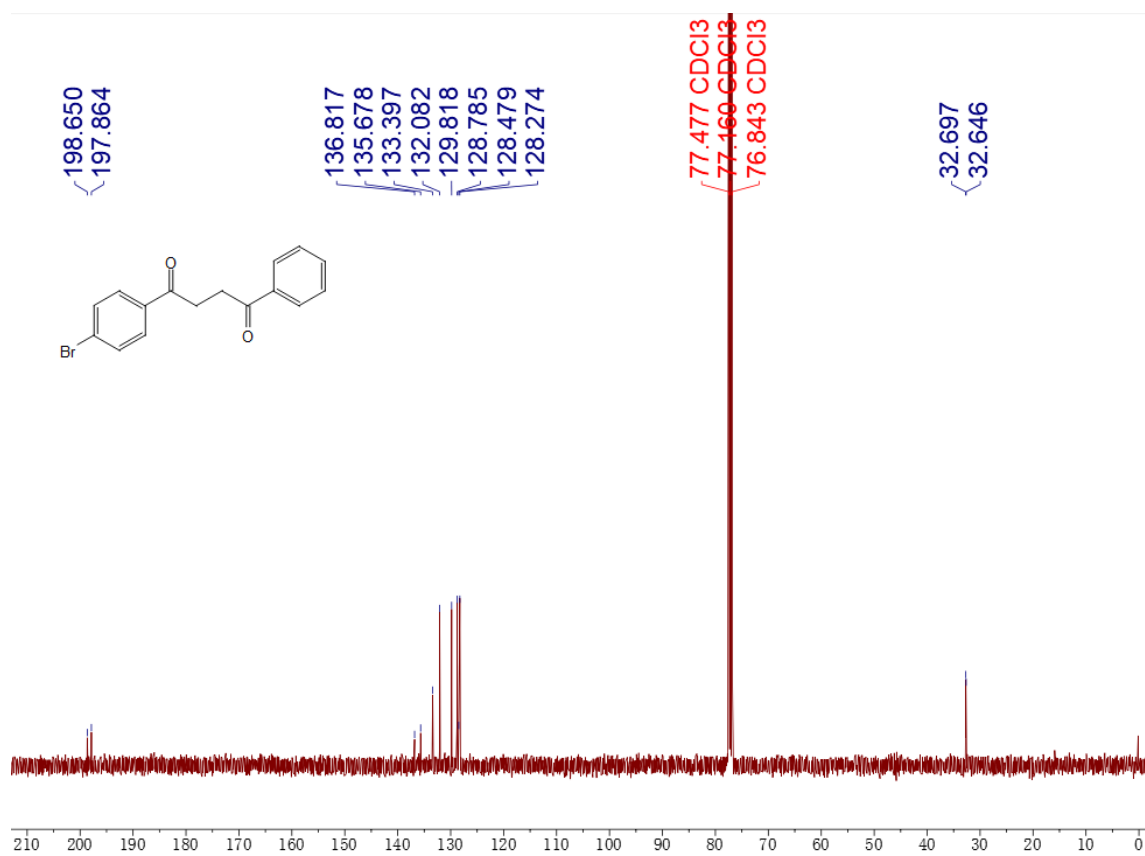
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3c (3w)**



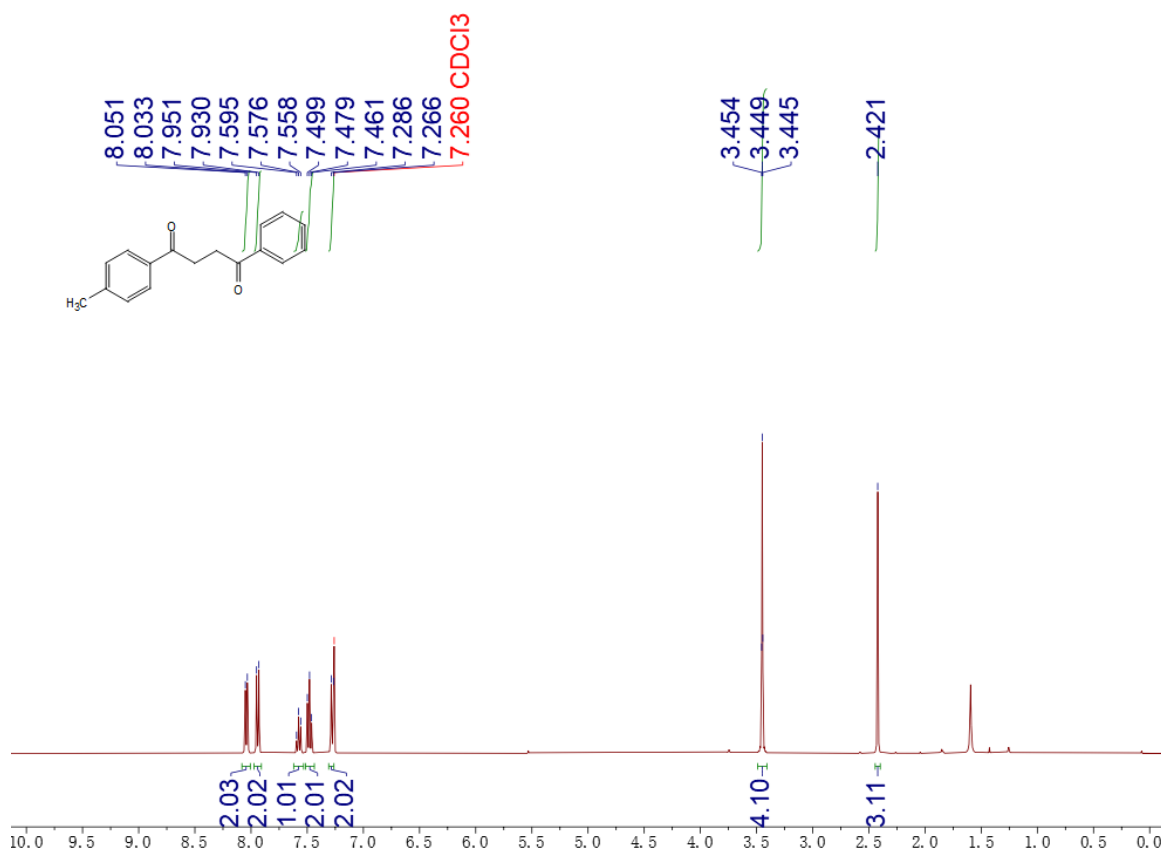
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3d (3x)**



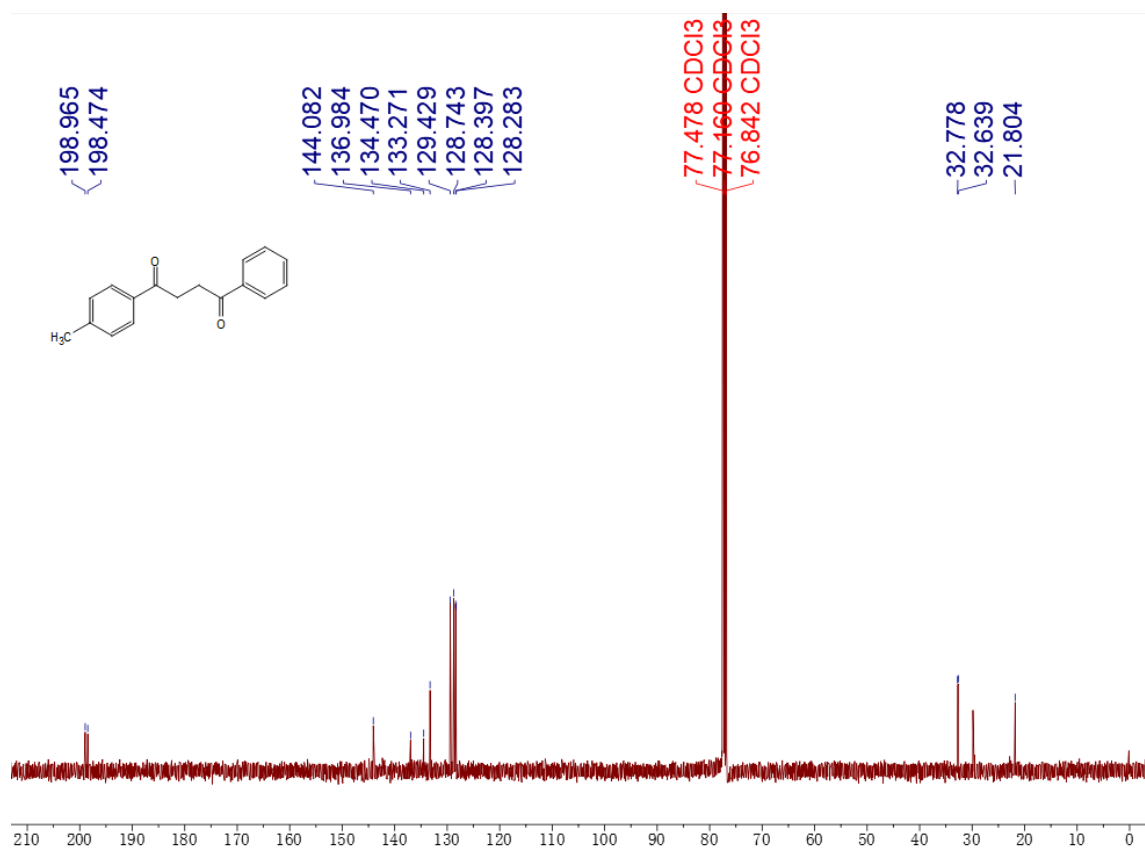
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3d (3x)**



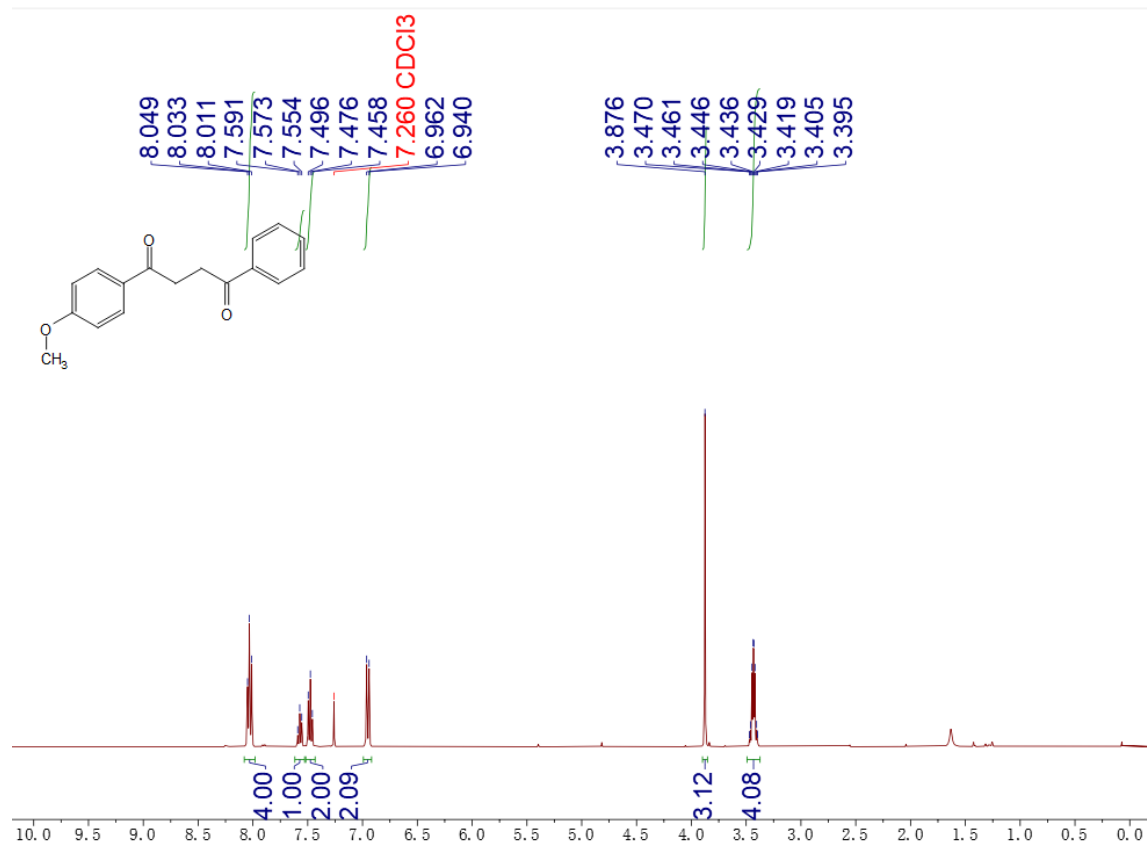
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3e (3y)**



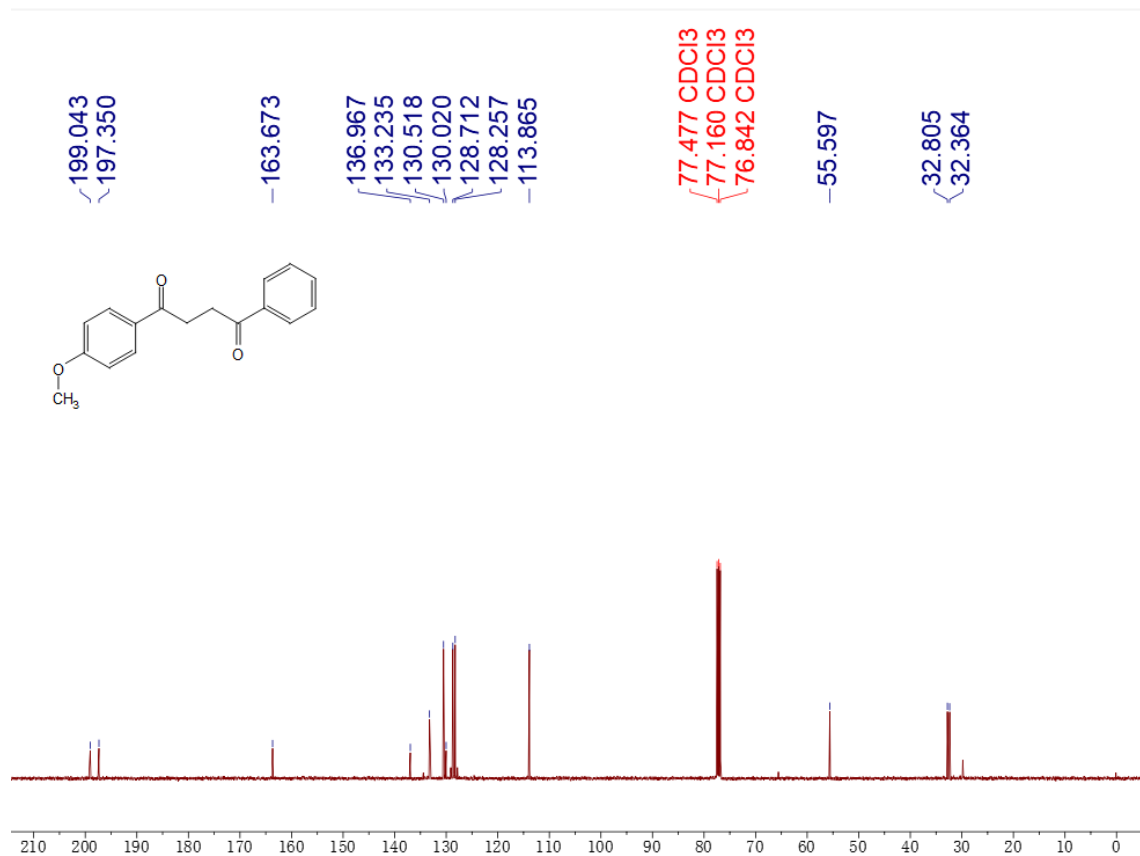
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3e (3y)**



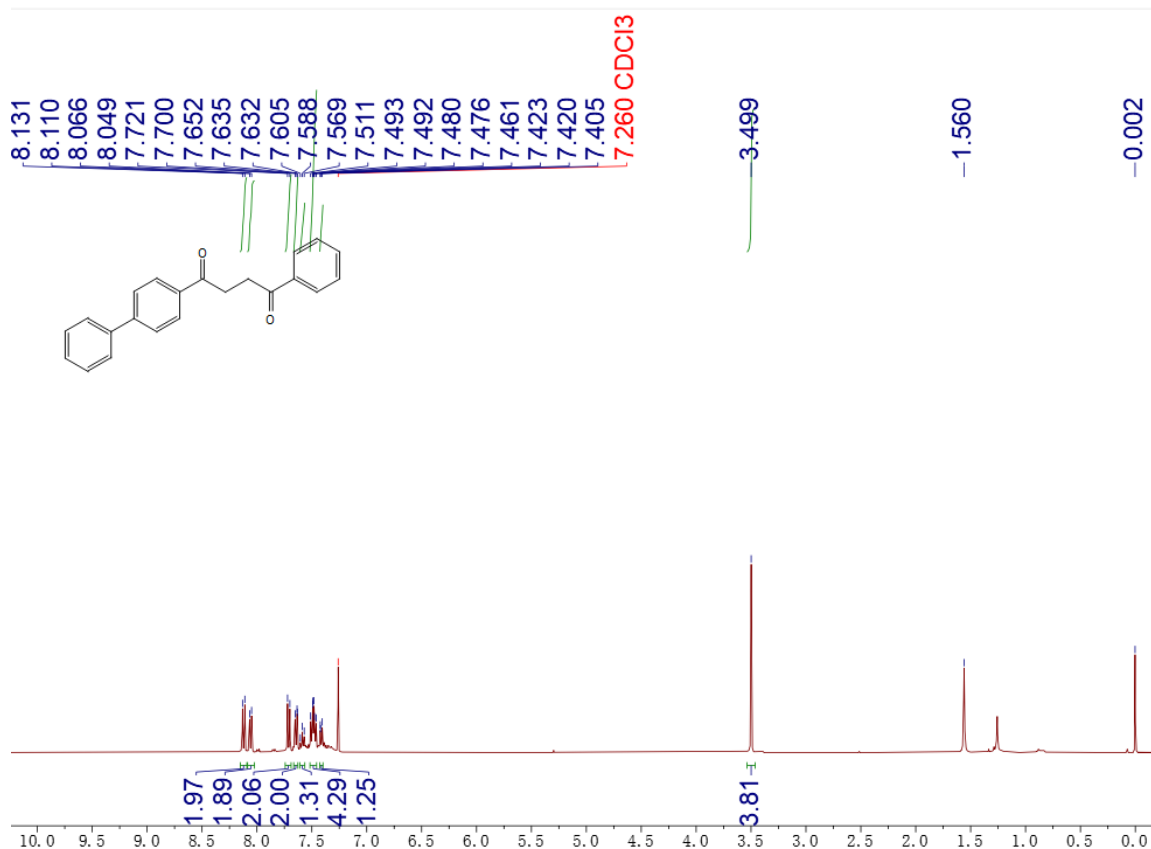
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3f** (**3s**)



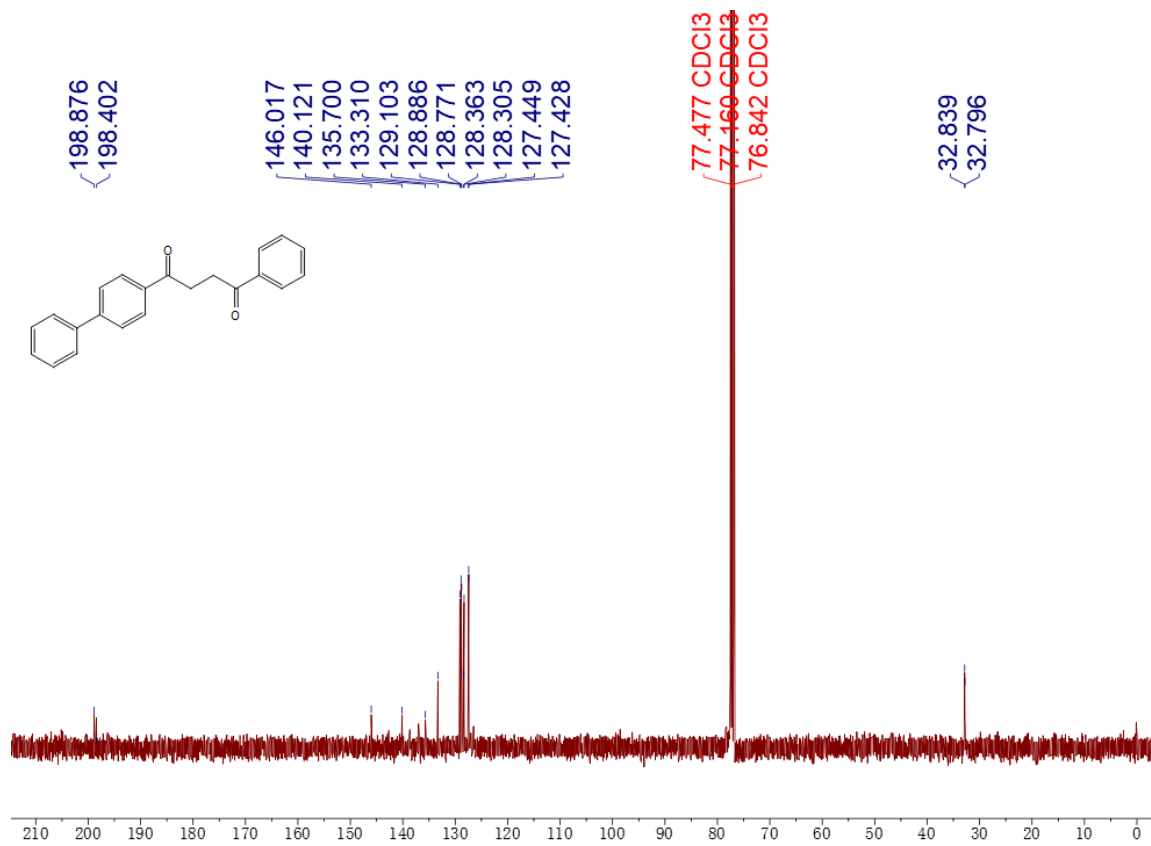
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3f** (**3s**)



