

Electronic Supplementary Information

Table of Contents

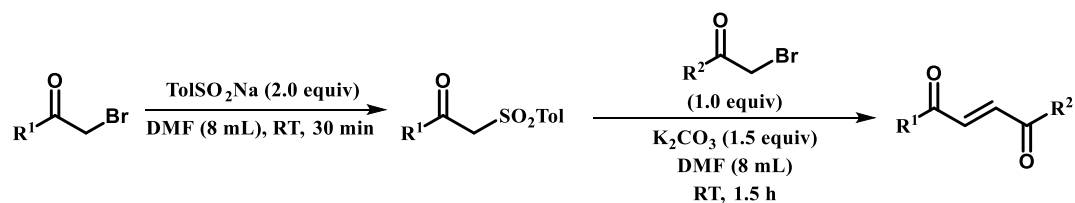
1. General information	1
2. General preparation of 1,4-enedione compounds	2
3. General preparation of 1,4-dione compounds	3
4. Optimization of reaction conditions	4
5. Gram-scale reaction	5
6. Decagram-scale reaction	7
7. Deuteration gram-scale reaction	8
8. Cyclic voltammetry study	9
9. Synthetic applications	12
10. Proposed reaction mechanism.....	15
11. Unsuccessful substrates.....	21
12. NMR spectra for the obtained compound	22
13. References.....	35
14. NMR spectra	37

1. General information

All reactions were carried out in sealed air using oven dried glassware. 1,1,1,3,3,3-hexafluoro-2-propanol, 1,2-dichloroethane, tetrabutylammonium perchlorate, and graphite felt are all available from commercial sources. Deionized water is obtained by ultra pure water machine. The electrochemical instrument is HONGSHENGFENG DPS-305BM. Column chromatography was performed on silica gel (200-300 mesh). NMR spectra were recorded in CDCl₃ on 500 MHz spectrometers. ¹H NMR chemical shifts (δ) are reported in parts per million relative to tetramethylsilane (0 ppm). The following abbreviations are used for multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, and m = multiplet. HRMS were obtained on an Ultima Global spectrometer with an ESI source. Melting points are uncorrected.

2. General preparation of 1,4-enedione compounds

Method :



Scheme S1. General preparation of 1,4-enedione compounds

Method:

Generally procedure for the 1,4-enedione compounds: add DMF (8 mL) into a 25 mL flask α -Bromoacetophenone (4.0 mmol), and then aromatic sodium sulfonate (8.0 mmol) was added at room temperature. The reaction mixture was stirred for 30 min. Dilute with brine (50 mL) and extract the mixture with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered, and evaporated to afford intermediates in nearly quantitative yields. Without further purification, K_2CO_3 (7.5 mmol), the resulting intermediates (4.0 mmol), α -Bromoacetophenone (4.0 mmol) was added to DMF (8 mL) at RT. The reaction mixture was stirred at RT for 2 h. The solvent was diluted with water (50 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered, and evaporated to afford crude product. Purification on silica gel (PE/ EA = 20/1~10/1) afforded the desired 1,4-enedione as a yellow solid.

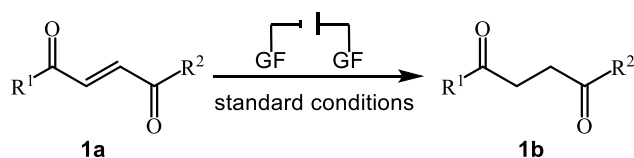
3. General preparation of 1,4-dione compounds



Figure S1. Components required for the reaction



Figure S2. Typical reaction set up



To the mixed solution of dichloroethane (4.5 mL), HFIP (0.5 mL), and 100 μL water, 1,4-enedione **1a** (0.1 mmol, 1.0 eq.) and tetrabutylammonium perchlorate (0.2 mmol, 2.0 eq.) were added. Equipped with graphite felt (10 \times 10 \times 5 mm) as an anode, graphite felt (10 \times 10 \times 5 mm) as a cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 $^\circ\text{C}$ in the Air for 2 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO_4 , filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate (PE/EA = 15:1).

4. Optimization of reaction conditions

Table S1. Screening of solvents

Entry	Deviation from standard conditions ^a	Yield (%) ^b
1	HFIP instead of solvent	70%
2	DCE instead of solvent	15%
3	CH ₃ CN instead of solvent	8%
4	THF instead of solvent	11%
5	acetone instead of solvent	trace
6	DMSO instead of solvent	n.r. ^c
7	CH ₃ NO ₂ instead of solvent	10%
8	DMF instead of solvent	n.d. ^d
9	HFIP : DCM = 0.5 : 4.5	83%
10	HFIP : acetone = 0.5 : 4.5	60%
11	Acetone : DCE = 0.5 : 4.5	20%
12	DMSO : DCE = 0.5 : 4.5	28%
13	HFIP : DCE = 1 : 4	92%
14	HFIP : DCE = 2 : 3	89%
15	HFIP : DCE = 3 : 2	68%
16	HFIP : DCE = 4 : 1	69%

^aStandard condition: graphite felt anode (10 mm × 10 mm × 5 mm), graphite felt cathode (10 mm × 10 mm × 5 mm), undivided cell, constant current = 5 mA, **1a** (0.1 mmol), electrolyte (2.0 eq.), DCE = 4.5 mL, HFIP = 0.5 mL, H₂O = 0.1 mL, 20 °C, Air, RT, 2 h (3.73 F·mol⁻¹), ^bIsolated yields, ^cn.r. = not reaction, ^dn.d. = not detected.

Table S2. Screening of H₂O

Entry	Deviation from standard conditions ^a	Yield (%) ^b
1	No H ₂ O	68%
2	10 μL H ₂ O	83%
3	50 μL H ₂ O	92%
4	150 μL H ₂ O	86%

^aStandard condition: graphite felt anode (10 mm × 10 mm × 5 mm), graphite felt cathode (10 mm × 10 mm × 5 mm), undivided cell, constant current = 5 mA, **1a** (0.1 mmol), electrolyte (2.0 eq.), DCE = 4.5 mL, HFIP = 0.5 mL, H₂O = 0.1 mL, 20 °C, Air, 2 h (3.73 F·mol⁻¹), ^bIsolated yields.

Table S3. Screening of electrode

Entry	Deviation from standard conditions ^a	Yield (%) ^b
1	GF(+) / Pt(-)	67%
2	GF(+) / C(-)	83%

^aStandard condition: graphite felt anode (10 mm × 10 mm × 5 mm), graphite felt cathode (10 mm × 10 mm × 5 mm), undivided cell, constant current = 5 mA, **1a** (0.1 mmol), electrolyte (2.0 eq.), DCE = 4.5 mL, HFIP = 0.5 mL, H₂O = 0.1 mL, 20 °C, Air, RT, 2 h (3.73 F·mol⁻¹), ^bIsolated yields.