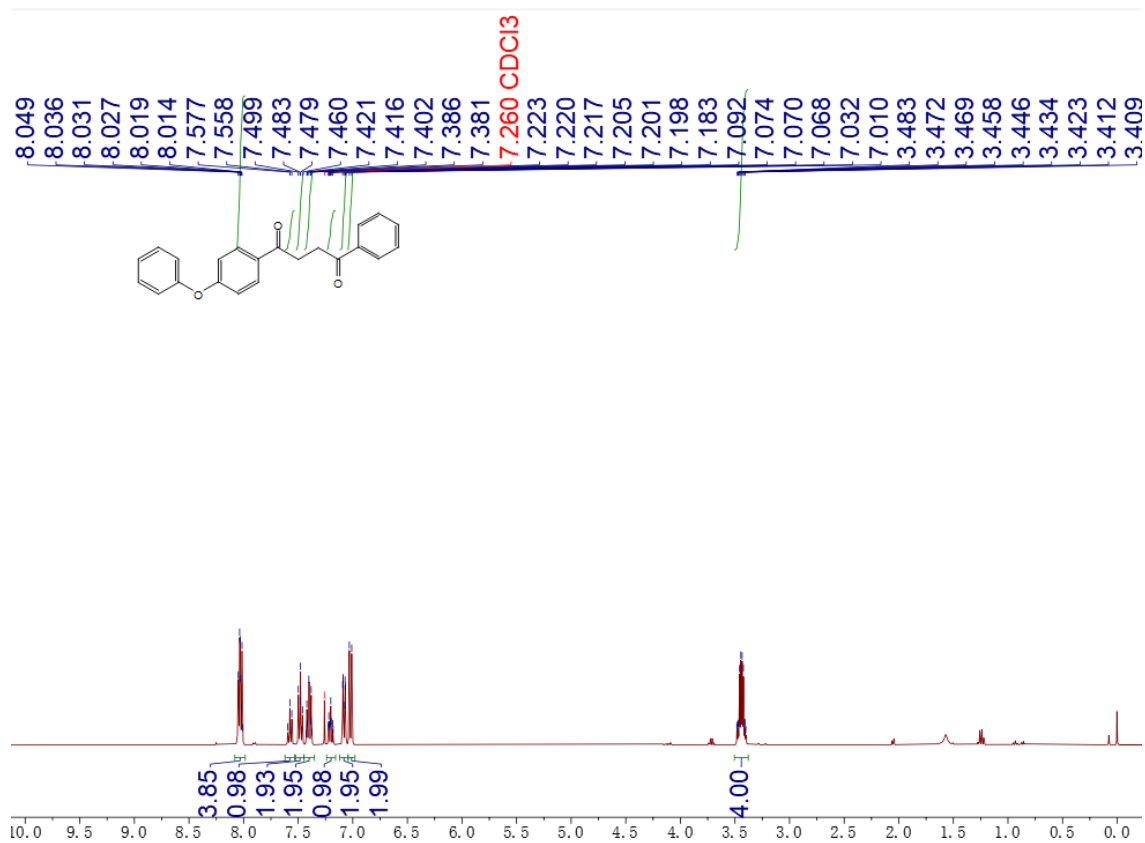
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3g (3z)**



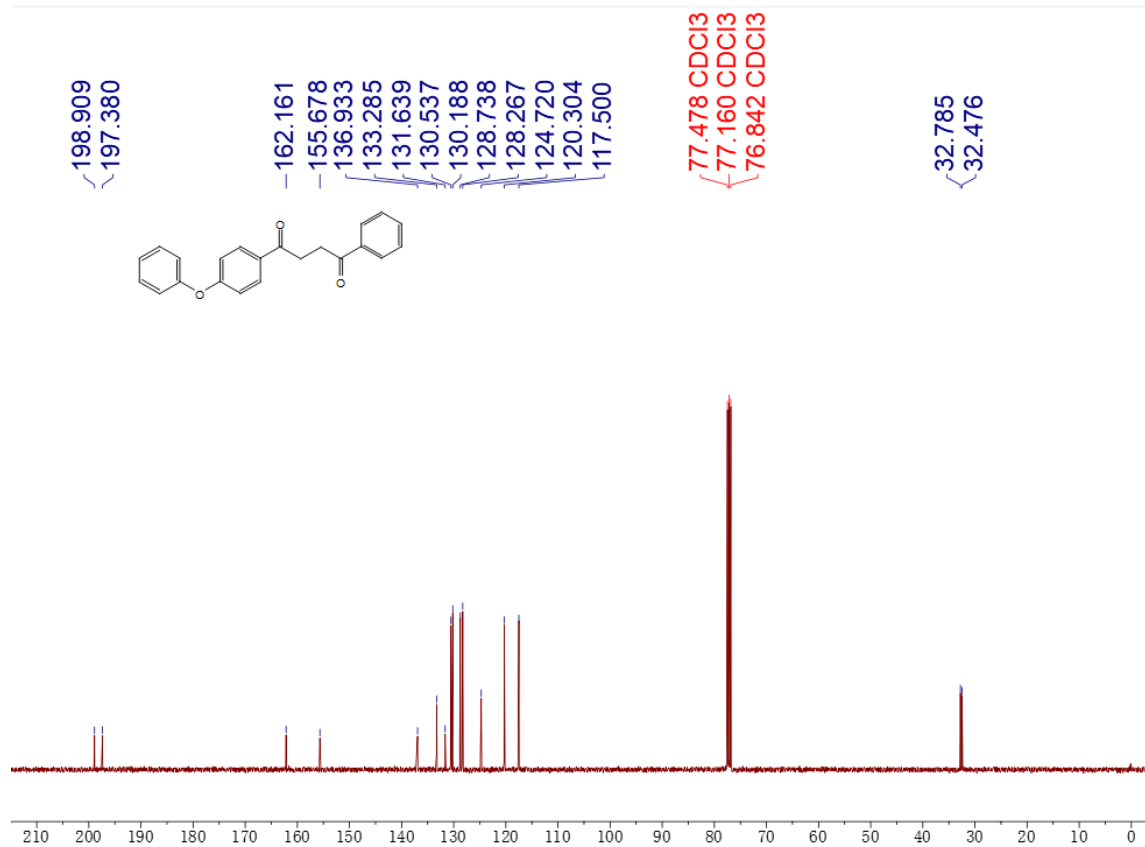
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3g (3z)**



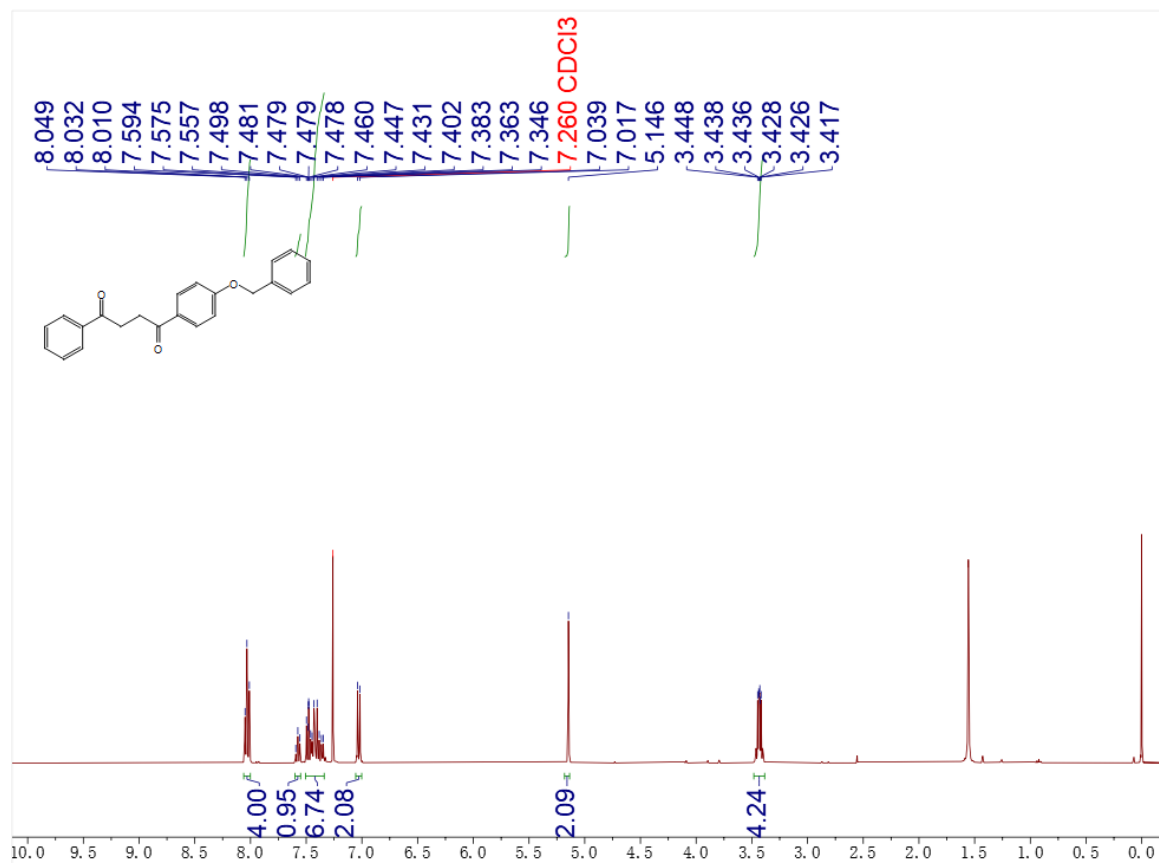
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3h (3aa)**



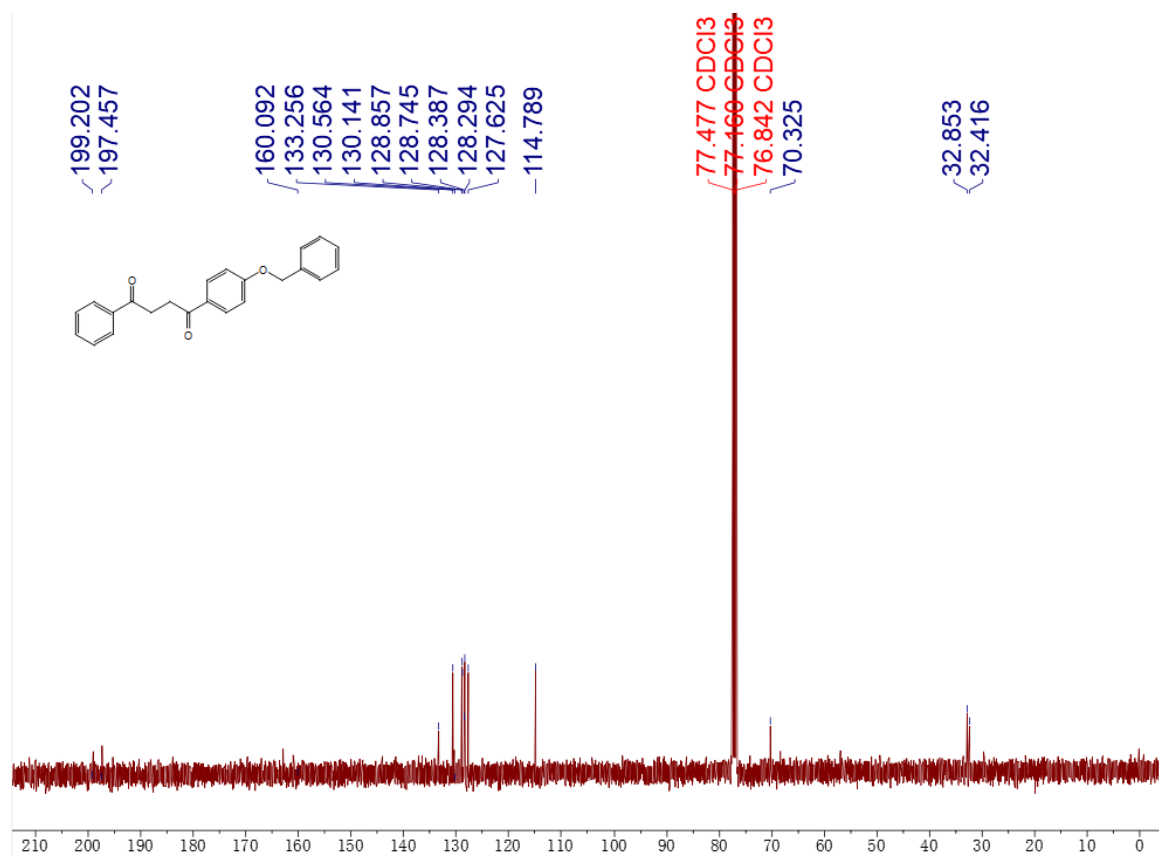
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3h (3aa)**



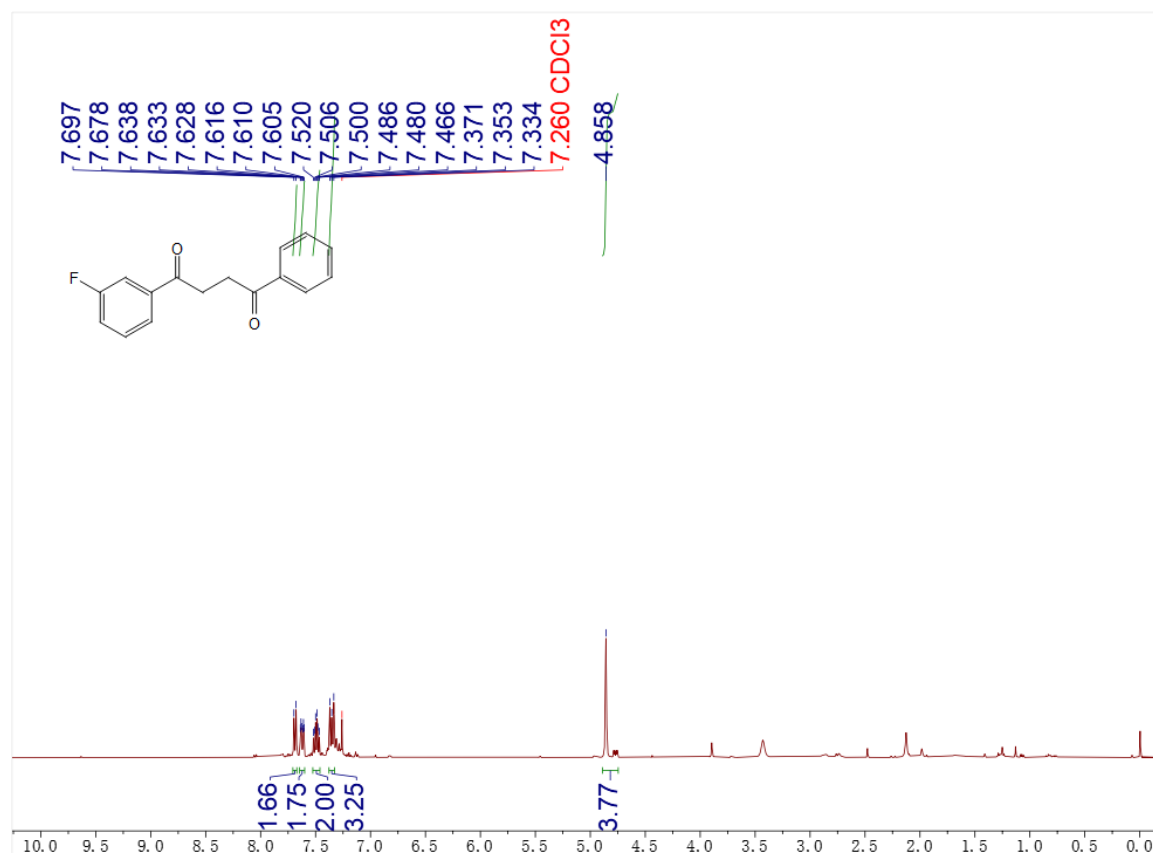
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3i**



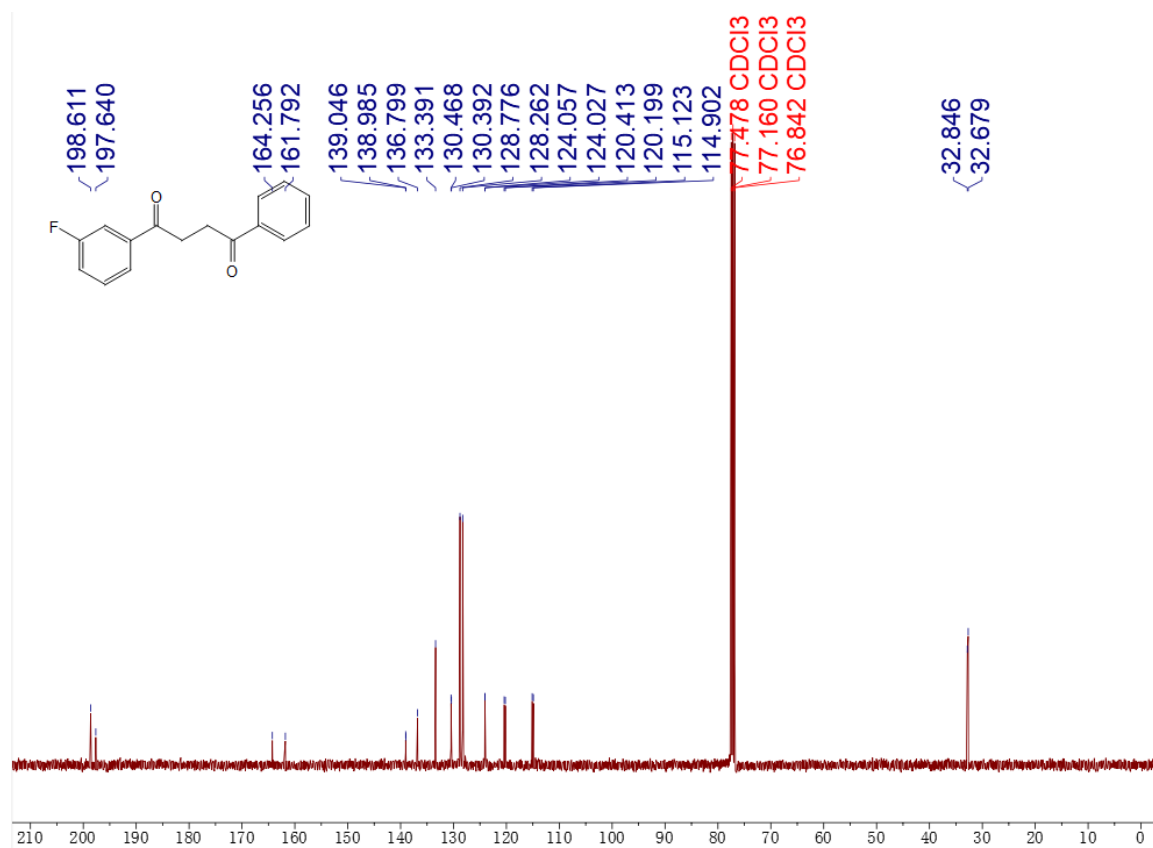
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3i**



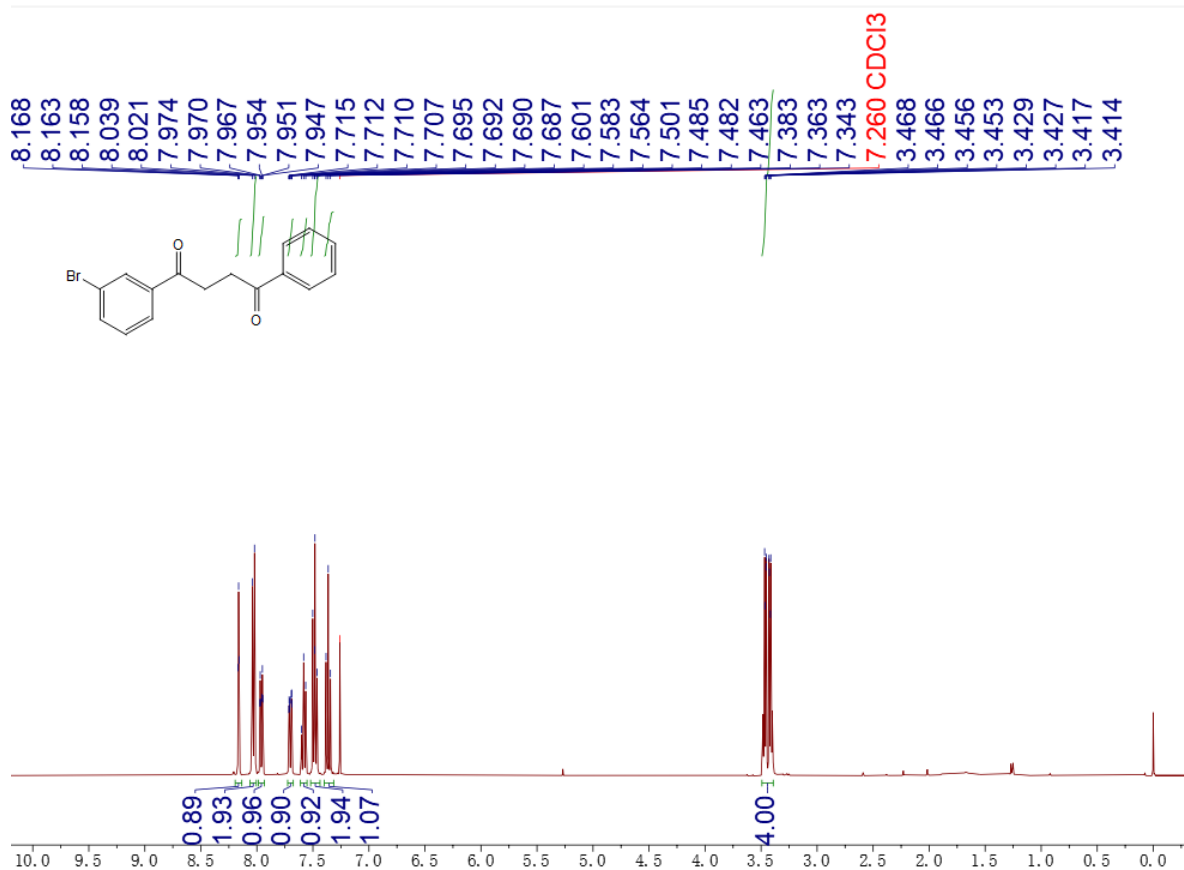
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3j** (**3ae**)