Table S4. Screening of electrolyte

Entry	Deviation from standard conditions ^a	Yield (%) ^b
1	<i>n</i> -Bu ₄ NPF ₆ instead of <i>n</i> -Bu ₄ NCIO ₄	68%
2	<i>n</i> -Bu ₄ NBF ₄ instead of <i>n</i> -Bu ₄ NCIO ₄	62%
3	<i>n</i> -Bu ₄ NI instead of <i>n</i> -Bu ₄ NCIO ₄	trace
4	<i>n</i> -Bu ₄ NBr instead of <i>n</i> -Bu ₄ NCIO ₄	trace
5	<i>n</i> -Bu ₄ NOAc instead of <i>n</i> -Bu ₄ NCIO ₄	55%
6	Et ₄ NPF ₆ instead of <i>n</i> -Bu ₄ NCIO ₄	52%
7	Me ₄ NPF ₆ instead of <i>n</i> -Bu ₄ NCIO ₄	24%

^aStandard condition: graphite felt anode (10 mm × 10 mm × 5 mm), graphite felt cathode (10 mm × 10 mm × 5 mm), undivided cell, constant current = 5 mA, **1a** (0.1 mmol), electrolyte (2.0 eq.), DCE = 4.5 mL, HFIP = 0.5 mL, H₂O = 0.1 mL, 20 °C, Air, RT, 2 h (3.73 F·mol⁻¹), ^bIsolated yields.

Table S5. Screening of other hydrogen donor

Entry	Deviation from standard conditions ^a	Yield (%) ^b
1	Cheng's condition: ^{12a} D ₂ O as hydrogen donor	trace
2	Cheng's condition: ^{12b} NH ₃ as hydrogen donor	trace
3	Xia's condition: ^{12c} MeOH and NH ₄ Cl as hydrogen donor	trace
4	Huang's condition: ^{12d} DMSO and H ₂ O as hydrogen donor	trace
5	Wen's condition: ^{12e} MeOH as hydrogen donor	trace

^aStandard condition: graphite felt anode (10 mm × 10 mm × 5 mm), graphite felt cathode (10 mm × 10 mm × 5 mm), undivided cell, constant current = 5 mA, **1a** (0.1 mmol), electrolyte (2.0 eq.), DCE = 4.5 mL, HFIP = 0.5 mL, H₂O = 0.1 mL, 20 °C, Air, RT, 2 h (3.73 F·mol⁻¹), ^bIsolated yields.

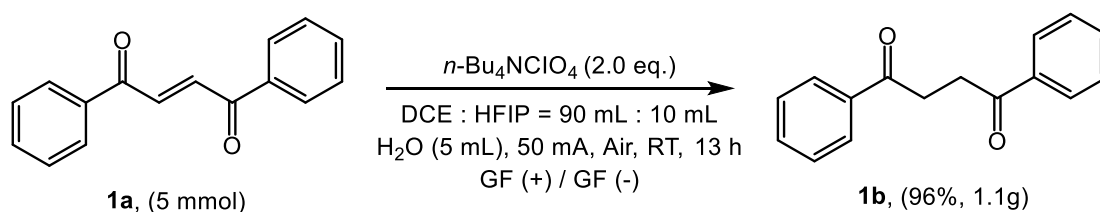
5. Gram-scale reaction



Figure S3. Components required for gram-scale reaction



Figure S4. Typical reaction set up for gram-scale reaction



To the mixed solution of dichloroethane (90 mL), HFIP (10 mL), and 5 mL water, 1,4-enedione **1a** (5 mmol, 1.0 eq.) and tetrabutylammonium perchlorate (10 mmol, 2.0 eq.) were added. Equipped with graphite felt (20mm × 10mm × 5 mm) as anode, graphite felt (20mm × 10mm × 5 mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 50 mA at 20 °C in the Air for 13 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO₄, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate (PE/EA = 15:1).



Figure S5. Pure product

6. Decagram-scale reaction



Figure S6. Components required for decagram-scale reaction

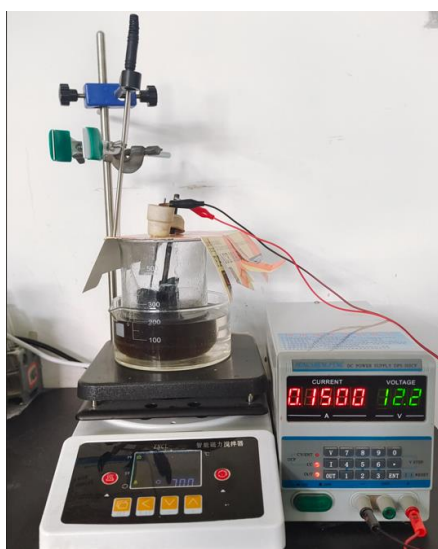
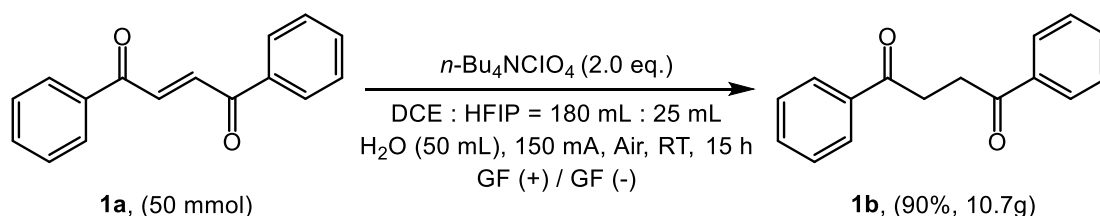


Figure S7. Typical reaction set up for decagram-scale reaction



To the mixed solution of dichloroethane (180 mL), HFIP (25 mL), and 50 mL water, 1,4-enedione **1a** (50 mmol, 1.0 eq.) and tetrabutylammonium perchlorate (100 mmol, 2.0 eq.) were added. Equipped with graphite felt (40mm × 20mm × 10 mm) as anode, graphite felt (40mm × 20mm × 10 mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 150 mA at 20 °C in Air for 15 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO_4 , filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate (PE/EA = 15:1).



Figure S8. Pure product

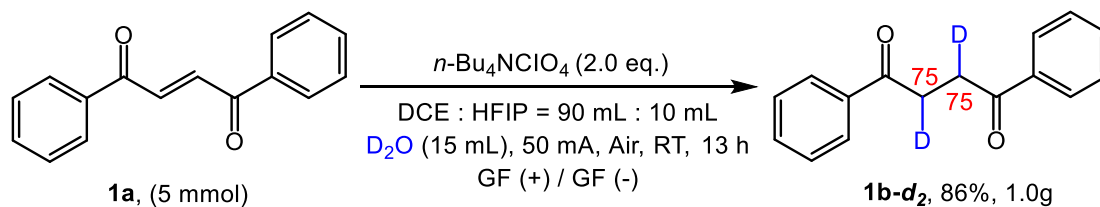
7. Deuteration gram-scale reaction



Figure S9. Components required for deuteration gram-scale reaction



Figure S10. Typical reaction set up for deuteration gram-scale reaction



To the mixed solution of dichloroethane (90 mL), HFIP (10 mL), and 15 mL D₂O, 1,4-enedione **1a** (5 mmol, 1.0 eq.) and tetrabutylammonium perchlorate (10 mmol, 2.0 eq.) were added. Equipped with graphite felt (20mm × 10mm × 5 mm) as anode, graphite felt (20mm × 10mm × 5 mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at

constant current of 50 mA at 20 °C in Air for 13 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO_4 , filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate (PE/ EA = 15:1).



Figure S11. Pure product

8. Cyclic voltammetry study

The cyclic voltammetry experiments were carried out with a computer-controlled electrochemical analyzer for electrochemical measurements. The data was collected with the CS300H potentiostat (Wuhan Coster Instrument Co., LTD).

Working electrode: The working electrode is a 3 mm diameter glassy carbon working electrode. Polished with 0.05 μm aluminum oxide and then sonicated in distilled water and ethanol before measurements.

Reference electrode: The reference electrode is SCE (saturated aqueous KCl) that was washed with water and ethanol before measurements.

Counter electrode: The counter electrode is a platinum wire that was polished with 0.05 μm aluminum oxide and then sonicated in distilled water and ethanol before measurements.

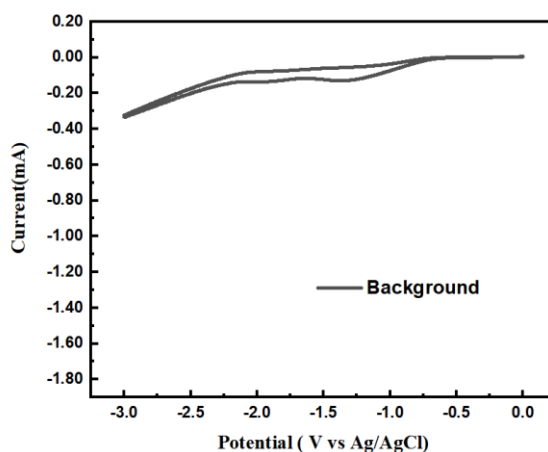


Figure S12. Cyclic voltammogram of **blank** sample

General procedure for cyclic voltammetry (CV): Cyclic voltammogram of **blank** sample was performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. A solution of *n*-Bu₄NClO₄ (0.2 mmol) in 5 mL DCE and 0.1 mL H₂O was subject to cyclic voltammetry experiment. The scan rate was 100 mV/s, ranging from -3.0 V to 0.0 V.

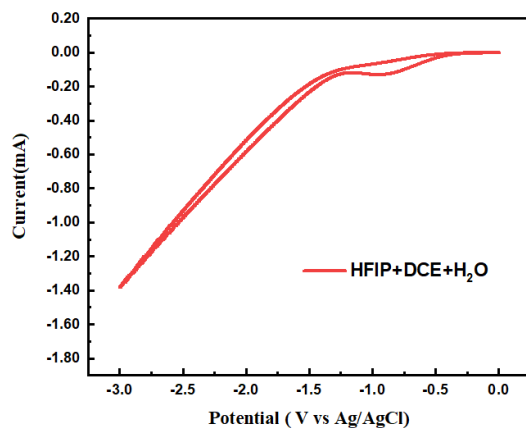


Figure S13. Cyclic voltammogram of HFIP

General procedure for cyclic voltammetry (CV): Cyclic voltammogram of HFIP was performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. A solution of *n*-Bu₄NClO₄ (0.2 mmol) in 4.5 mL DCE, 0.5 mL HFIP and 0.1 mL H₂O was subject to cyclic voltammetry experiment. The scan rate was 100 mV/s, ranging from -3.0 V to 0.0 V.

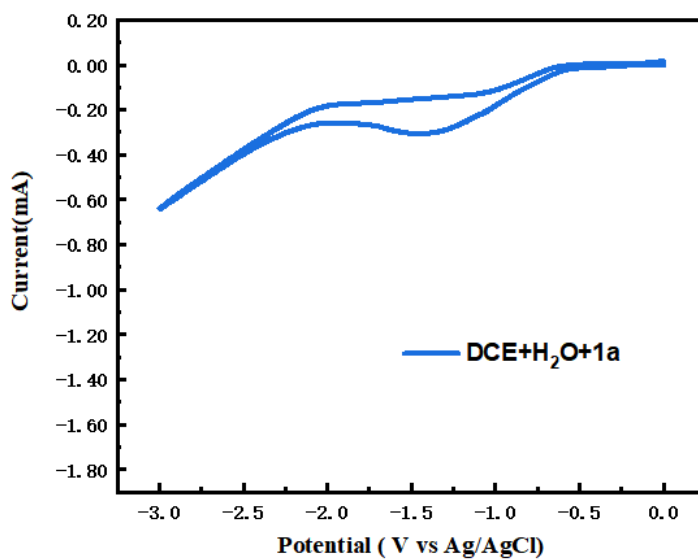


Figure S14. Cyclic voltammogram of 1a

General procedure for cyclic voltammetry (CV): Cyclic voltammogram of **1a** was performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. A solution of **1a** (0.1 mmol) and *n*-Bu₄NClO₄ (0.2 mmol) in 5.0 mL DCE and 0.1 mL H₂O was subject to cyclic voltammetry experiment. The scan rate was 100 mV/s, ranging from -3.0 V to 0.0 V.

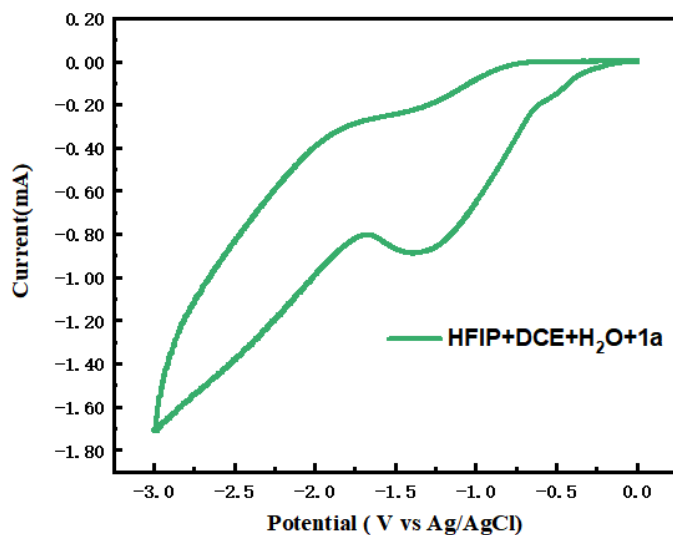


Figure S15. Cyclic voltammograms of **1a** and HFIP

General procedure for cyclic voltammetry (CV): Cyclic voltammograms of **1a** and HFIP were performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. A solution of **1a** (0.1 mmol) and *n*-Bu₄NClO₄ (0.2 mmol) in 4.5 mL DCE, 0.5 mL HFIP and 0.1 mL H₂O was subject to cyclic voltammetry experiment. The scan rate was 100 mV/s, ranging from -3.0 V to 0.0 V.