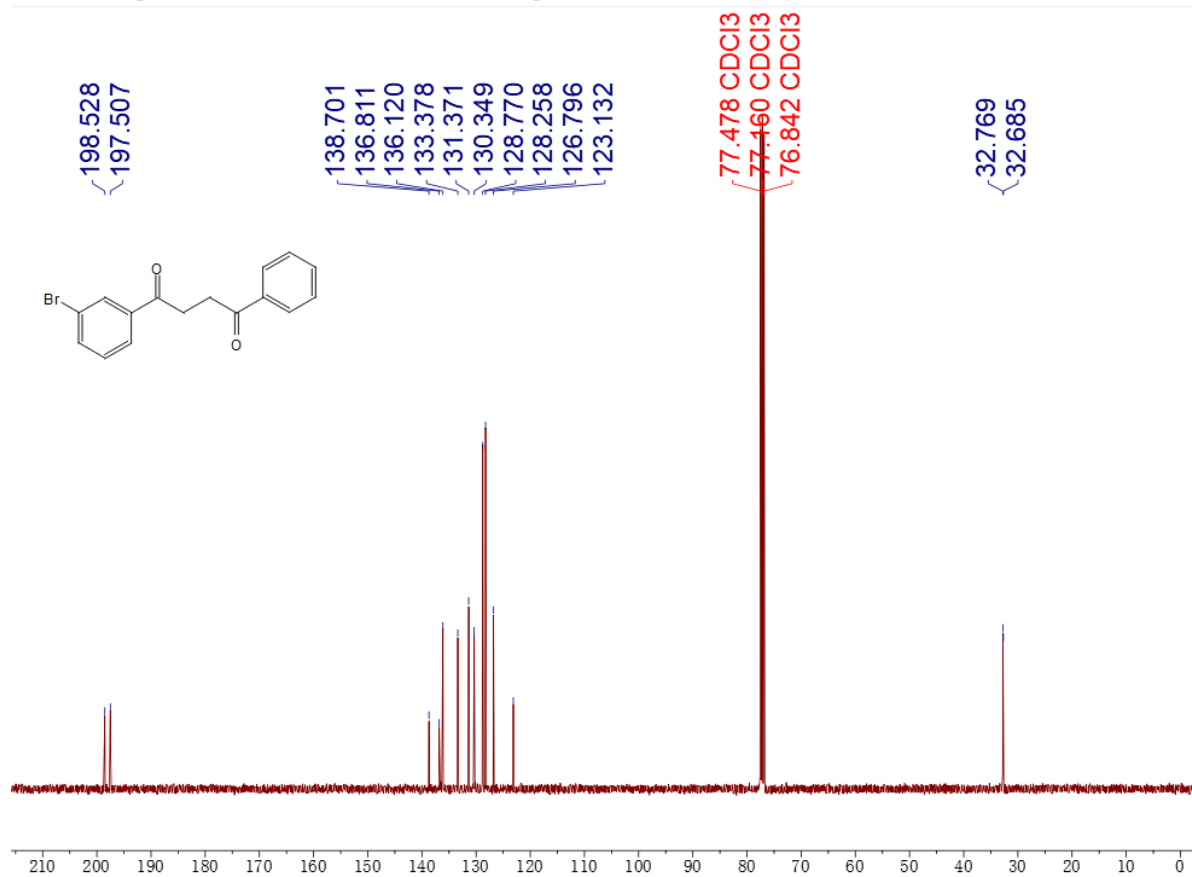
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3j** (**3ae**)



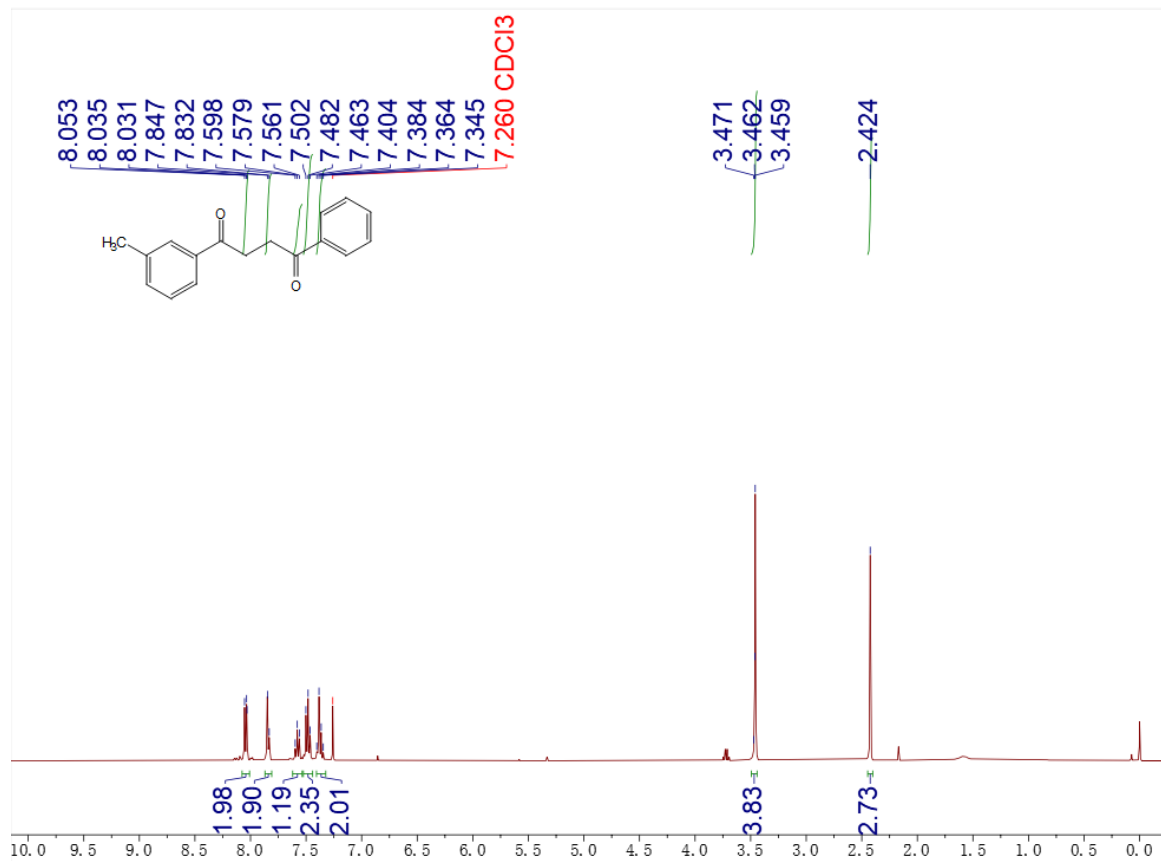
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3k**



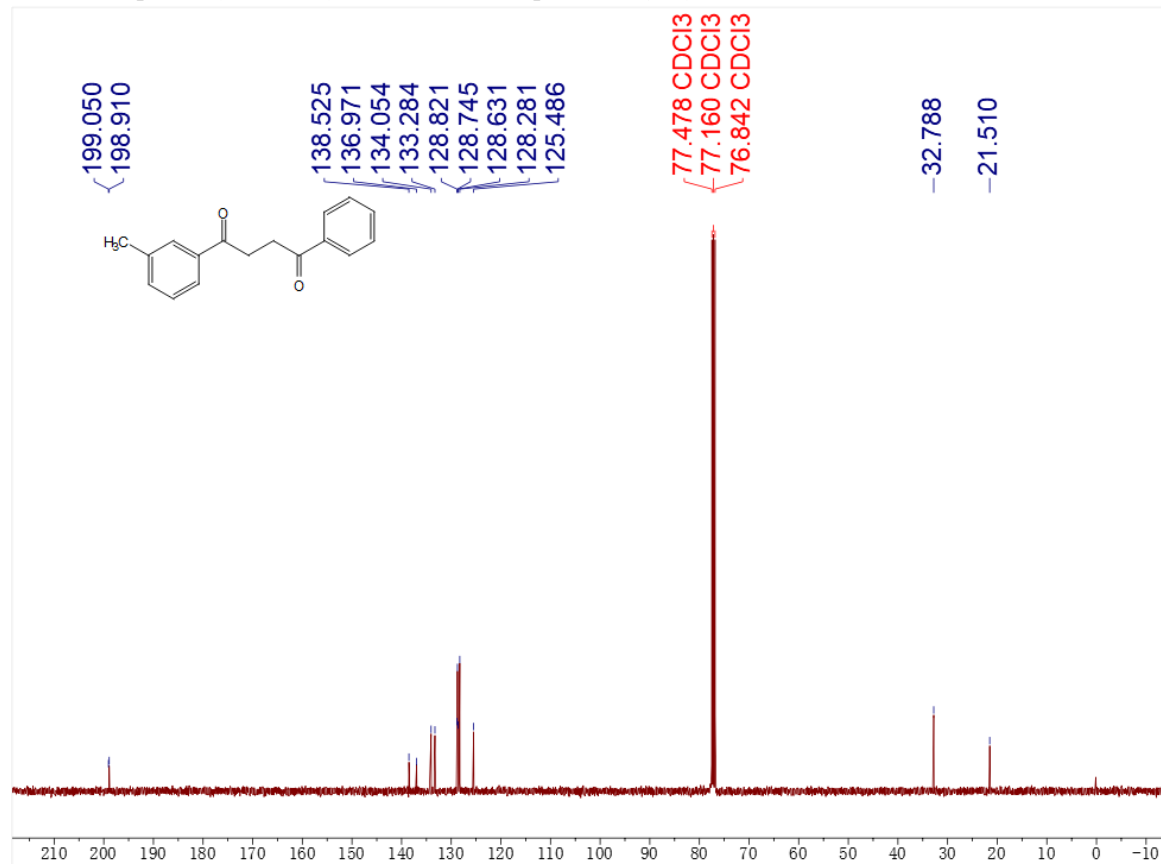
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3k**



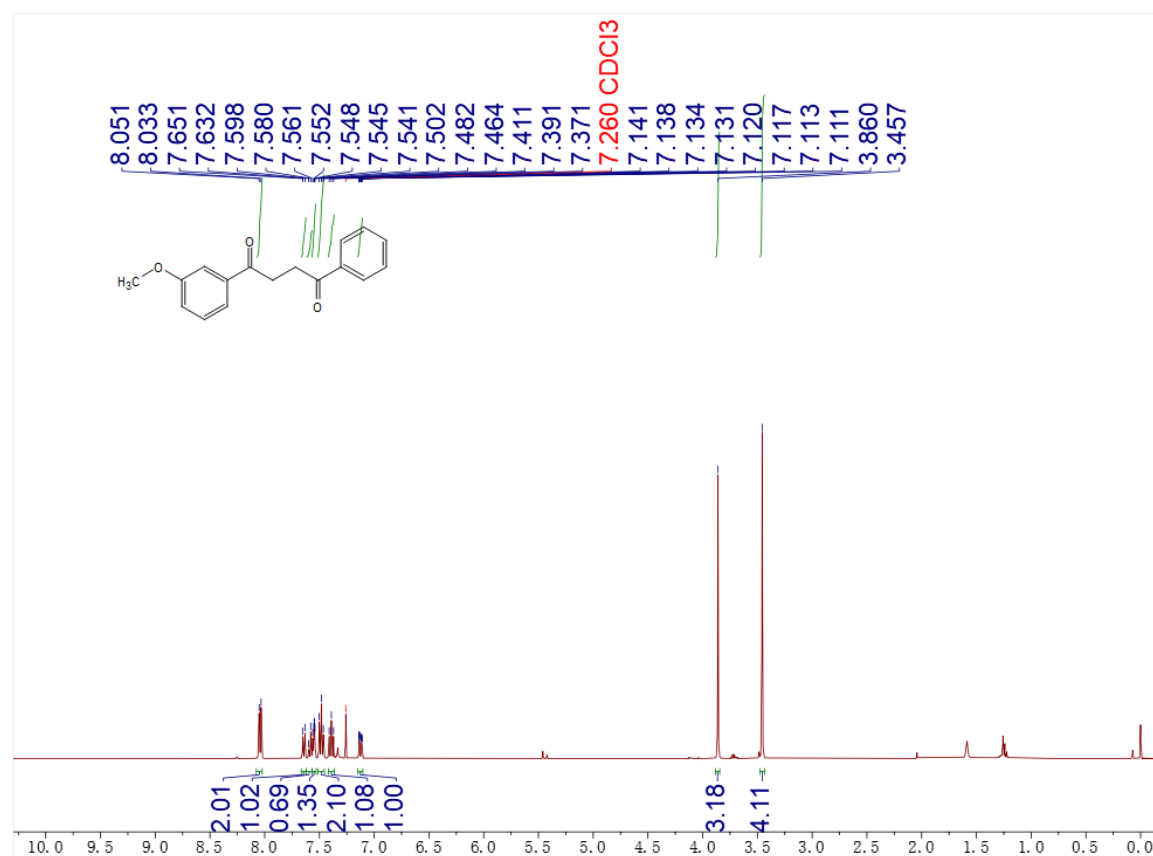
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3l (3af)**



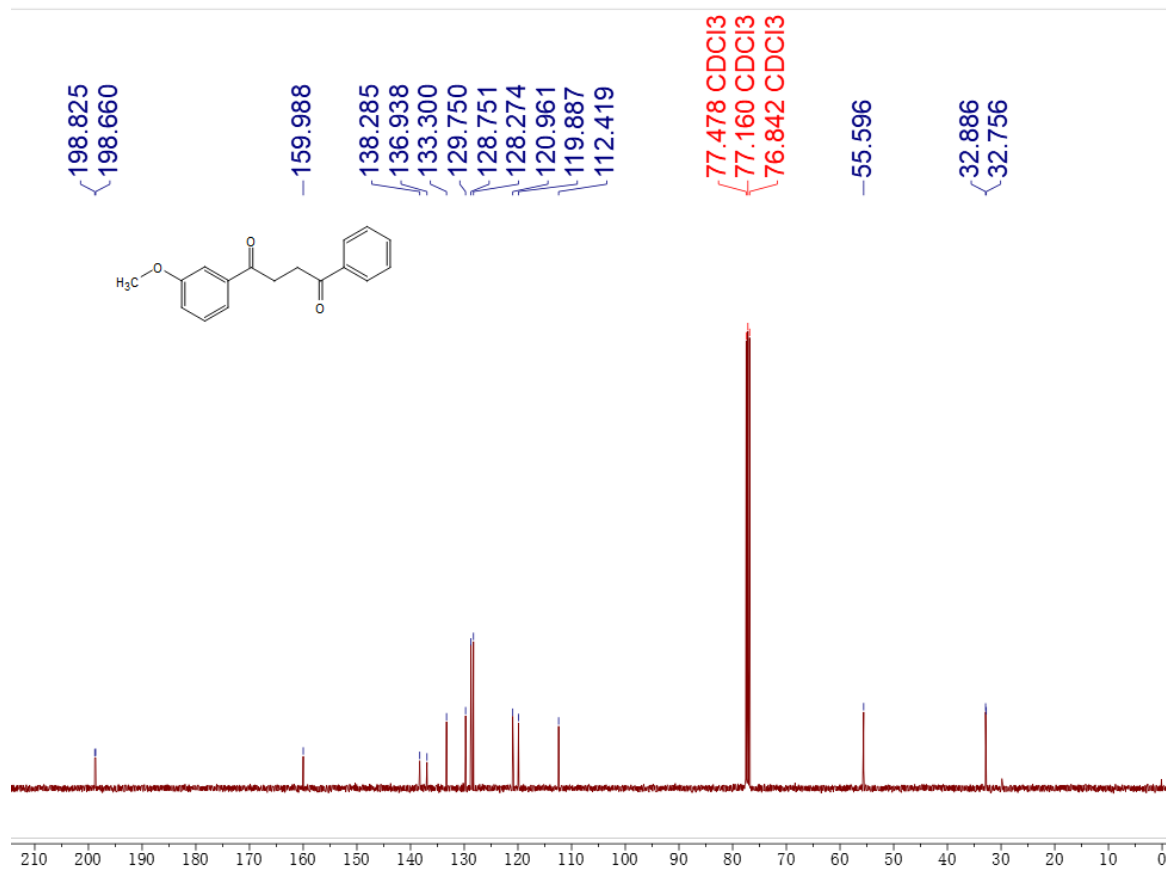
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3l (3af)**



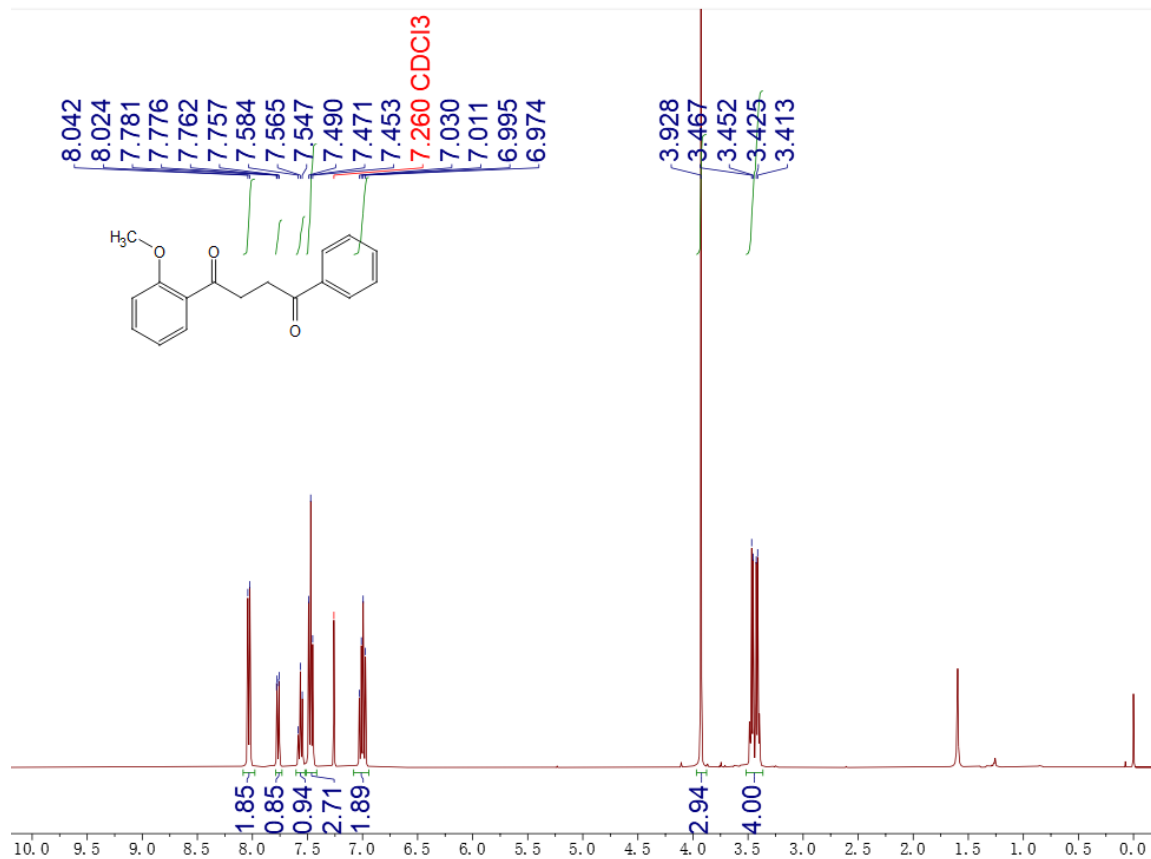
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3m**