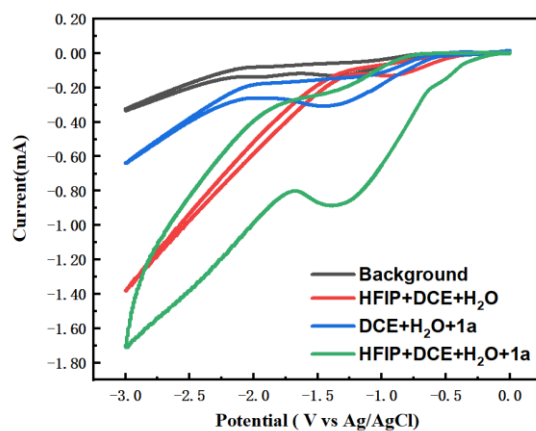
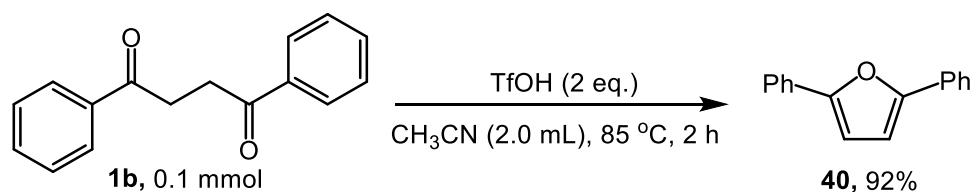


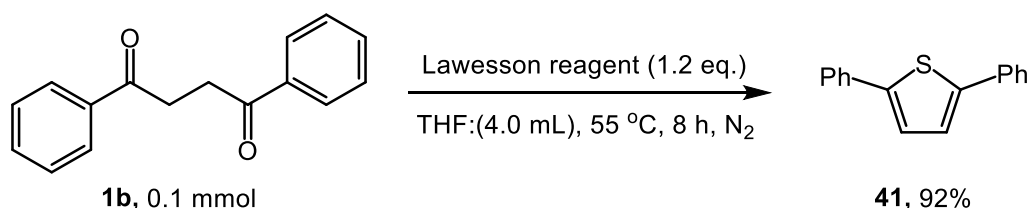
Figure S16. Cyclic voltammograms of HFIP, DCE, H₂O and **1a**

9. Synthetic applications

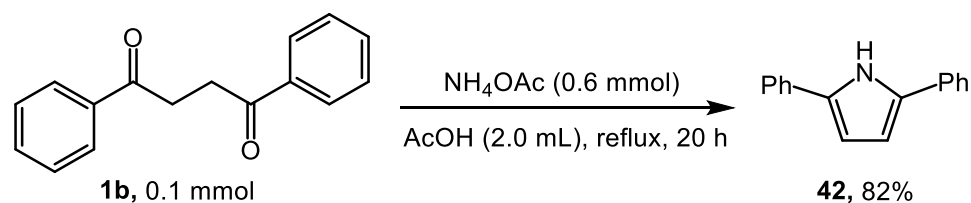
Conversions of **1b** to Compounds **40–46**, **40-*d*₂**, and **41-*d*₂**



2,5-diphenylfuran (40).¹⁴ To a dry schlenk tube that charged with nitrogen, 1,4-diketone **1b** (0.1 mmol, 24 mg) was added, then freshly distilled CH₃CN (2.0 mL) was injected through a syringe. Subsequently, TfOH (17.3 μL, 0.2 mmol, 30 mg) was added to the mixture, and the resulting solution was stirred at 85 °C for 2 h. Then the resulting solution was cooled to room temperature, and solvent was evaporated under vacuum, the residue was purified by flash column chromatography on silica gel (eluent: PE/ EA= 100:1) to give the furan **40** as a yellow solid (20.3 mg, 92% yield). ¹H NMR (500 MHz, CDCl₃) δ 7.77 (d, *J* = 7.4 Hz, 4H), 7.42 (t, *J* = 7.8 Hz, 4H), 7.29 (t, *J* = 7.3 Hz, 2H), 6.75 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 153.4, 130.8, 128.7, 127.4, 123.7, 107.2.

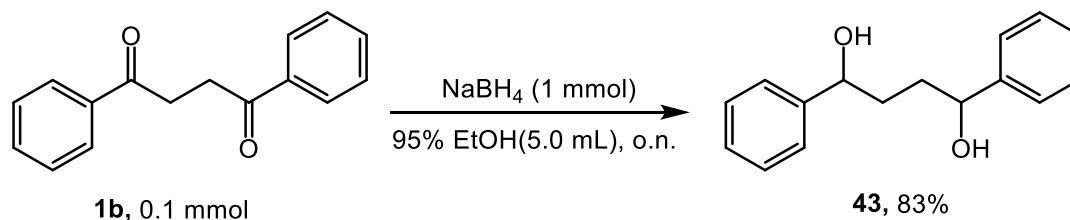


2,5-diphenylthiophene (41).¹⁴ A schlenk tube was charged under N₂ atmosphere with 1,4-diketones **1b** (0.1 mmol, 24 mg), Lawesson's reagent (0.12 mmol, 49 mg), THF (4 mL), and heated to 55 °C. After 8 h, when the reaction was completed as determined by TLC, 2 g alumina was added and the THF was evaporated. The resulting solid was placed on a short column packed with silica which was eluted with acetonitrile to give **41** as a white solid (21.7 mg, 92% yield). ¹H NMR (500 MHz, CDCl₃) δ 7.64 (d, *J* = 7.6 Hz, 4H), 7.40 (t, *J* = 7.7 Hz, 4H), 7.31 – 7.28 (m, 4H). ¹³C NMR (125 MHz, CDCl₃) δ 143.6, 134.3, 128.9, 127.5, 125.6, 124.0.

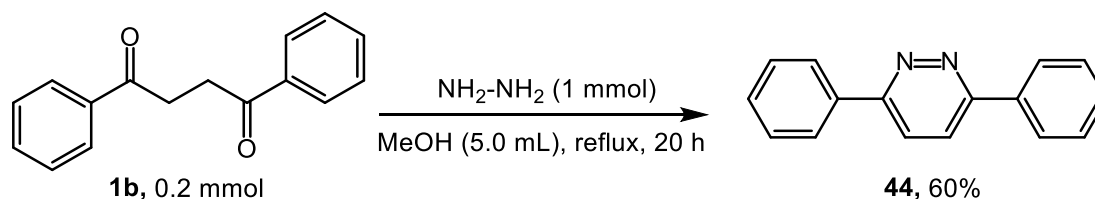


2,5-diphenyl-1H-pyrrole (42).¹⁴ A mixture of 1,4-diketone **1b** (0.1 mmol, 24 mg), ammonium acetate (0.6 mmol, 47 mg), and acetic acid (2 mL) was heated to reflux for 20 h. After cooling, the reaction mixture was poured into ice-water. The formed solid was collected and washed with H₂O and dried to give pure product **42** as a white solid (18.0 mg, 82% yield). ¹H NMR (500 MHz,

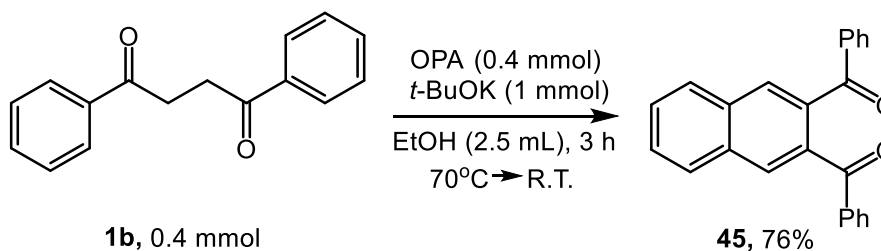
CDCl₃) δ 8.55 (s, 1H), 7.51 (d, J = 7.7 Hz, 4H), 7.37 (t, J = 7.7 Hz, 4H), 7.21 (t, J = 7.2 Hz, 2H), 6.57 (d, J = 2.6 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 133.2, 132.5, 129.0, 126.4, 123.8, 108.0.