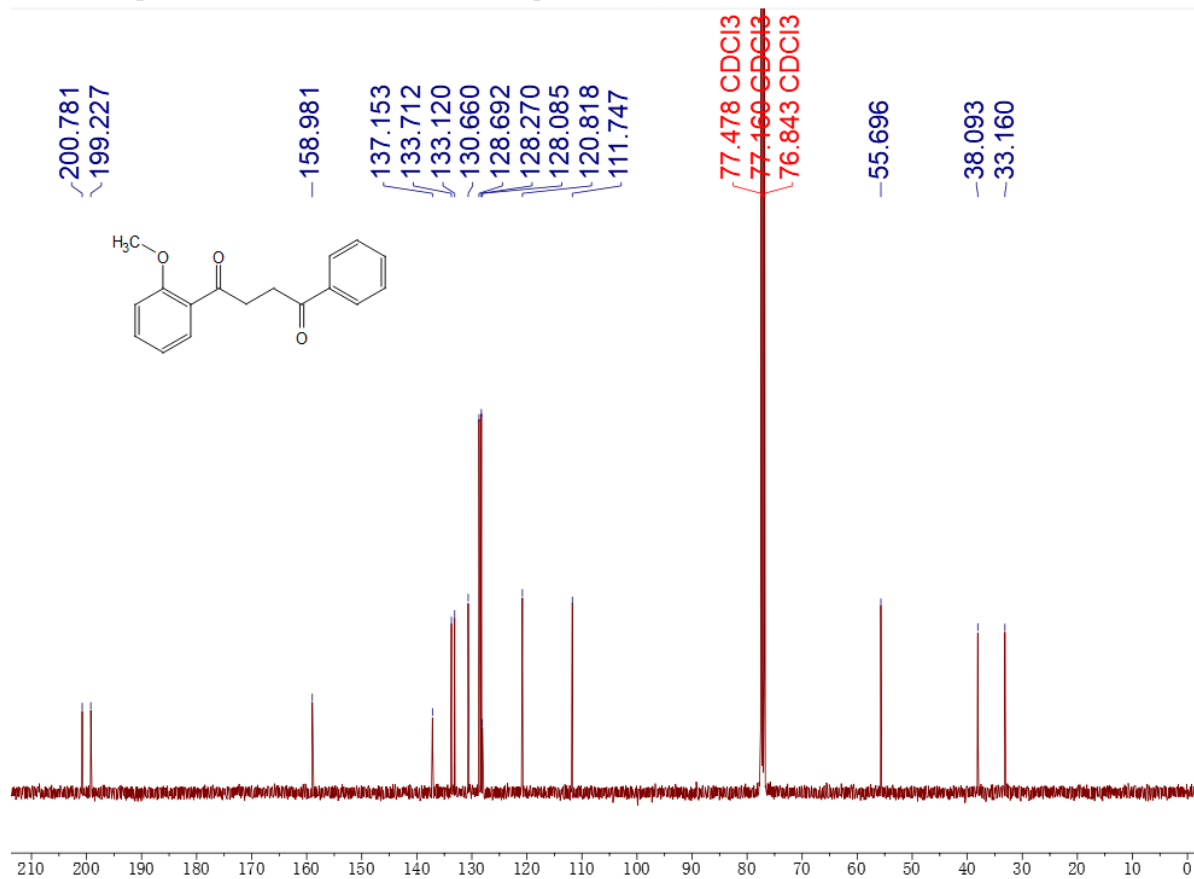
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3m**



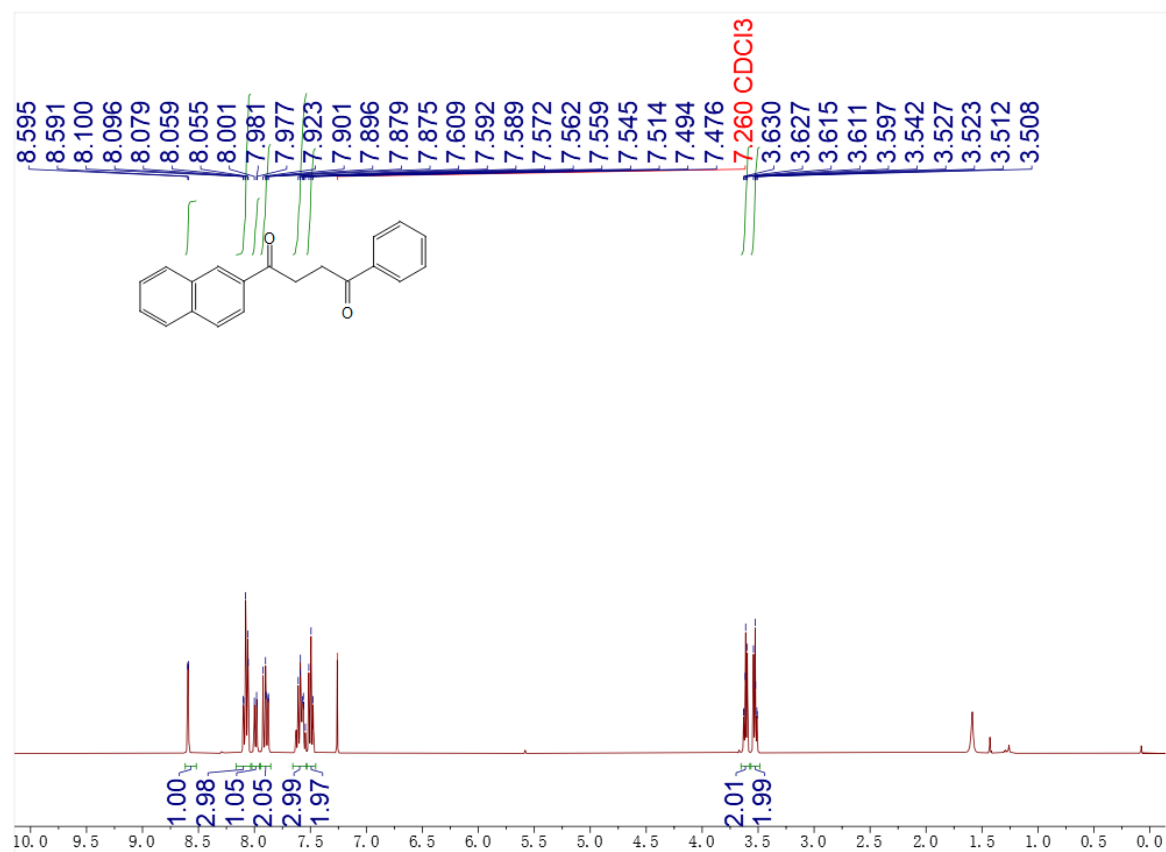
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3n (3ab)**



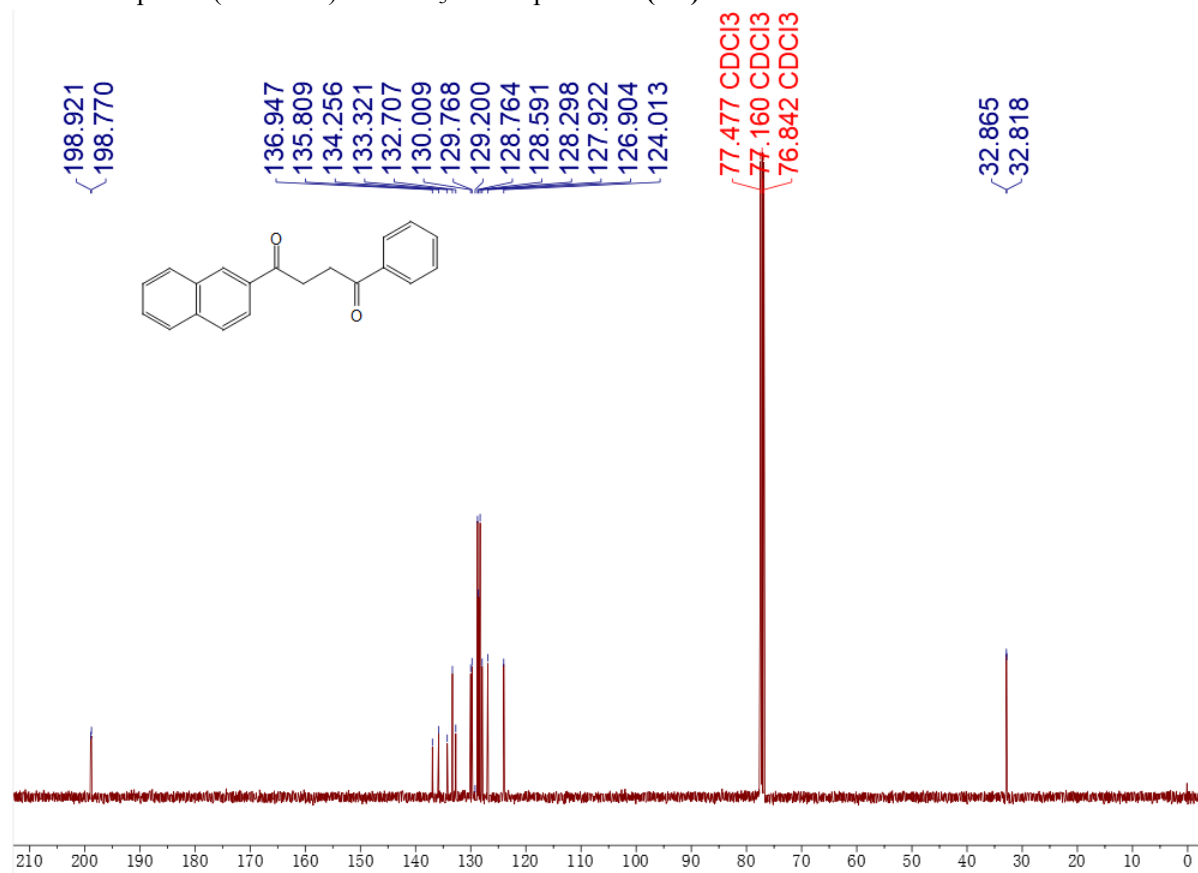
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3n (3ab)**



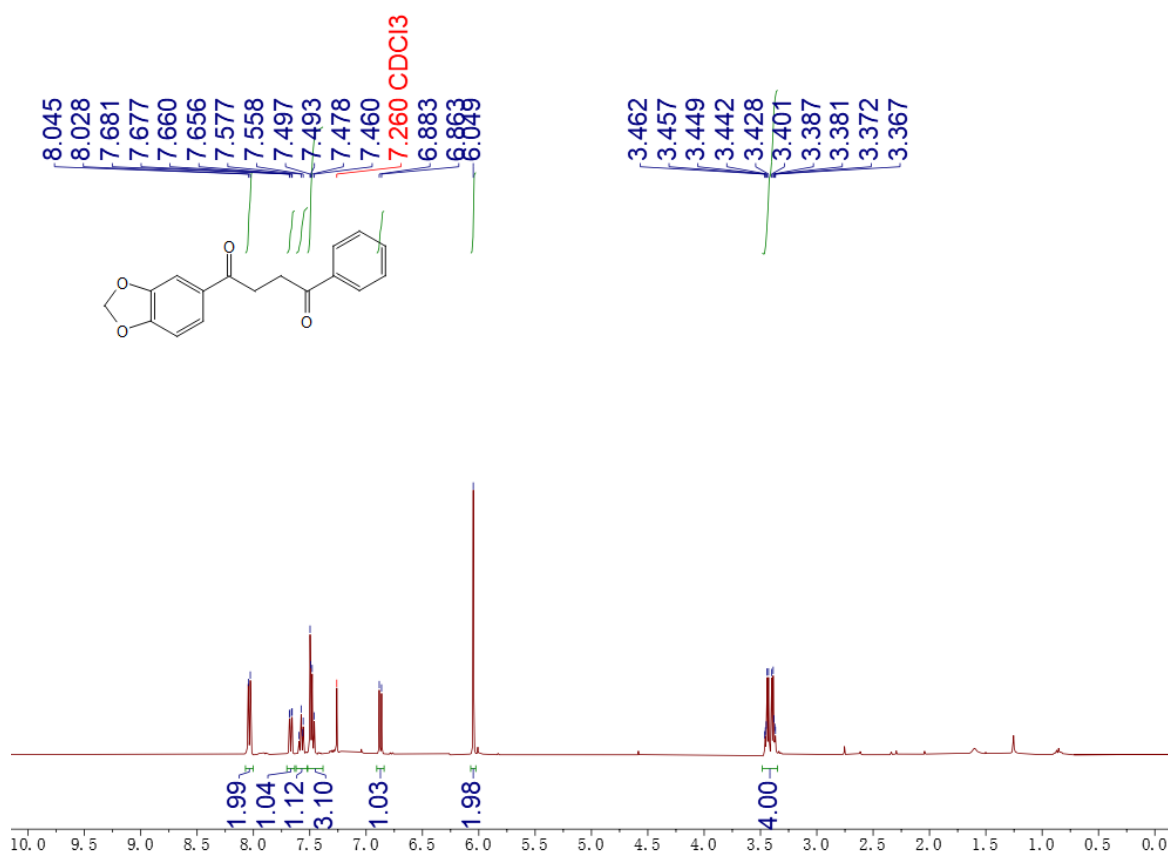
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3o** (**3ai**)



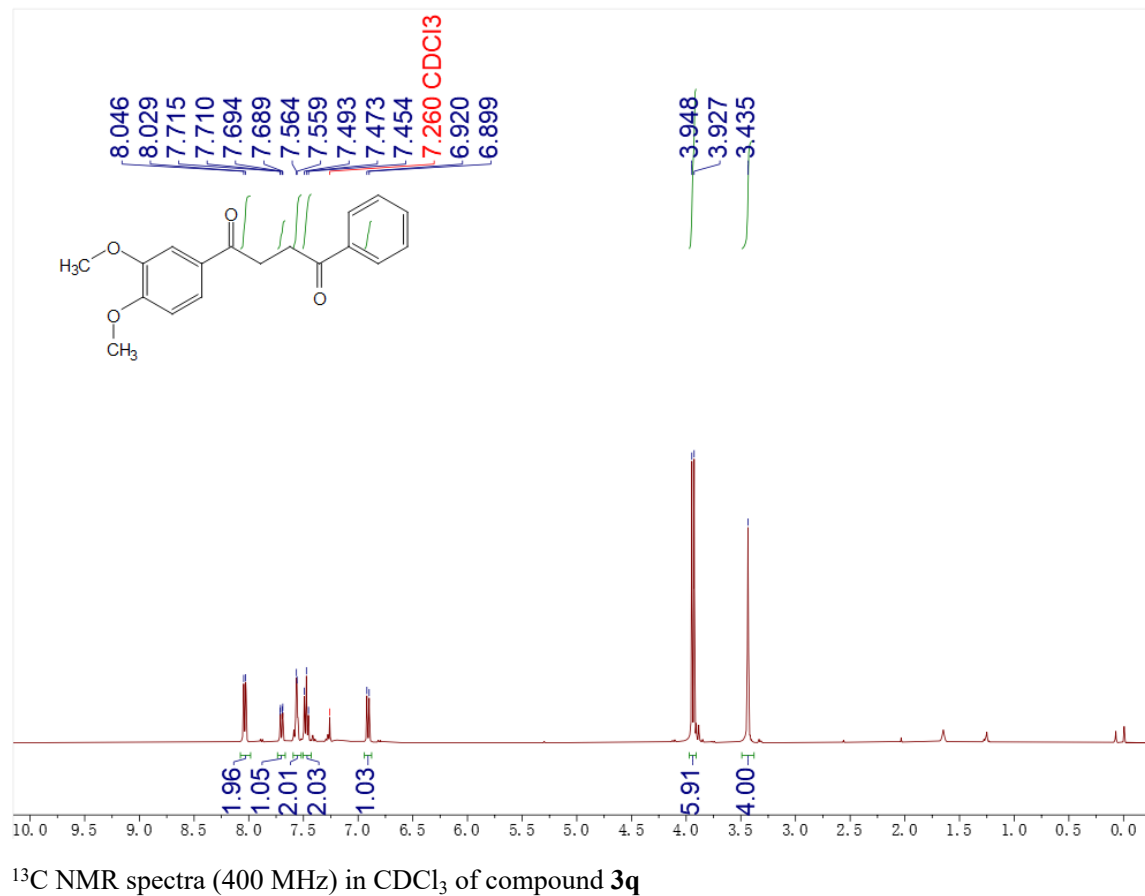
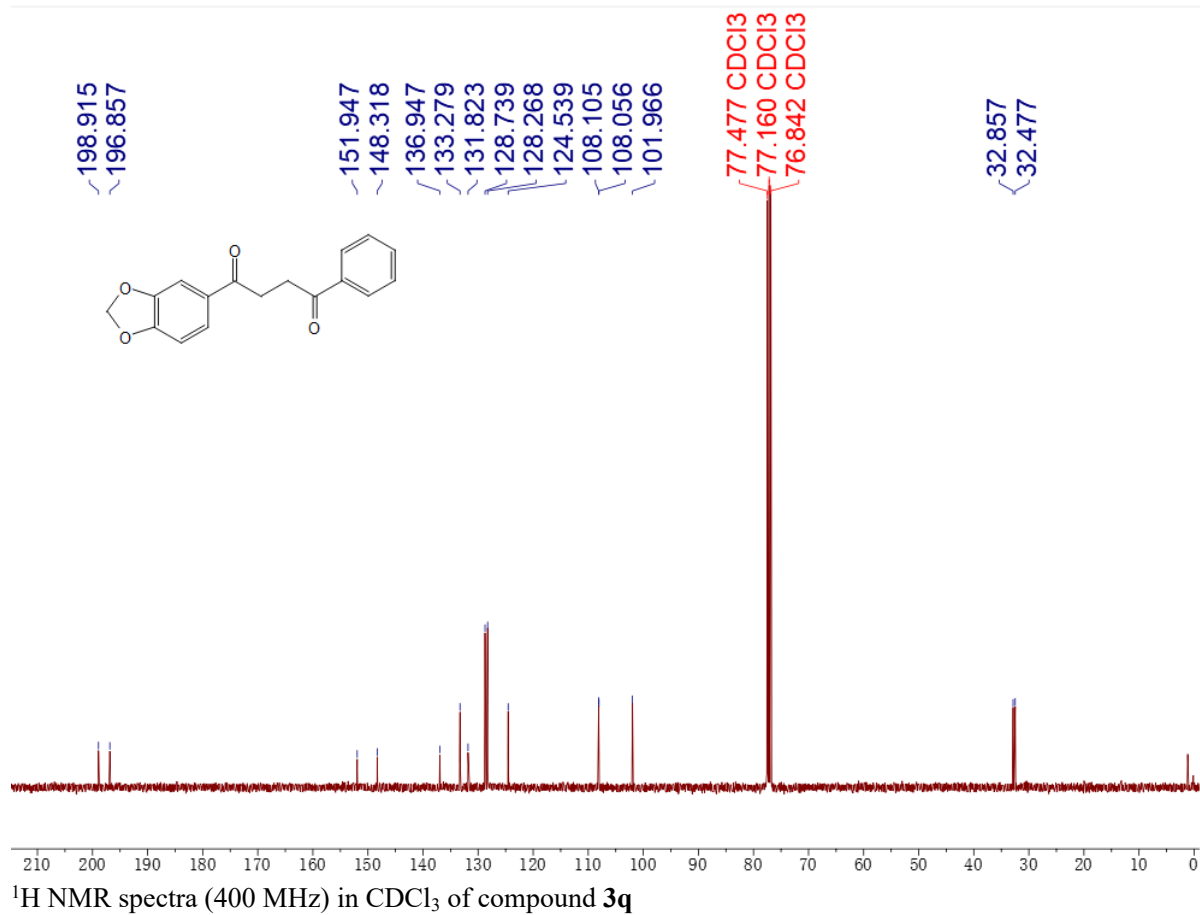
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3o** (**3ai**)