1,4-diphenylbutane-1,4-diol (43).¹⁶ 1,4-diketone **1b** (0.1 mmol, 24 mg) was added to a flask containing ethanol (95%) in an ice bath at 0 °C. NaBH₄ (1 mmol, 38 mg) was added and the mixture was kept stirring at 0 °C for 1 h. After that time, the ice bath was removed and the mixture was stirred at room temperature overnight. Water (40 mL) and aqueous HCl (2 M) were added to the mixture until pH 7 was reached. The resulting aqueous phase was washed with ethyl acetate (3 x 20 mL) and the combined organic phases were dried over MgSO₄ and filtered off. The organic solvent was removed under vacuum and the residue was purified by column chromatography using petroleum ether and ethyl acetate (9:1 v/v) as eluent. (20.1 mg, 83% yield). ¹H NMR (500 MHz, CDCl₃) δ 7.25 – 7.19 (m, 10H), 4.57 (d, J = 4.8 Hz, 1H), 4.53 (d, J = 7.7 Hz, 1H), 3.65 (s, 1H), 3.42 (s, 1H), 1.82 – 1.68 (m, 4H). ¹³C NMR (125 MHz, CDCl₃) δ 144.7, 144.6, 128.4, 128.4, 127.4, 127.4, 125.9, 74.4, 74.0, 36.0, 35.0.

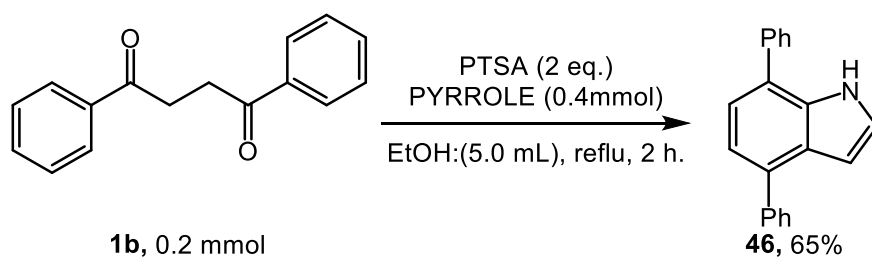


3,6-diphenylpyridazine (44).¹⁷ A hot methanolic solution of 1,4-diketone **1b** (24mg, 0.1 mmol) was dropped in excess hydrazine hydrate (12.5 mg, 0.25 mmol) and the mixture was refluxed at 70 °C in oil bath for 12 hours. After being cooled to room temperature, the mixture was filtered and the residue was washed with methanol to give the desired **44**. (27.9 mg, 60% yield). ¹H NMR (500 MHz, CDCl₃) δ 8.16 (d, J = 7.2 Hz, 4H), 7.93 (s, 2H), 7.56 – 7.49 (m, 6H). ¹³C NMR (125 MHz, CDCl₃) δ 157.7, 136.2, 130.0, 129.0, 127.0, 124.2.

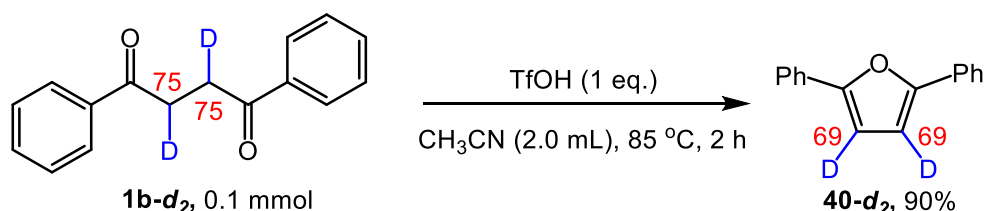


Naphthalene-2,3-diylbis(phenylmethanone) (45).¹⁵ The 1,4-diketone **1b** (0.4 mmol, 95.2 mg)

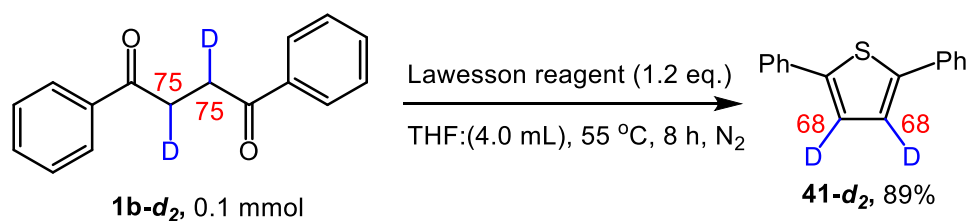
and phthalaldehyde (0.4 mmol, 53.7 mg) were dissolved in hot ethanol (5 mL). To this, *t*-BuOK (1 mmol, 1.122 mg) was slowly added and the reaction mixture was stirred for 3 h at room temperature. The solid obtained was filtered and washed with methanol to afford **45** (102.3 mg, 76%) as a colorless solid. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.10 (s, 2H), 7.92 (dd, $J = 6.2, 3.3$ Hz, 2H), 7.84 – 7.82 (m, 4H), 7.64 (dd, $J = 6.2, 3.3$ Hz, 2H), 7.53 (t, $J = 7.3$ Hz, 2H), 7.41 (t, $J = 7.3$ Hz, 4H). $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 196.4, 137.4, 137.0, 133.0, 133.0, 130.9, 130.0, 128.8, 128.8, 128.4.



4,7-diphenyl-1H-indole (46).¹⁸ Add 1,4-diketone **1b** (0.2 mmol, 48 mg), *p*-toluenesulfonic acid (0.4 mmol, 47 mg), and pyrrole (0.4 mmol, 47 mg) to ethanol (5 mL), heat and reflux for 2 hours. Then the resulting solution was cooled to room temperature, and solvent was evaporated under vacuum, the residue was purified by flash column chromatography on silica gel (eluent: PE/ EA = 15:1) to give **46**. (35.0 mg, 65% yield). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.47 (s, 1H), 7.73 (d, $J = 7.3$ Hz, 2H), 7.65 (d, $J = 7.5$ Hz, 2H), 7.49 (dt, $J = 12.2, 7.6$ Hz, 4H), 7.38 (dd, $J = 13.3, 7.4$ Hz, 2H), 7.28 (d, $J = 2.5$ Hz, 2H), 7.21 (dd, $J = 5.6, 2.8$ Hz, 1H), 6.78 – 6.77 (m, 1H). $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 140.3, 138.2, 133.2, 133.0, 128.4, 127.9, 127.7, 127.4, 126.6, 126.1, 125.6, 123.9, 123.8, 121.5, 119.5, 101.8.