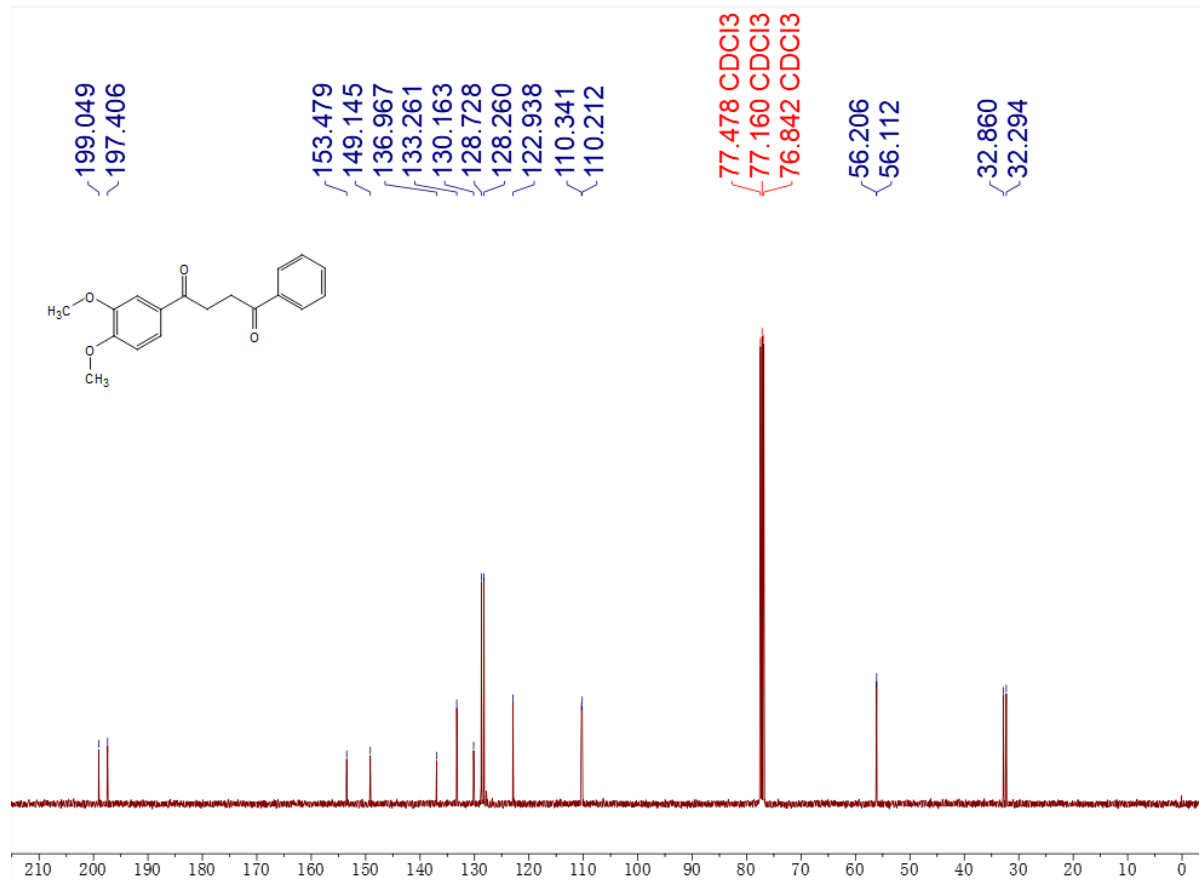


¹H NMR spectra (400 MHz) in CDCl₃ of compound **3p**

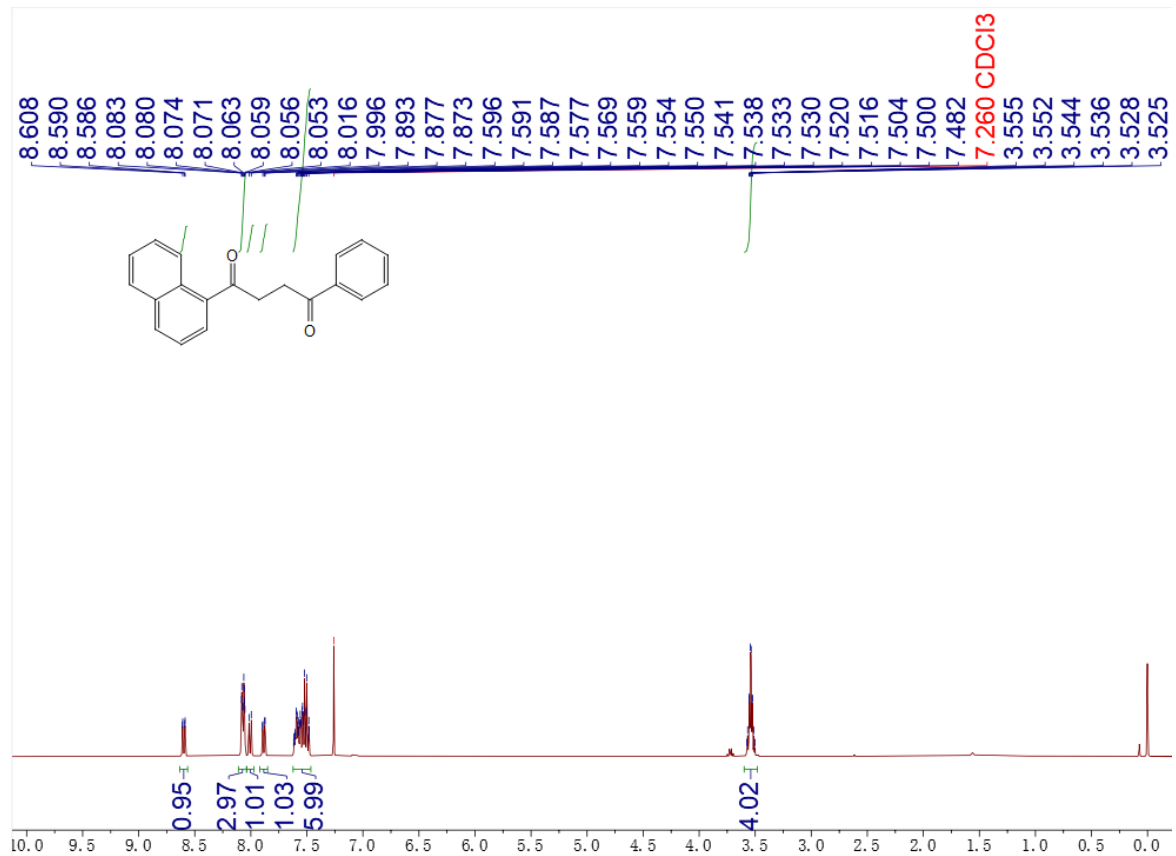


¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3p**

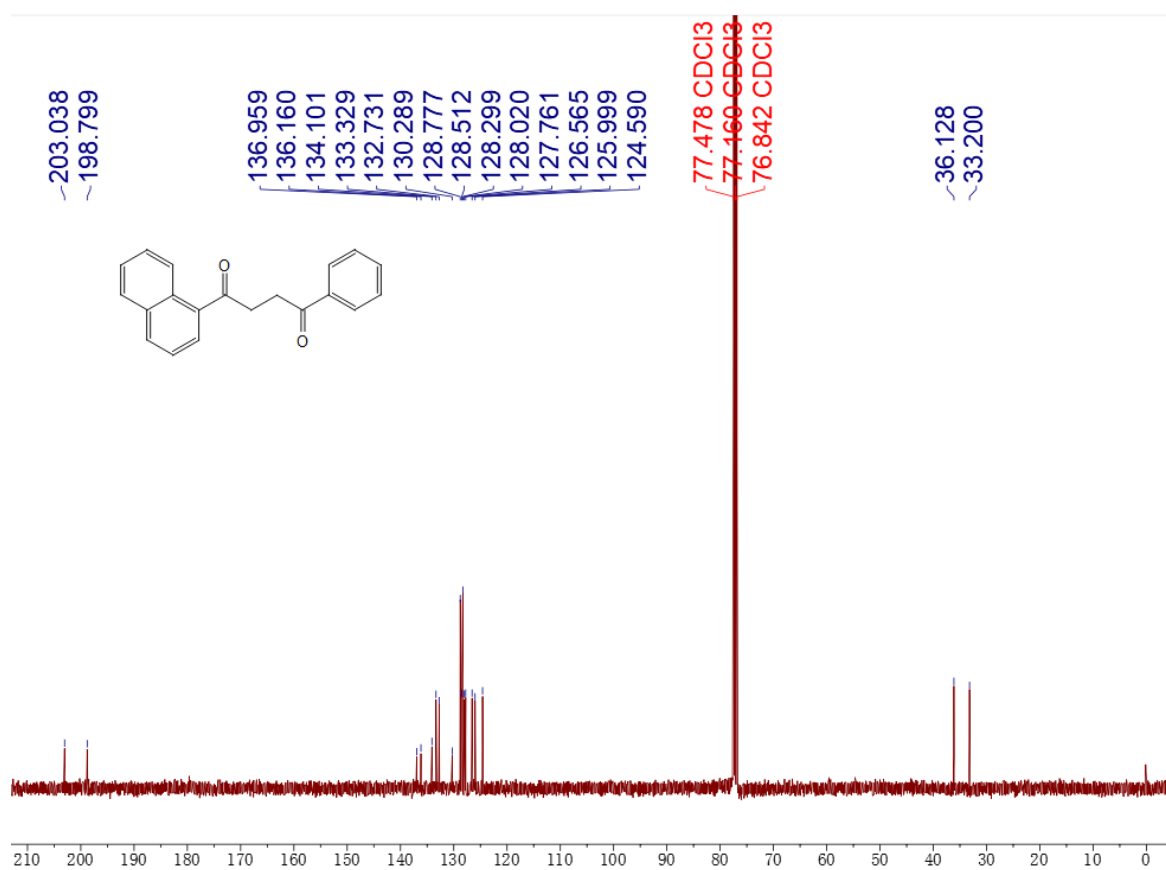




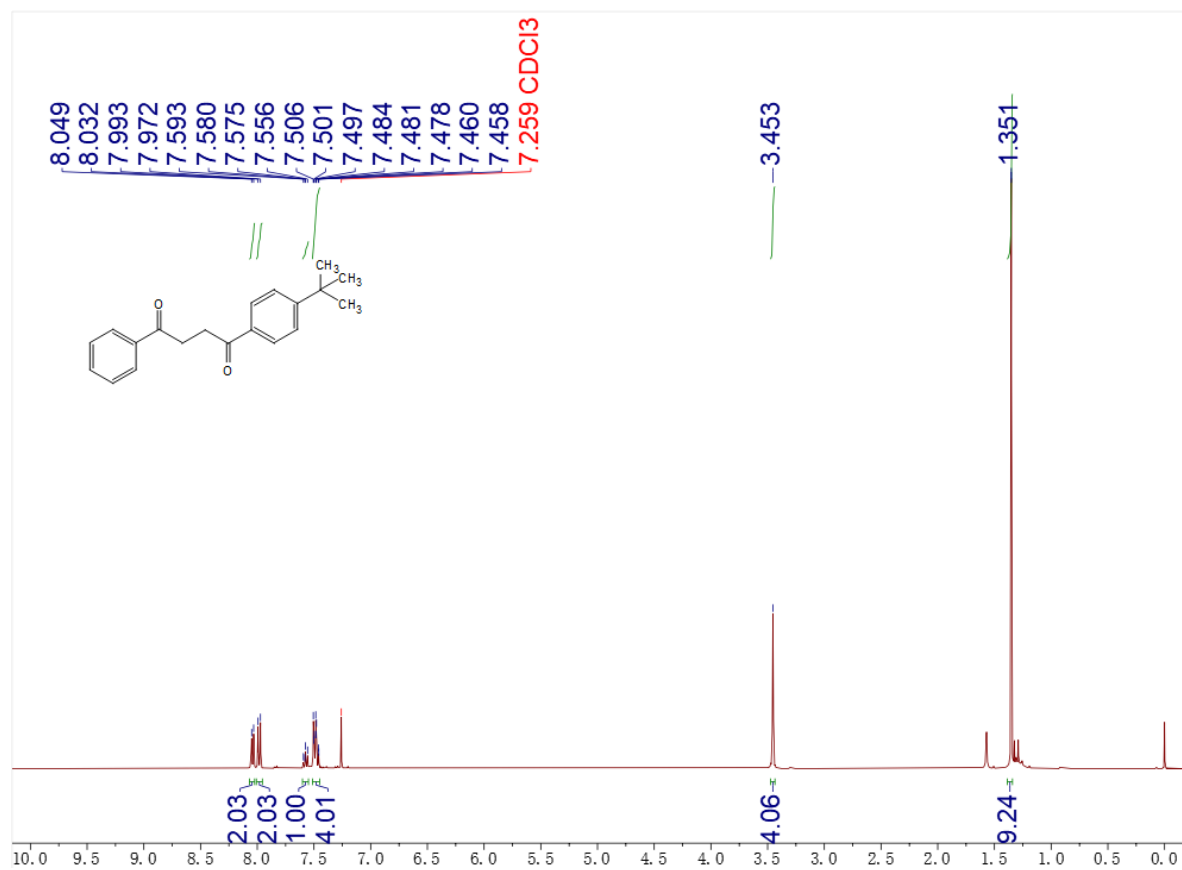
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3r**



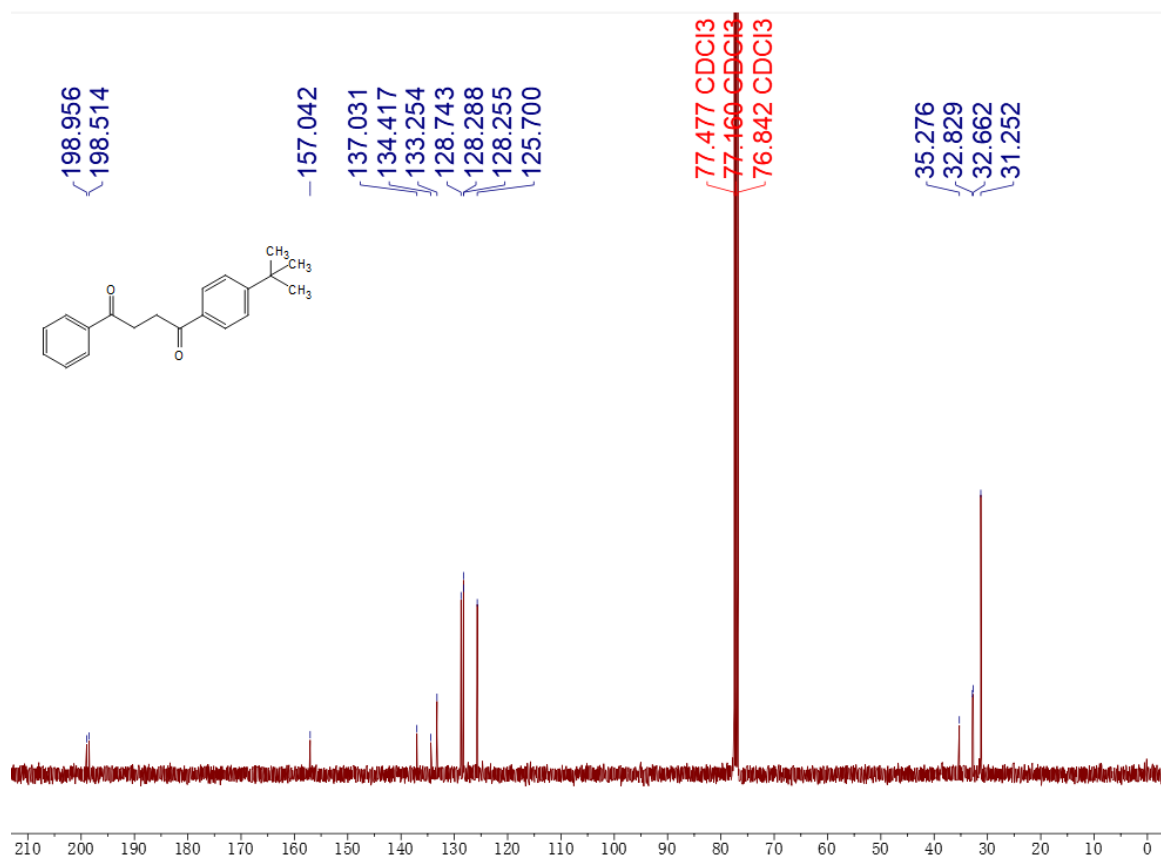
^{13}C NMR spectra (400 MHz) in CDCl_3 of compound **3r**



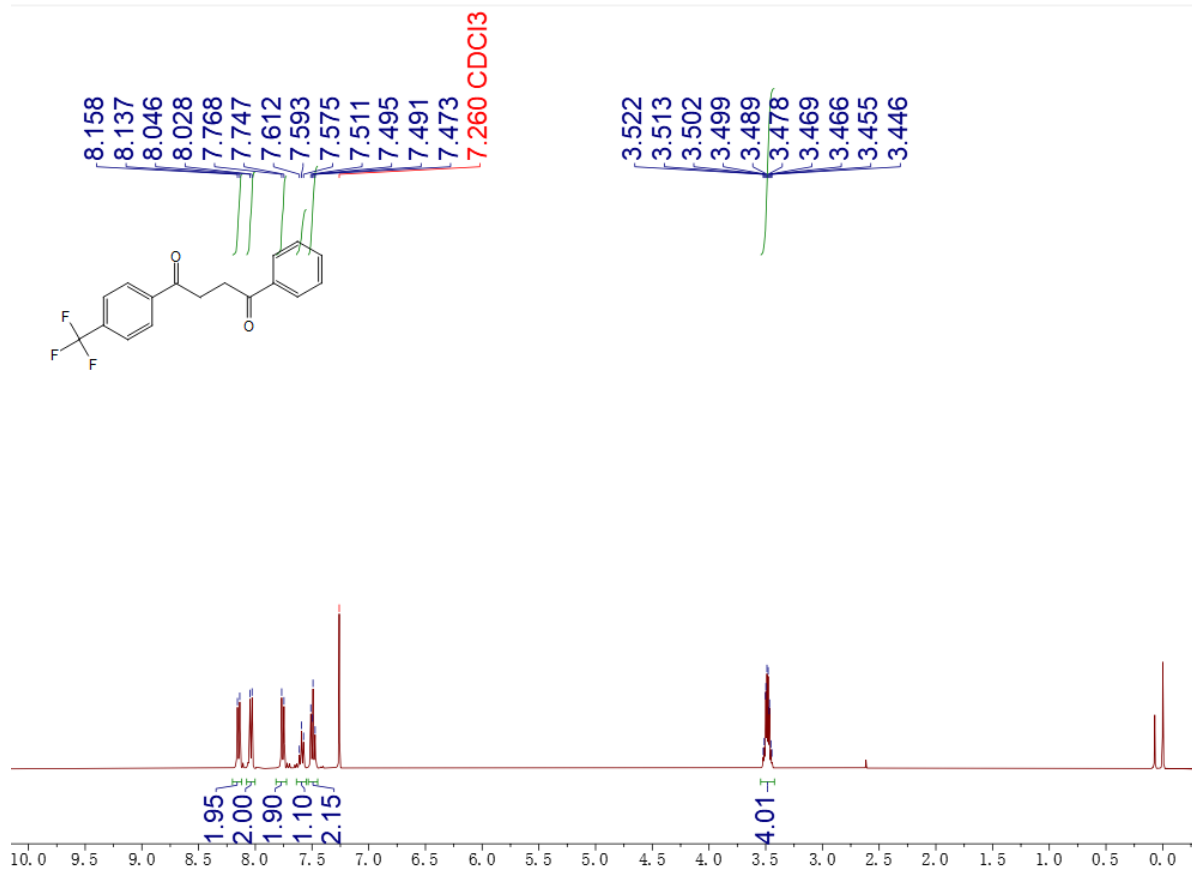
^1H NMR spectra (400 MHz) in CDCl_3 of compound **3t**



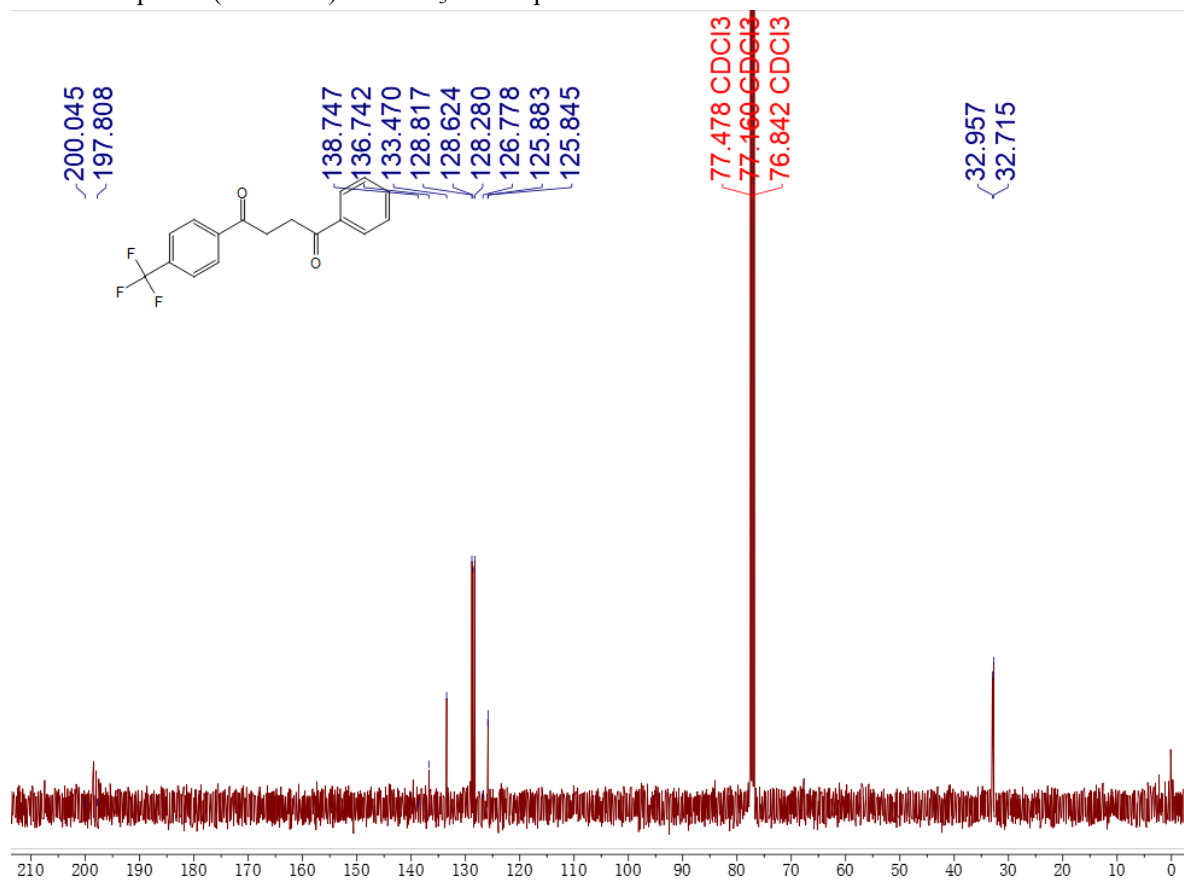
^{13}C NMR spectra (400 MHz) in CDCl_3 of compound **3t**