2,5-diphenylfuran-3,4-d₂ (40-d₂).¹⁴ To a dry schlenk tube that charged with nitrogen, 1,4-diphenylbutane-1,4-dione-2,3-d₂ **1b-d₂** (0.1 mmol, 24 mg) was added, then freshly distilled CH_3CN (2.0 mL) was injected through a syringe. Subsequently, TfOH (17.3 μL , 0.2 mmol, 30 mg) was added to the mixture, and the resulting solution was stirred at 85 $^\circ\text{C}$ for 2 h. Then the resulting solution was cooled to room temperature, and solvent was evaporated under vacuum, the residue was purified by flash column chromatography on silica gel (eluent: PE/ EA = 100:1) to give the furan **40-d₂** as a yellow solid (20.0 mg, 90% yield). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.74 (d, $J = 7.6$ Hz, 4H), 7.40 (t, $J = 7.3$ Hz, 4H), 7.26 (t, $J = 7.3$ Hz, 2H), 6.73 (s, 1H).



2,5-diphenylthiophene-3,4-d₂ (41-d₂).¹⁴ A schlenk tube was charged under N₂ atmosphere with 1,4-diphenylbutane-1,4-dione-2,3-d₂ **1b-d₂** (0.1 mmol, 24 mg), Lawesson's reagent (0.12 mmol, 49 mg), THF (4 mL), and heated to 55°C. After 8 h, when the reaction was completed as determined by TLC, 2 g alumina was added and the THF was evaporated. The resulting solid was placed on a short column packed with silica which was eluted with acetonitrile to give **41-d₂** as a white solid (21.2 mg, 89% yield). ¹H NMR (500 MHz, CDCl₃) δ 7.66 (d, *J* = 7.6 Hz, 4H), 7.41 (t, *J* = 7.7 Hz, 4H), 7.31 (t, *J* = 3.8 Hz, 3H).

10. Proposed reaction mechanism

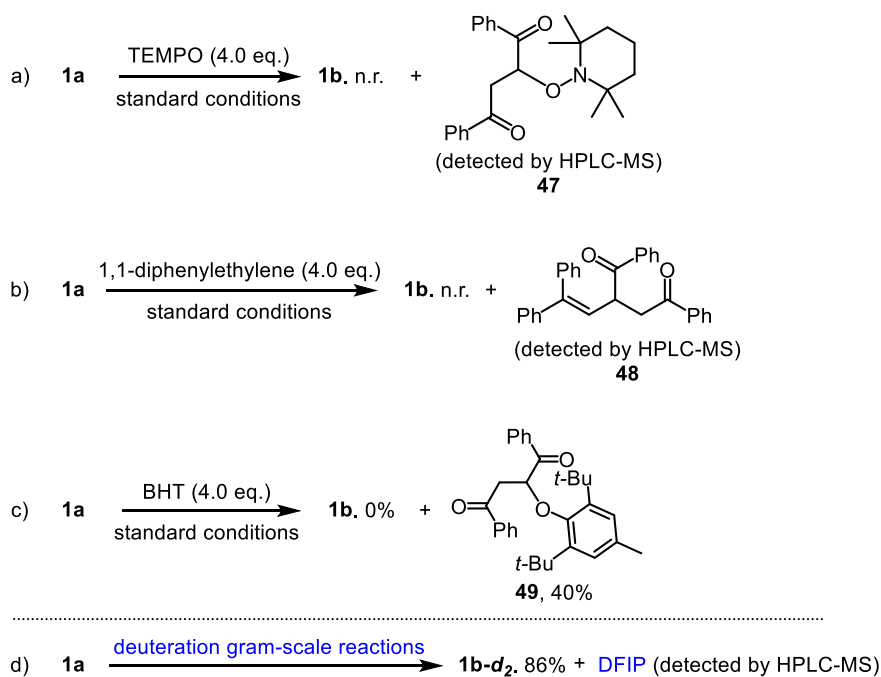
1) pH:



Figure S17. pH in the decagram-scale reaction water

The water phase is separated after the completion of the decagram-scale reaction, and the measured pH value is 2.10, which is acidic.

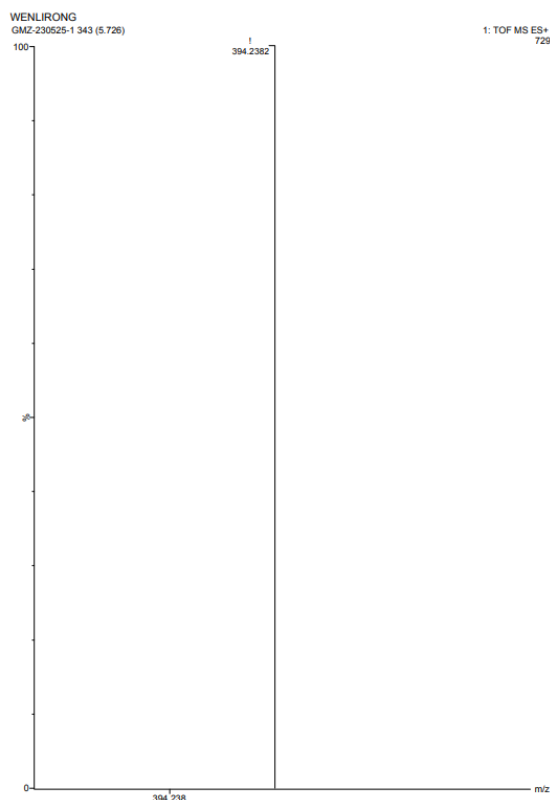
2) Control experiments



Scheme S2. Control experiments

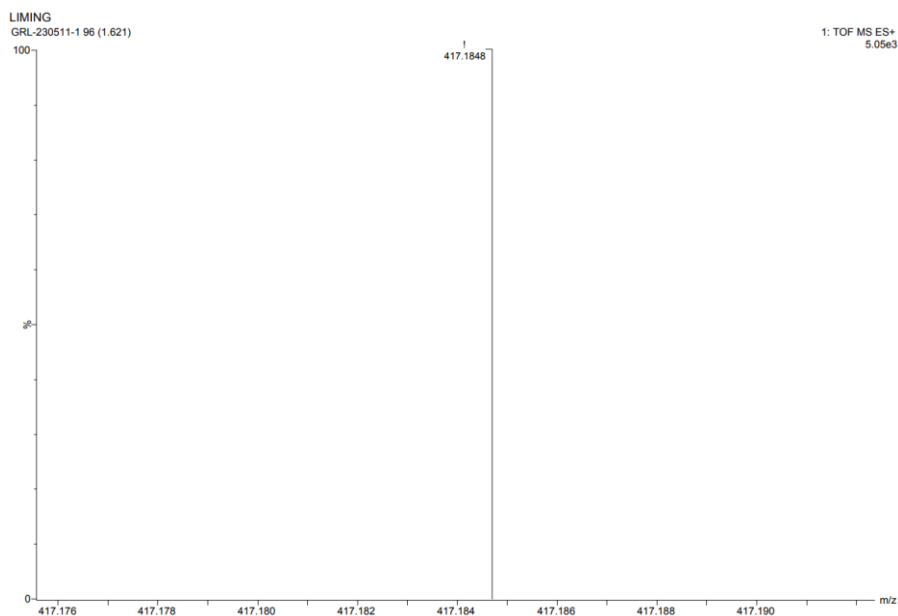
a) To the mixed solution of DCE (4.5 mL), HFIP (0.5 mL) and H₂O (0.1 mL), 1,4-enedione **1a** (0.1 mmol, 1.0 eq.), TEMPO (0.4 mmol, 4.0 eq.) and *n*-Bu₄NClO₄ (0.2 mmol, 2.0 eq.) were added. Equipped with graphite felt (10 × 10 × 5 mm) as anode and graphite felt (10 × 10 × 5 mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in Air for 2 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate. The target product was not obtained, and free radical capture products **47** detected under HRMS.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₂₅H₃₁NO₃ 394.2377, found 394.2382