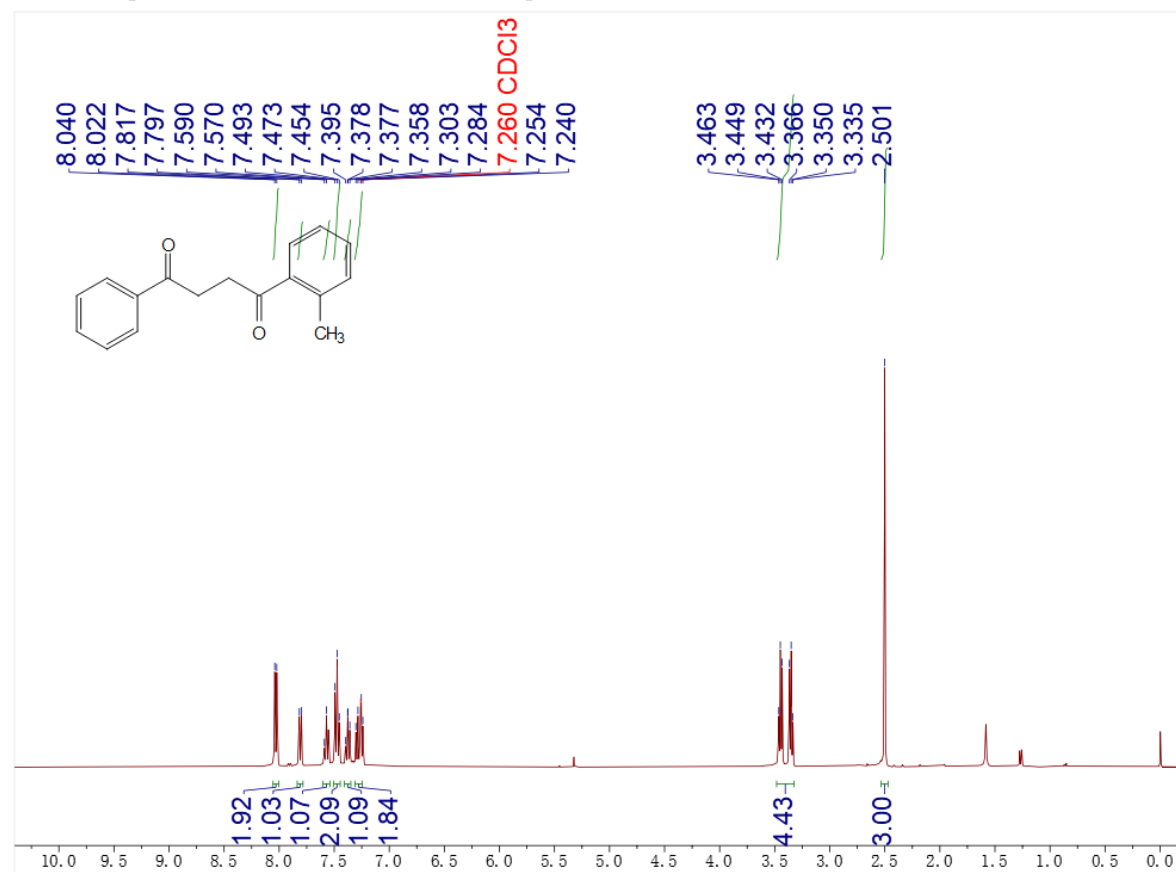
^1H NMR spectra (400 MHz) in CDCl_3 of compound **3u**



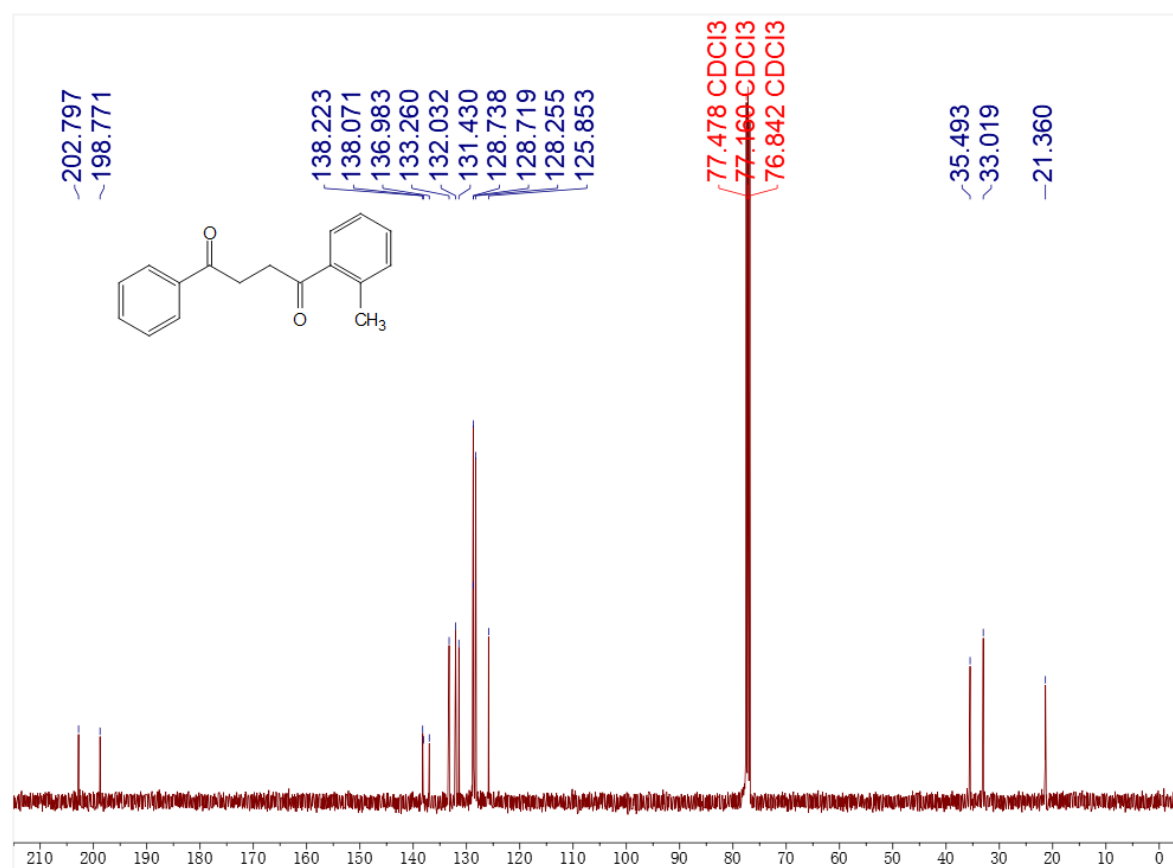
^{13}C NMR spectra (400 MHz) in CDCl_3 of compound **3u**



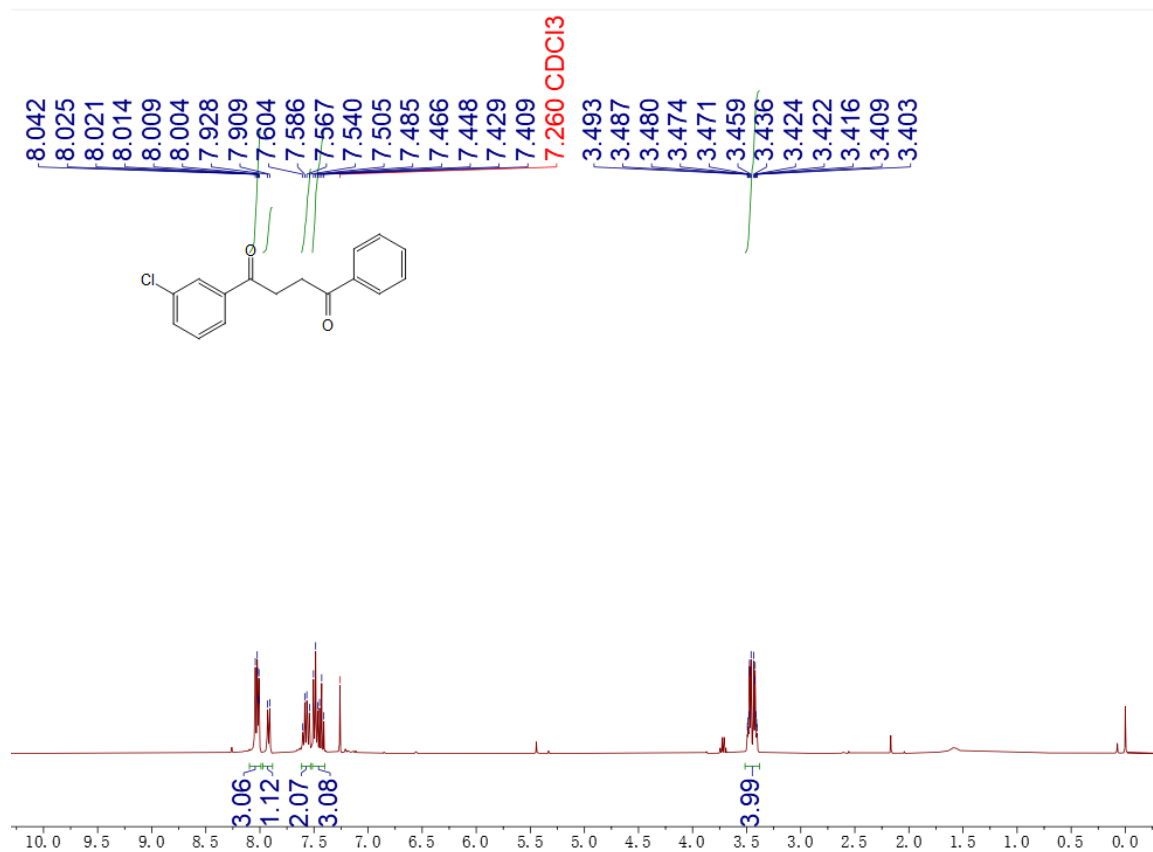
^1H NMR spectra (400 MHz) in CDCl_3 of compound **3ac**



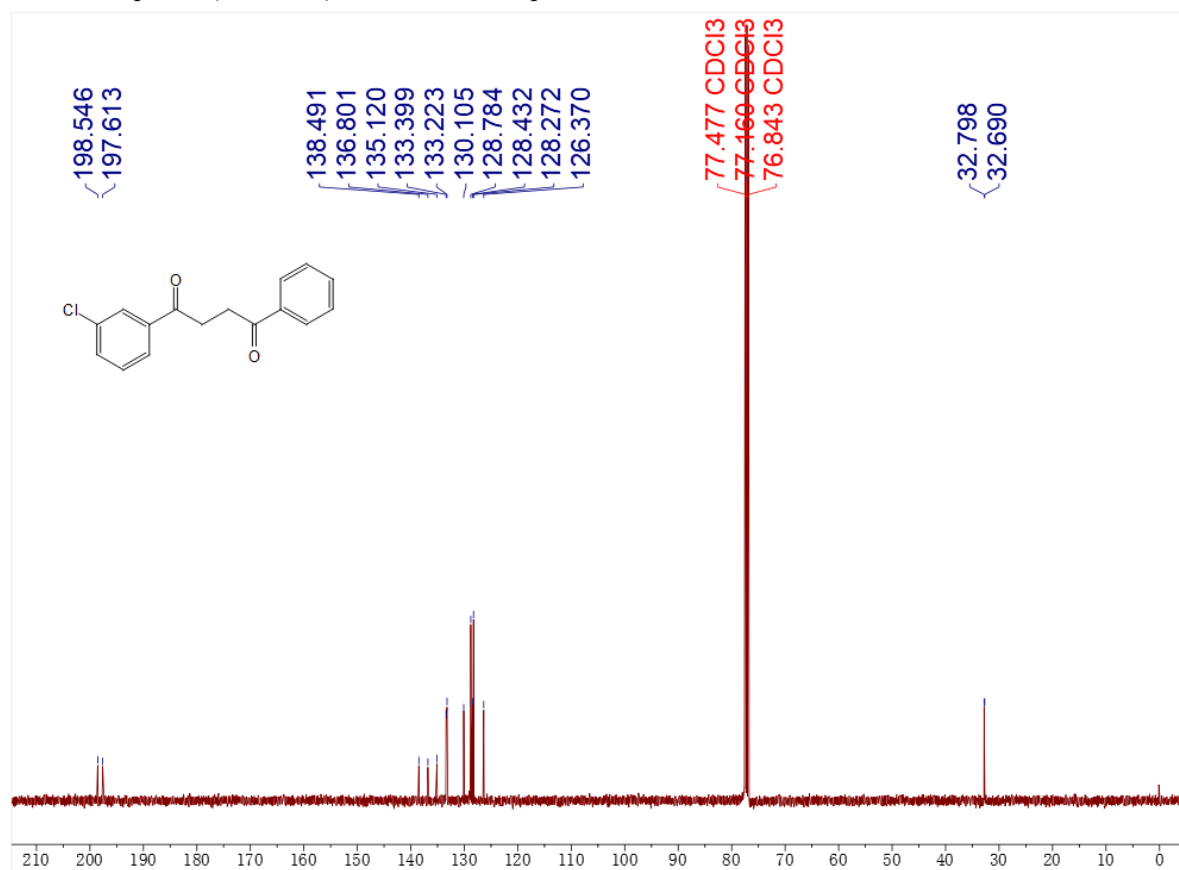
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3ac**



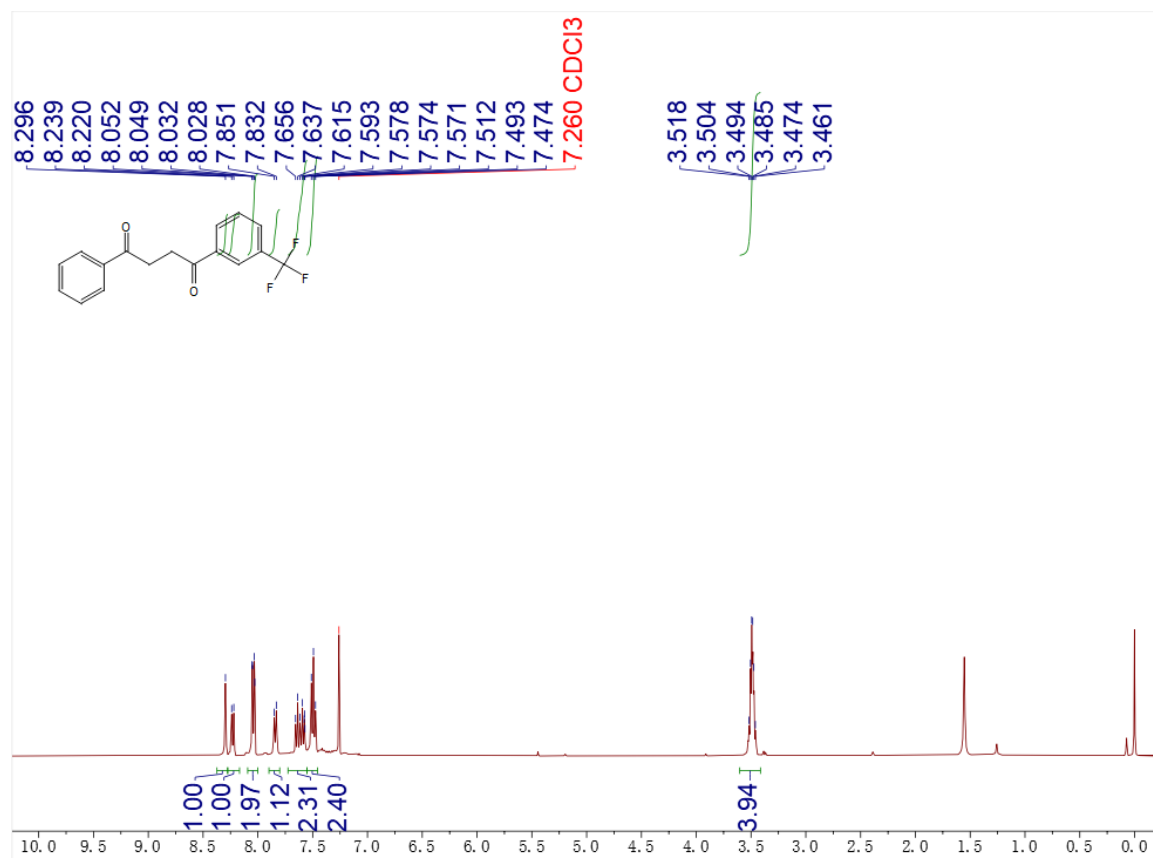
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3ad**



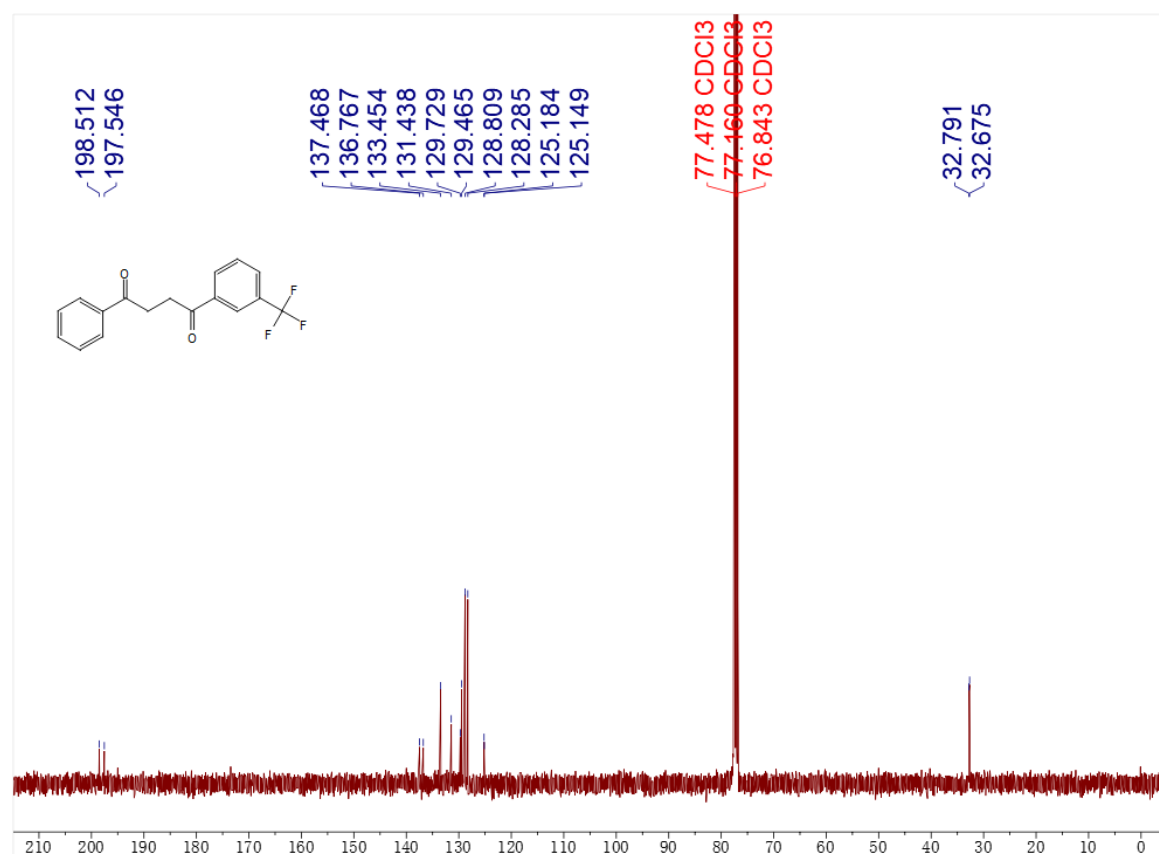
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3ad**



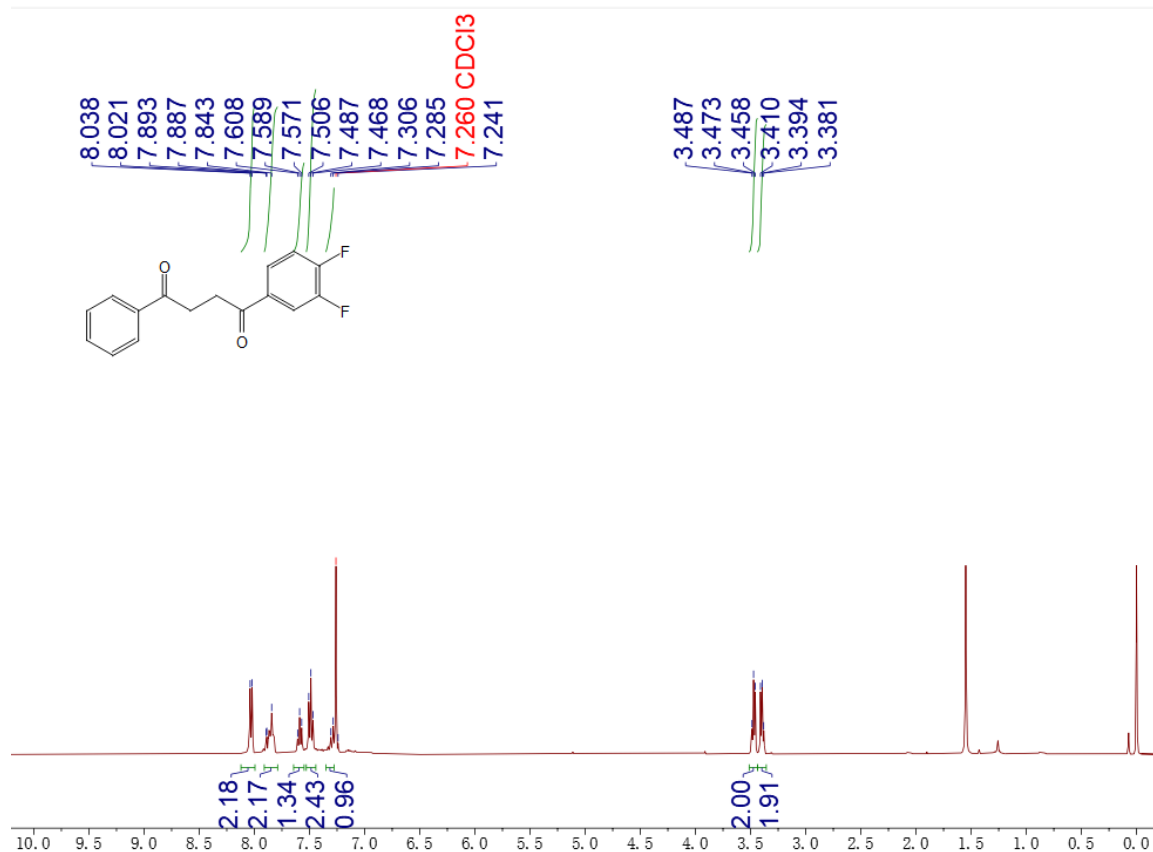
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3ag**