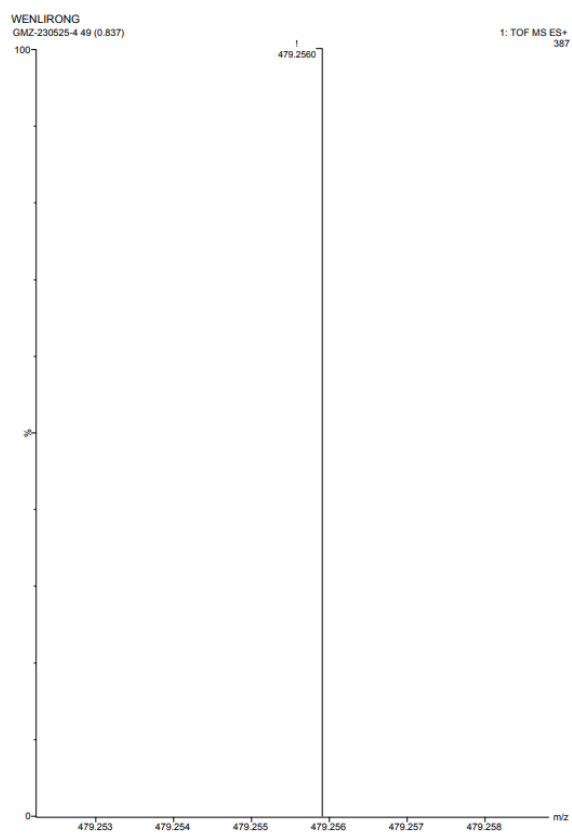
b) To the mixed solution of DCE (4.5 mL), HFIP (0.5 mL) and H₂O (0.1 mL), 1,4-enedione **1a** (0.1 mmol, 1.0 eq.), 1,1-diphenylethylene (0.4 mmol, 4.0 eq.) and *n*-Bu₄NClO₄ (0.2 mmol, 2.0 eq.) were added. Equipped with graphite felt (10 × 10 × 5 mm) as anode and graphite felt (10 × 10 × 5 mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in Air for 2 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate. The target product was not obtained, and free radical capture products **48** detected under HRMS.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₃₀H₂₄O₂ 417.1849, found 417.1848



c) To the mixed solution of DCE (4.5 mL), HFIP (0.5 mL) and H₂O (0.1 mL), 1,4-enedione **1a** (0.1 mmol, 1.0 eq.), BHT (0.4 mmol, 4.0 eq.) and *n*-Bu₄NClO₄ (0.2 mmol, 2.0 eq.) were added. Equipped with graphite felt (10 × 10 × 5 mm) as anode and graphite felt (10 × 10 × 5 mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in Air for 2 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate. The target product was not obtained and free radical capture product **49** was obtained in 40% yield. ¹H NMR (500 MHz, CDCl₃) δ 8.12 (d, *J* = 7.5 Hz, 2H), 7.83 (d, *J* = 7.1 Hz, 2H), 7.59 (t, *J* = 7.3 Hz, 1H), 7.54 – 7.50 (m, 3H), 7.42 (t, *J* = 7.7 Hz, 2H), 6.70 (d, *J* = 3.0 Hz, 1H), 6.48 (d, *J* = 3.0 Hz, 1H), 4.40 (dd, *J* = 10.0, 3.2 Hz, 1H), 3.55 (dd, *J* = 18.1, 9.9 Hz, 1H), 3.03 (dd, *J* = 18.1, 3.2 Hz, 1H), 1.20 (s, 9H), 1.18 (s, 9H), 1.16 (s, 3H).

HRMS (ESI-TOF, [M + Na⁺]): calcd for C₃₁H₃₆O₃ 479.2557, found 479.2560



d) DFIP detection in the system

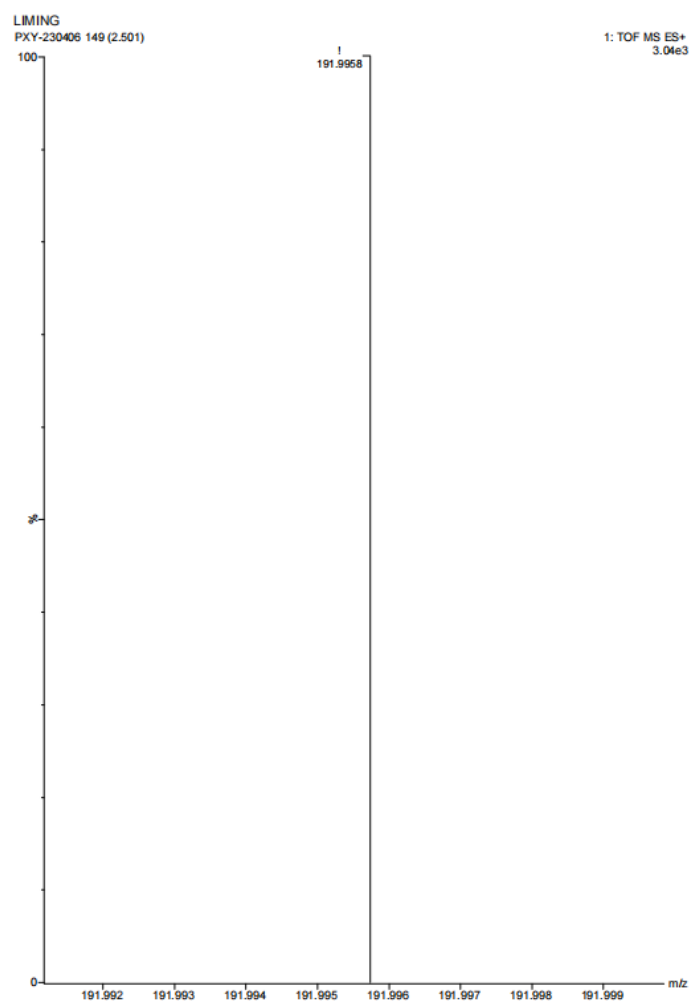
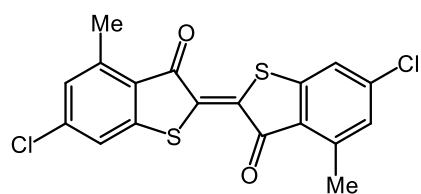


Figure S18. HRMS (ESI-TOF, [M + Na⁺]) of DFIP

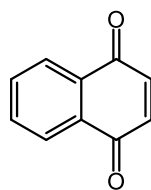
the organic phase was separated in the deuterium gram-scale reaction and perform HRMS testing to obtain [M + Na⁺] data for DFIP.