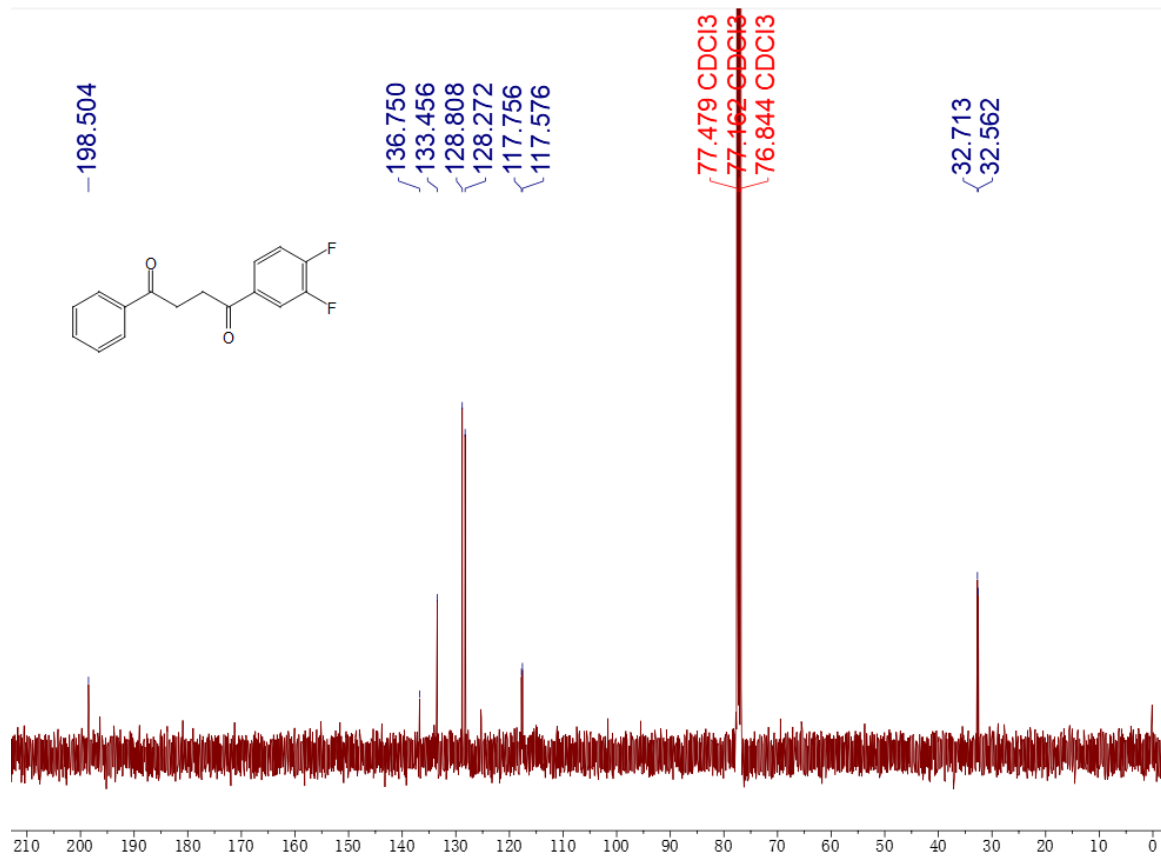
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3ag**



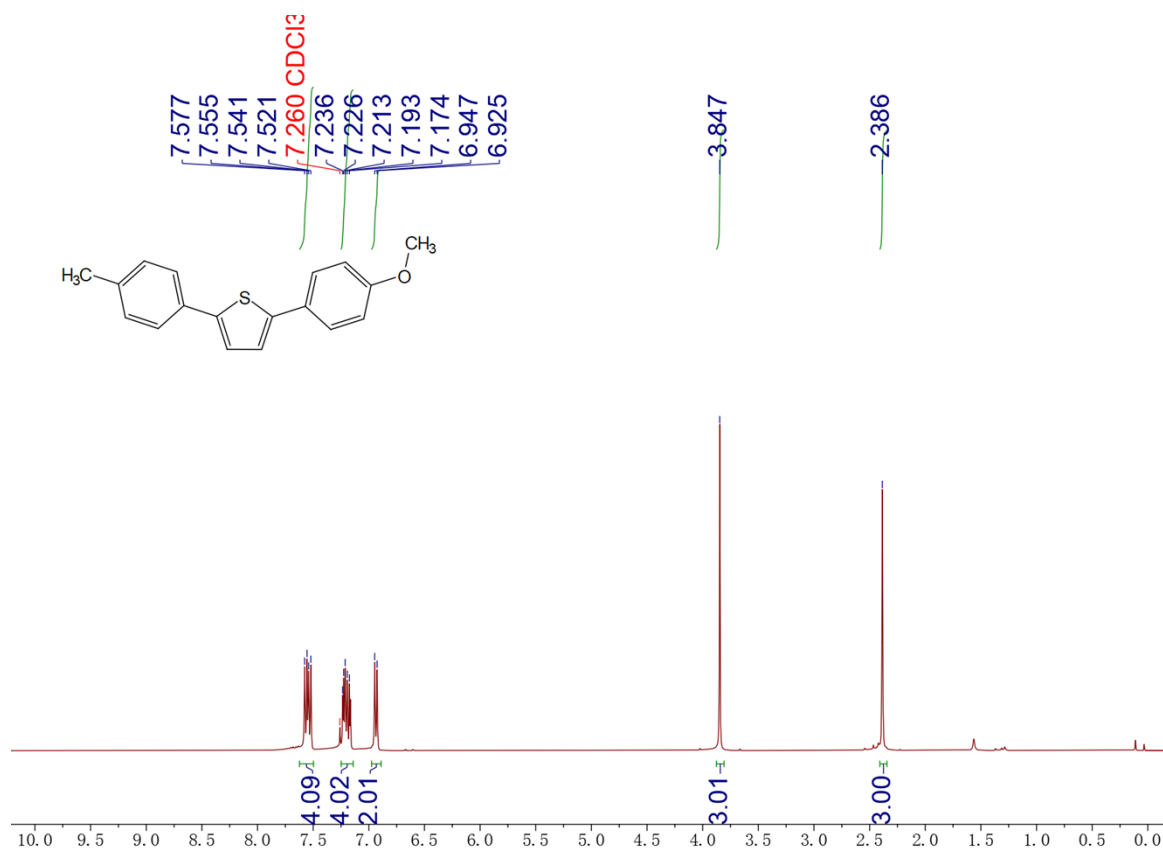
¹H NMR spectra (400 MHz) in CDCl₃ of compound **3ah**



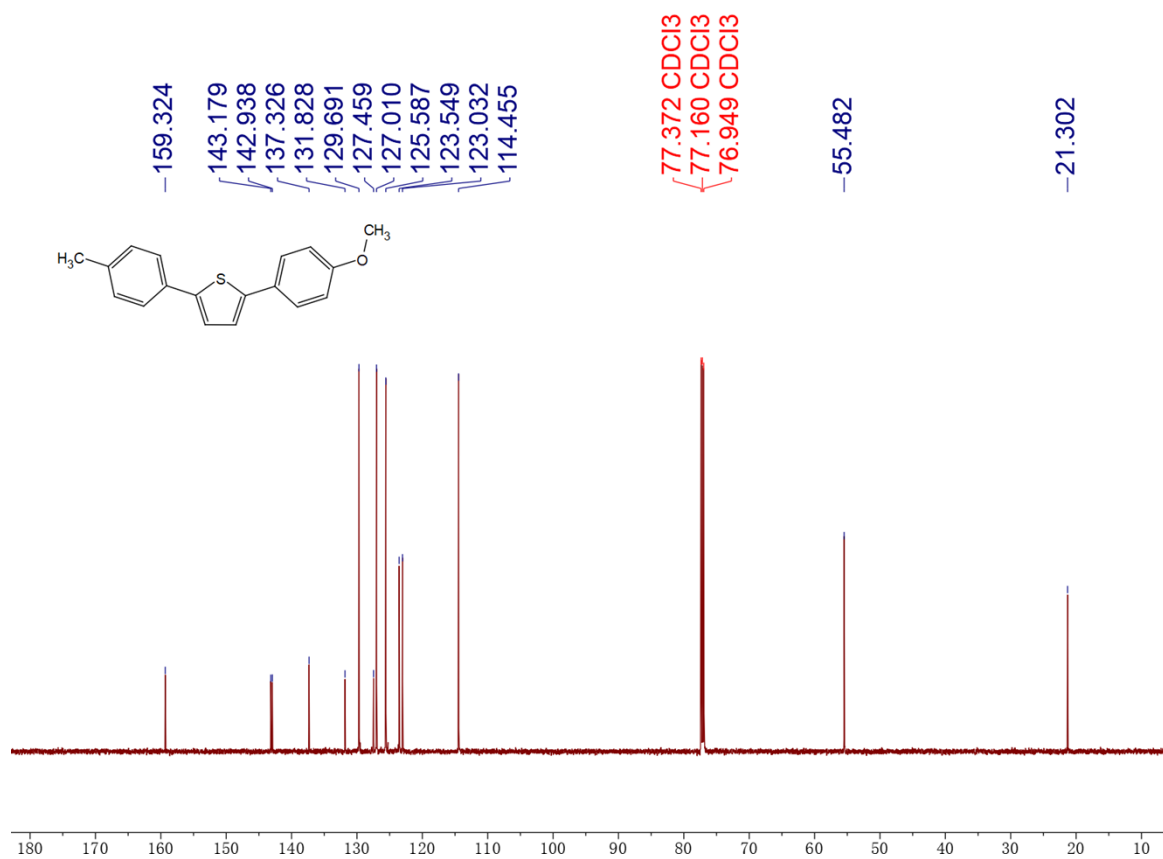
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **3ah**



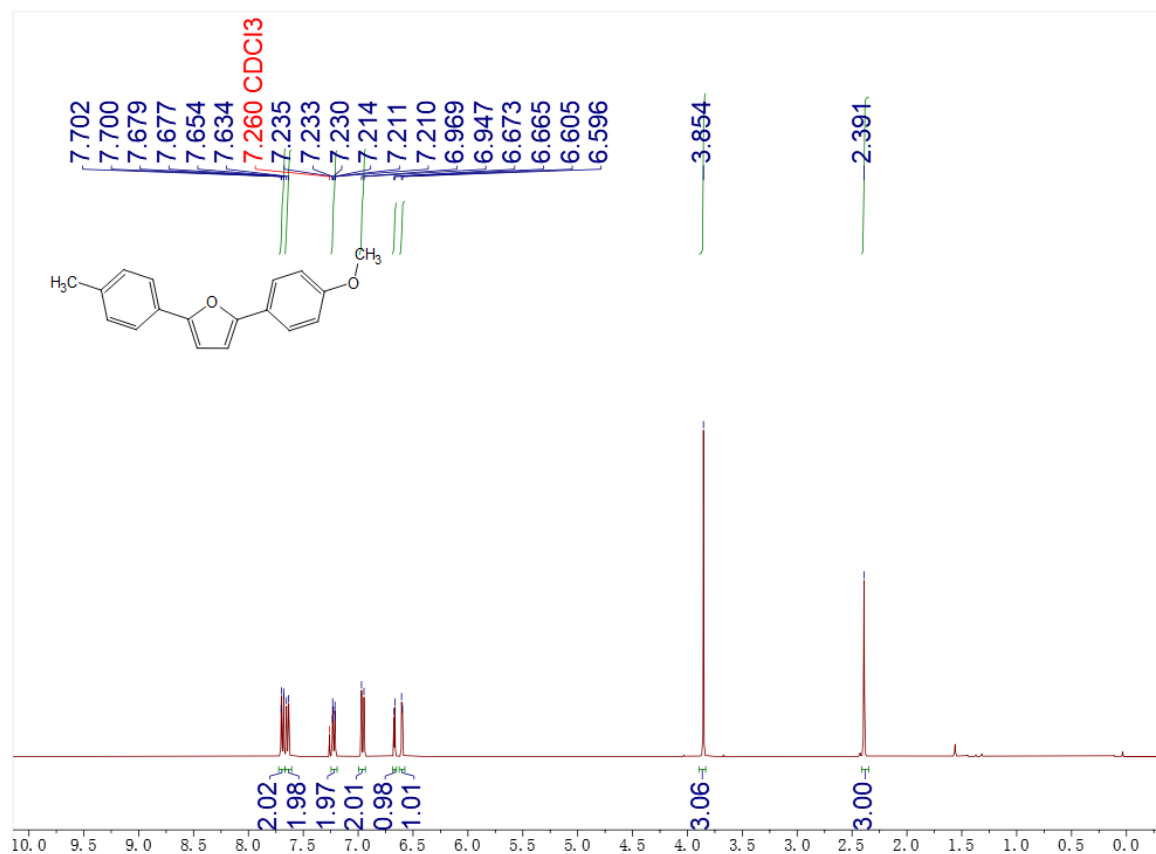
¹H NMR spectra (400 MHz) in CDCl₃ of compound **11**



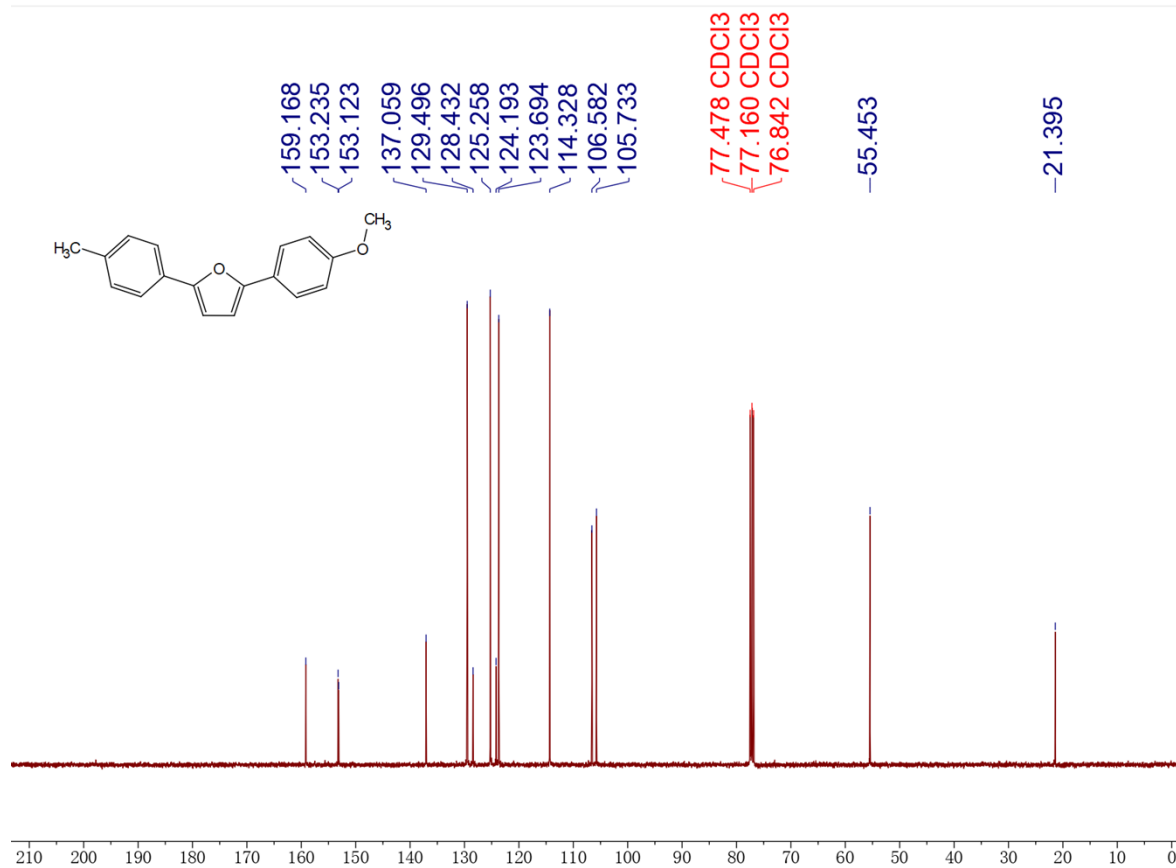
¹³C NMR spectra (600 MHz) in CDCl₃ of compound **11**



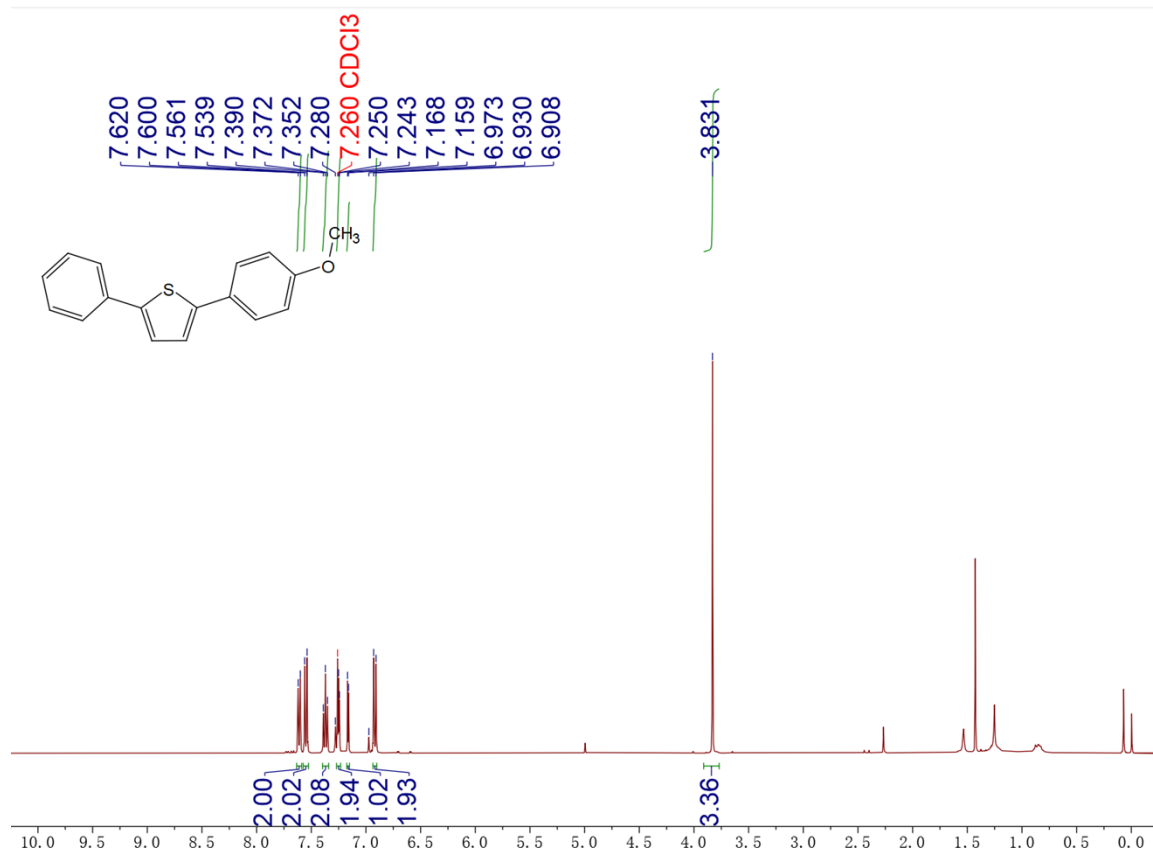
¹H NMR spectra (400 MHz) in CDCl₃ of compound **12**



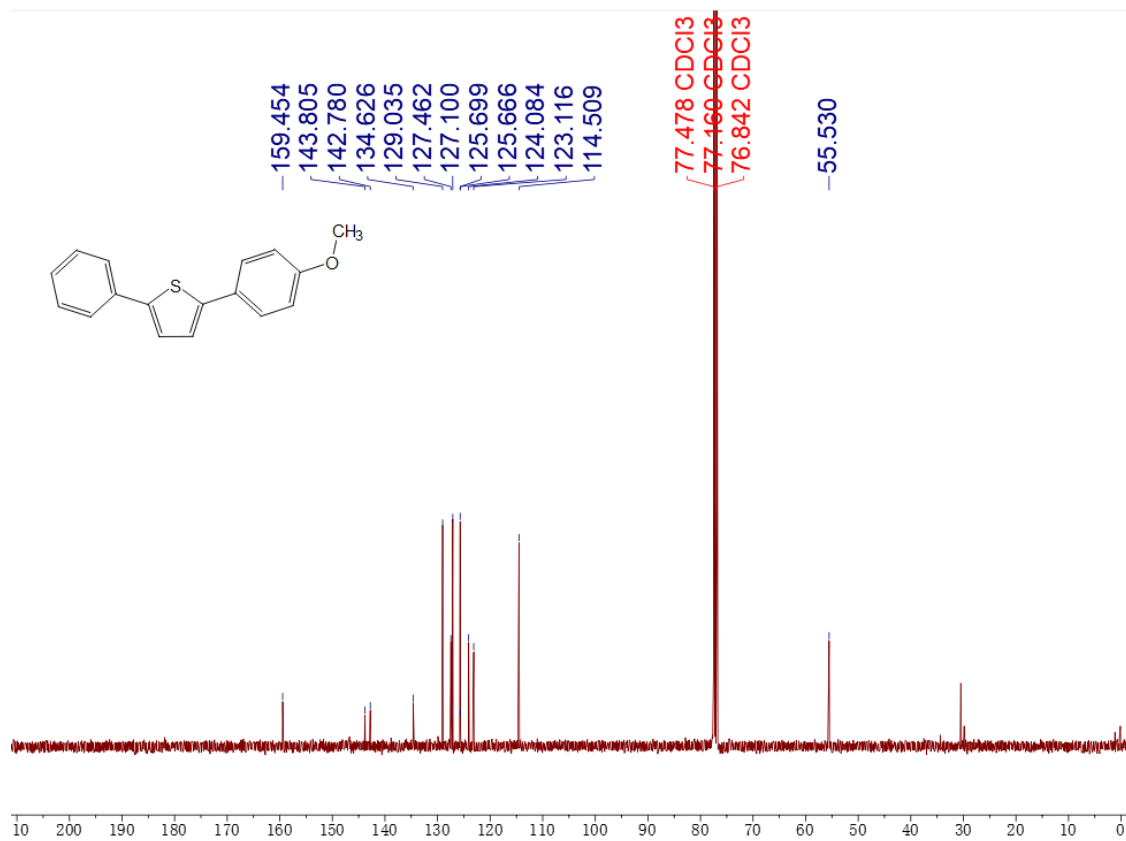
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **12**



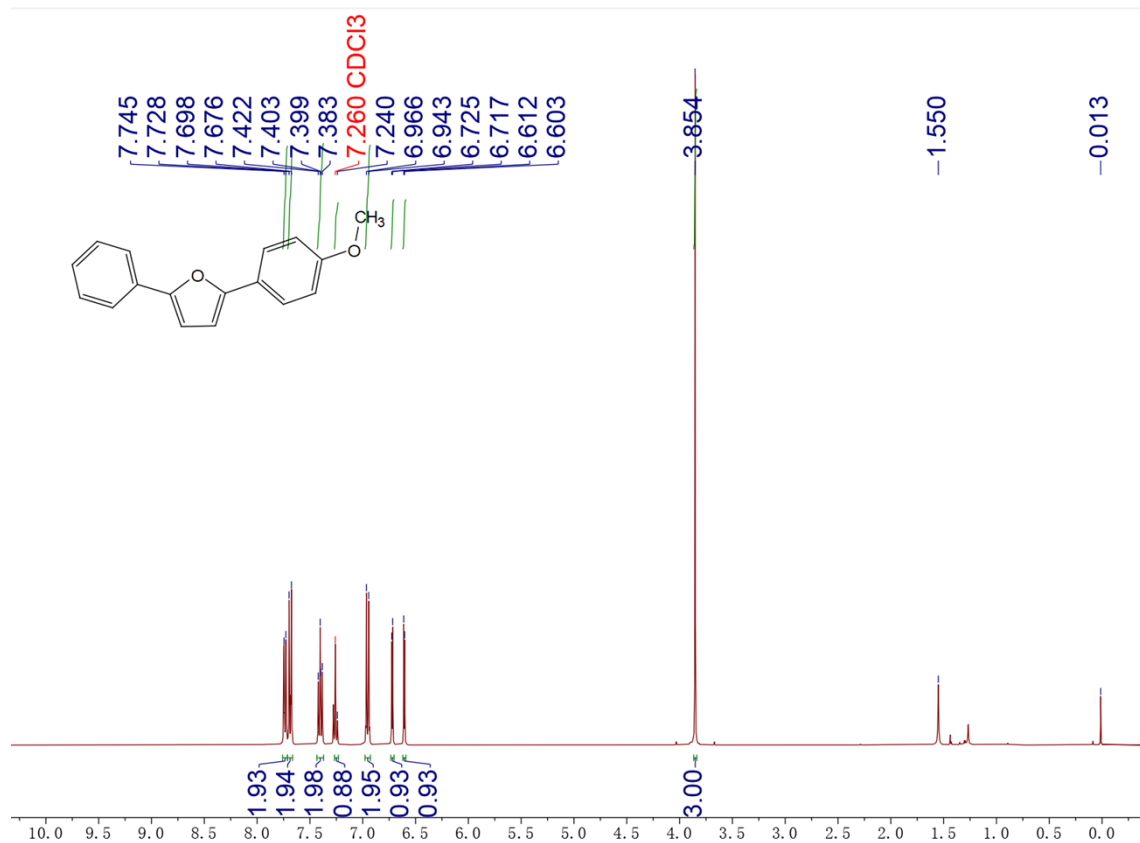
¹H NMR spectra (400 MHz) in CDCl₃ of compound **13**



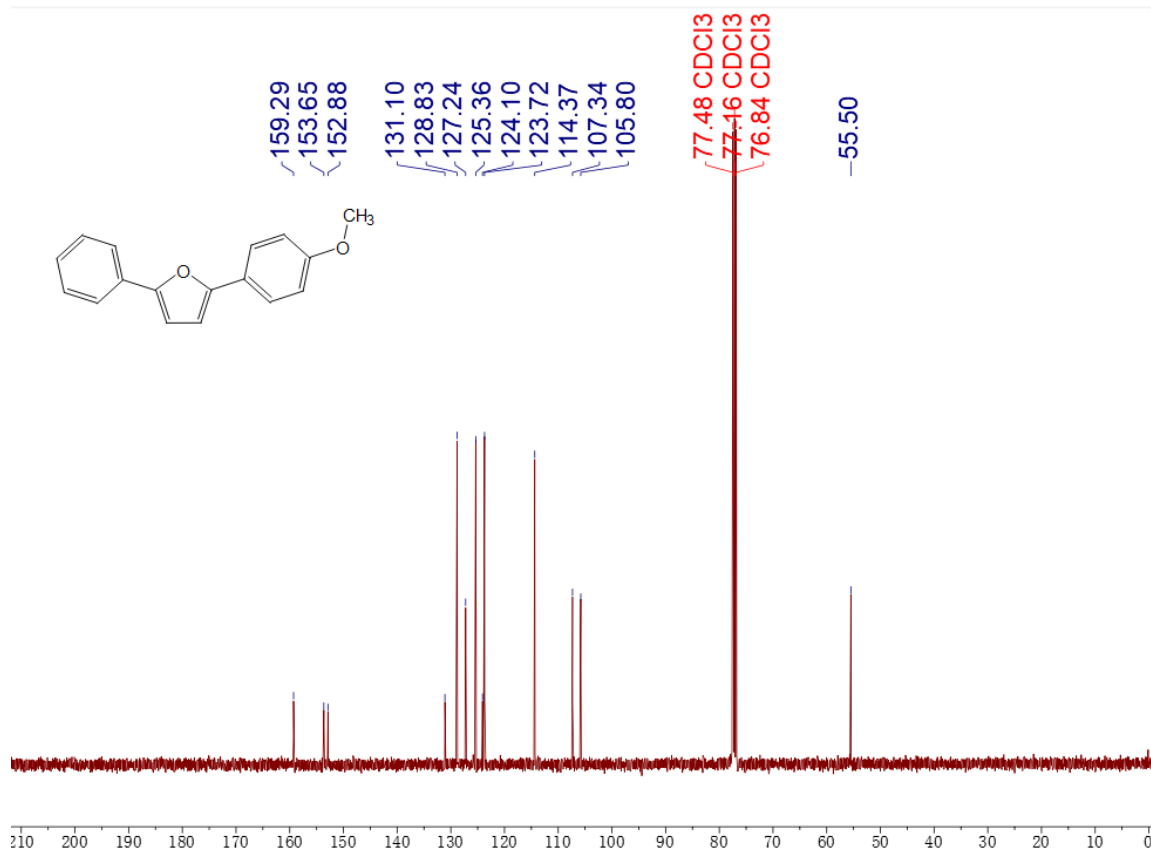
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **13**



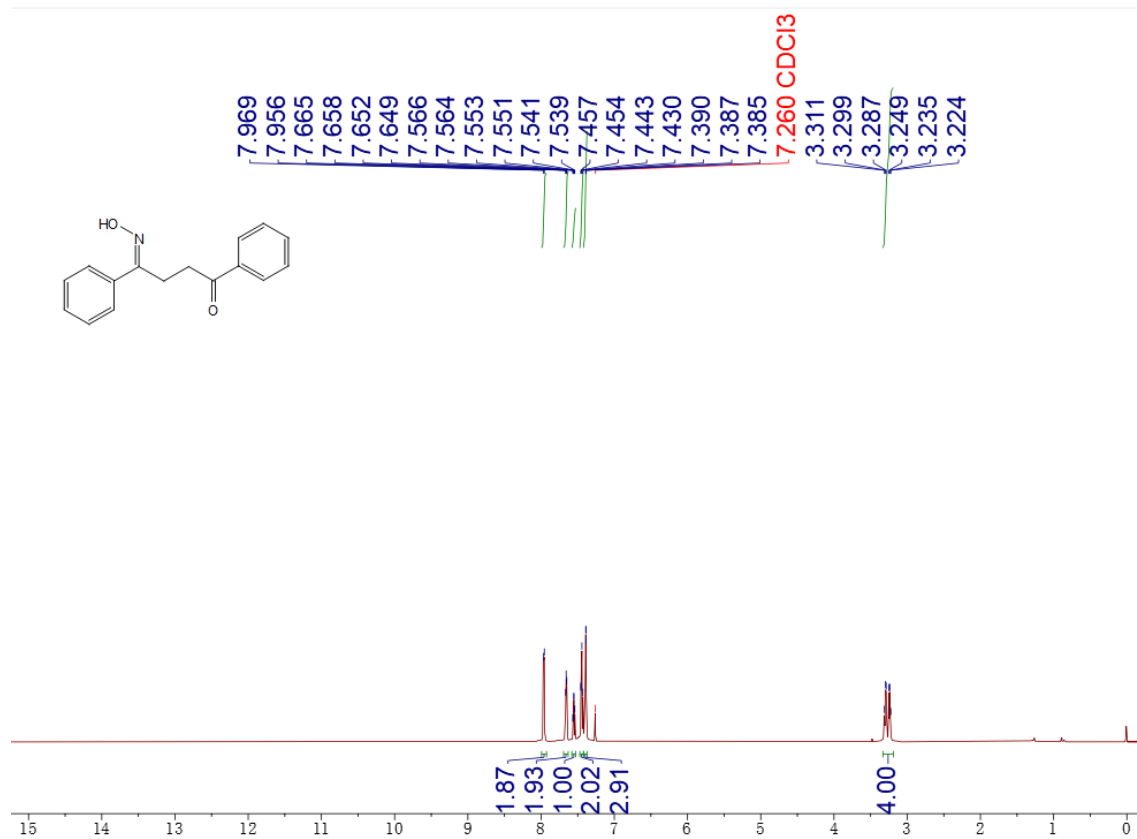
¹H NMR spectra (400 MHz) in CDCl₃ of compound **14**



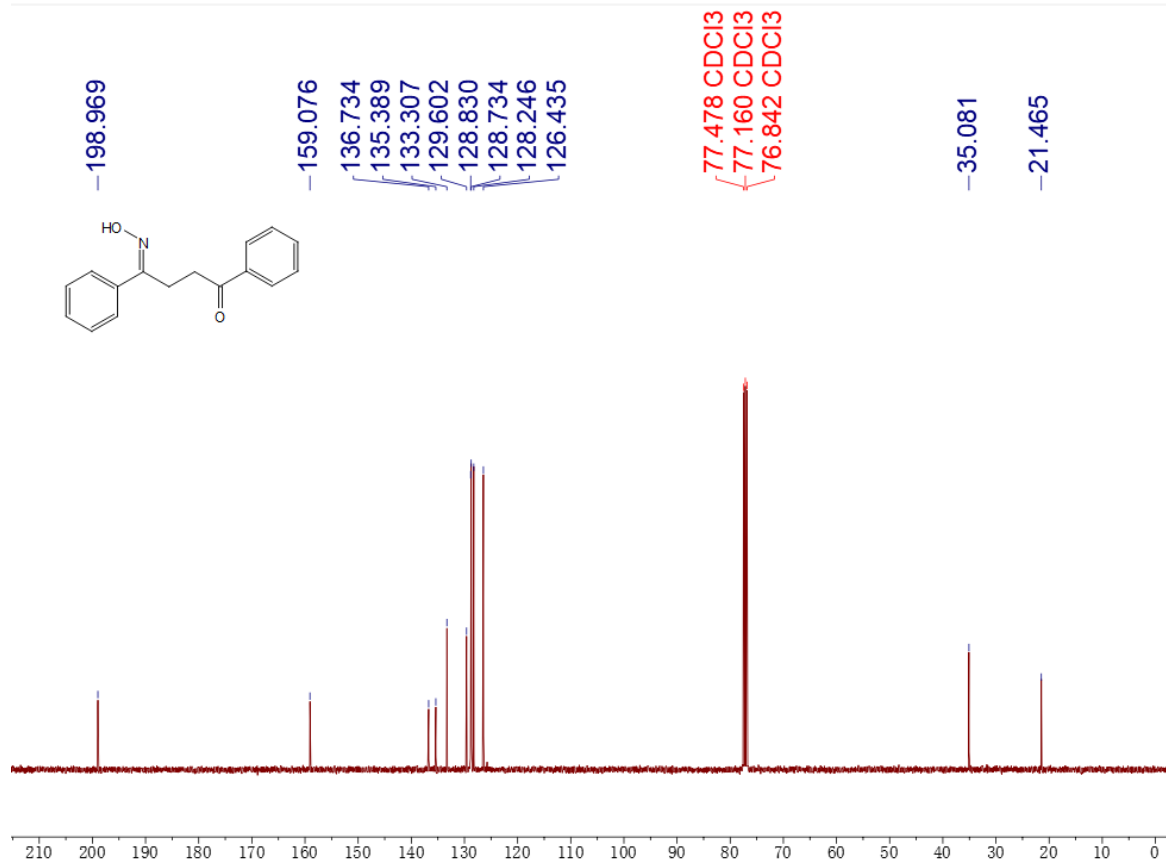
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **14**



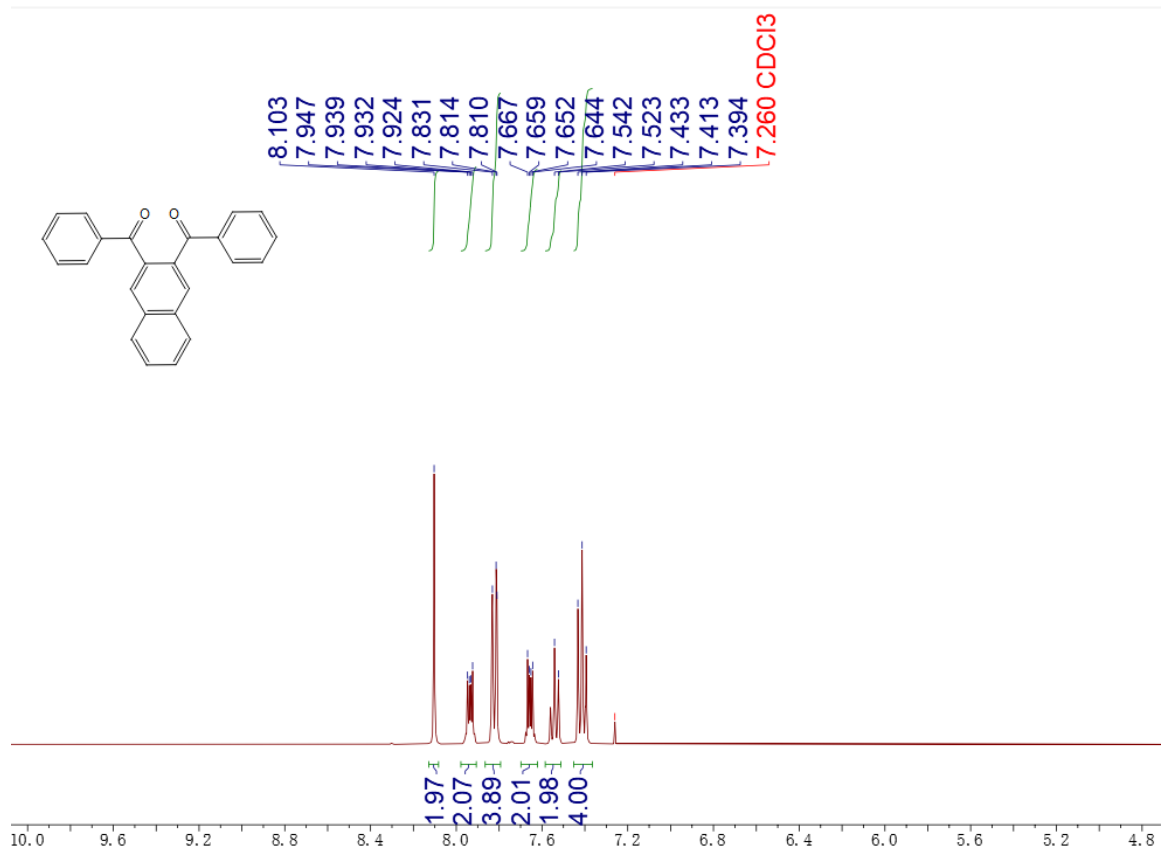
¹H NMR spectra (600 MHz) in CDCl₃ of compound **15**



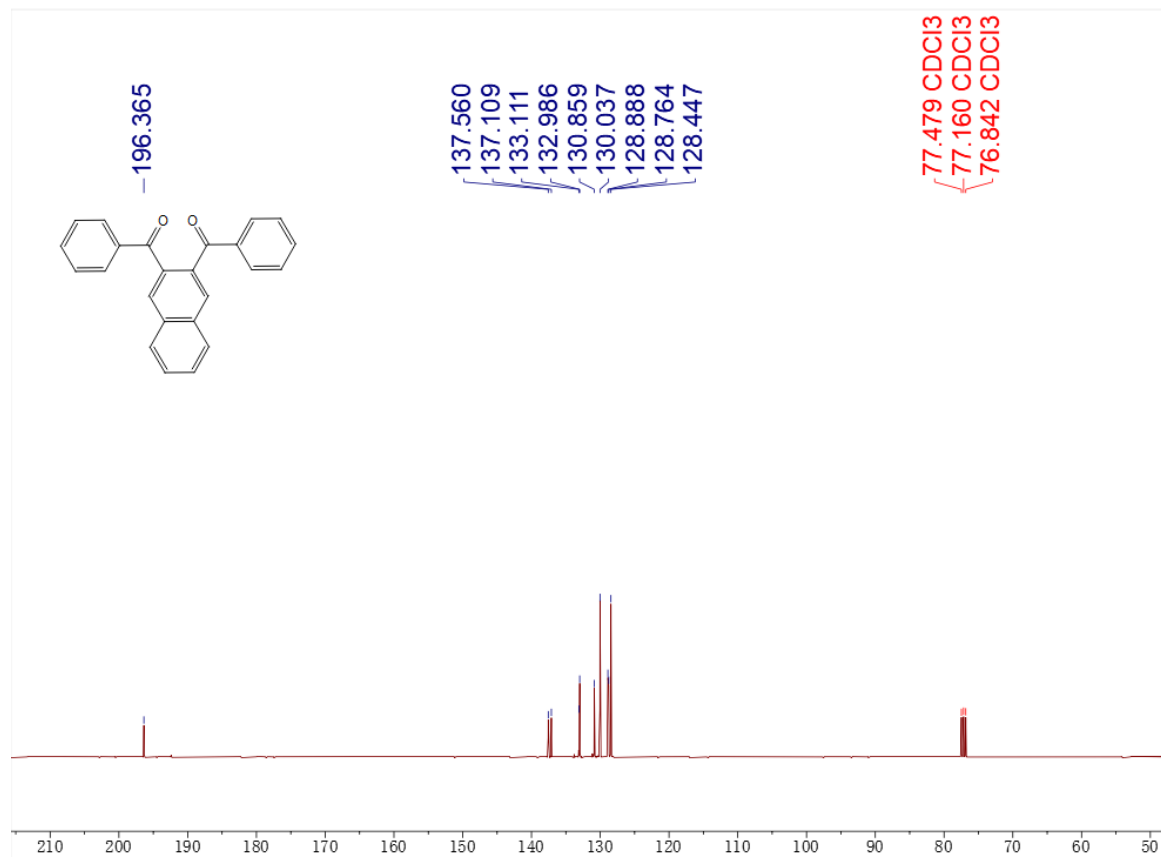
¹³C NMR spectra (400 MHz) in CDCl₃ of compound **15**



¹H NMR spectra (400 MHz) in CDCl₃ of compound **16**



¹³C NMR spectra (400 MHz) in CDCl₃ of compound **16**



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