HRMS (ESI-TOF, [M + Na⁺]): calcd for C₃HDF₆O, 191.9965, found 191.9958

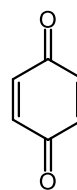
11. Unsuccessful substrates



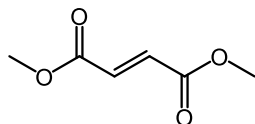
n.r.



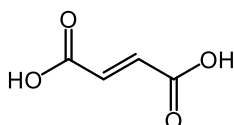
n.r.



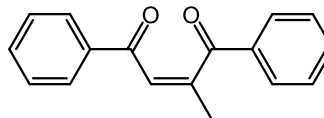
n.r.



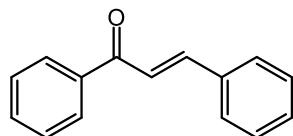
Trace



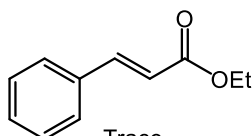
Trace



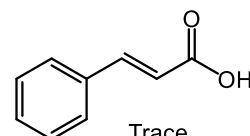
Trace



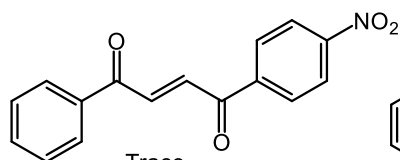
mass



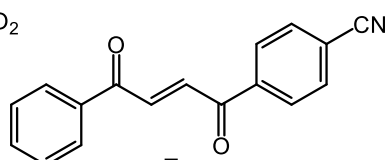
Trace



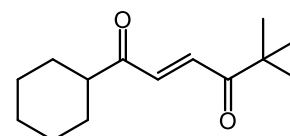
Trace



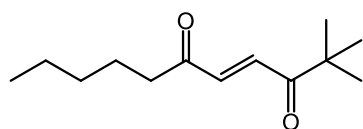
Trace



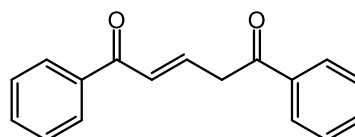
Trace



Trace

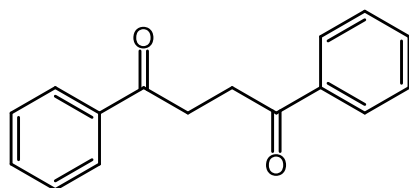


Trace



Trace

12. NMR spectra for the obtained compound



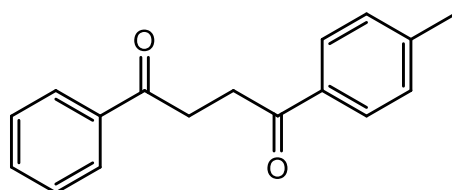
1b

22.9 mg, 96% yield. The spectra matched with the previous report.¹

¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 7.2 Hz, 4H), 7.58 (t, *J* = 7.0 Hz, 2H), 7.48 (t, *J* = 6.8 Hz, 4H), 3.47 (s, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 198.8, 136.9, 133.3, 128.7, 128.3, 32.7.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₄O₂ 239.1067, found 239.1070



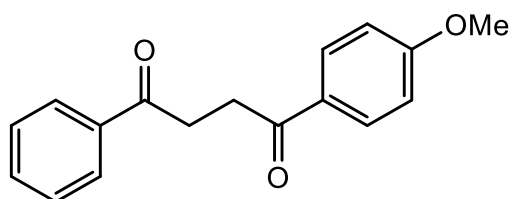
2b

24.0 mg, 95% yield. The spectra matched with the previous report.²

¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 7.6 Hz, 2H), 7.94 (d, *J* = 8.0 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 3.44 (d, *J* = 2.2 Hz, 4H), 2.42 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 198.8, 198.3, 143.9, 136.8, 134.3, 133.1, 129.3, 128.6, 128.2, 128.1, 32.6, 32.5, 21.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₆O₂ 253.1223, found 253.1227



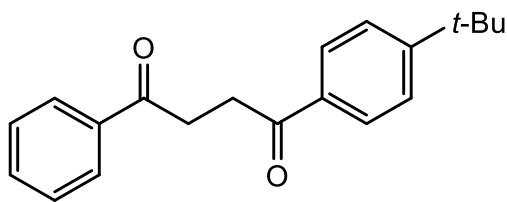
3b

20.9 mg, 78% yield. The spectra matched with the previous report.²

¹H NMR (500 MHz, CDCl₃) δ 8.03 (t, *J* = 8.6 Hz, 4H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 6.95 (d, *J* = 8.6 Hz, 2H), 3.87 (s, 3H), 3.52 – 3.16 (m, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 198.9, 197.2, 163.5, 136.8, 133.1, 130.4, 129.9, 128.6, 128.1, 113.7, 55.5, 32.7, 32.2.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₆O₃ 269.1172, found 269.1172



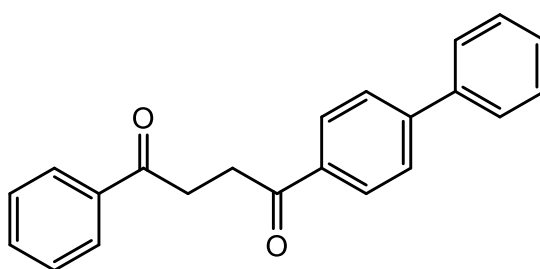
4b

28.3 mg, 96% yield. The spectra matched with the previous report.⁹

¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 7.7 Hz, 2H), 7.99 (d, *J* = 8.4 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.50 – 7.48 (m, 4H), 3.46 (s, 4H), 1.35 (s, 9H).

¹³C NMR (125 MHz, CDCl₃) δ 198.8, 198.4, 156.9, 136.8, 134.2, 133.1, 128.6, 128.1, 128.1, 125.5, 35.1, 32.7, 32.5, 31.1.

HRMS (ESI-TOF, [M + Na⁺]): calcd for C₂₀H₂₂O₂ 317.1512, found 317.1514



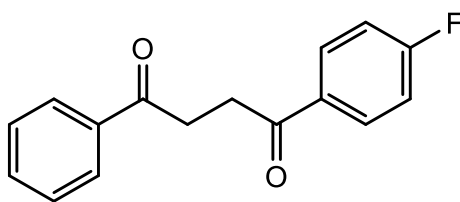
5b

23.6 mg, 75% yield. The spectra matched with the previous report.¹⁰

¹H NMR (500 MHz, CDCl₃) δ 8.15 (d, *J* = 8.2 Hz, 2H), 8.09 (d, *J* = 7.5 Hz, 2H), 7.74 (d, *J* = 8.1 Hz, 2H), 7.68 (d, *J* = 7.6 Hz, 2H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.52 (q, *J* = 7.4 Hz, 4H), 7.44 (t, *J* = 7.3 Hz, 1H), 3.53 (s, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 198.7, 198.3, 145.8, 139.9, 136.8, 135.5, 133.2, 129.0, 128.7, 128.6, 128.2, 128.1, 127.3, 127.3, 32.7, 32.6.

HRMS (ESI-TOF, [M + Na⁺]): calcd for C₂₂H₁₈O₂ 337.1199, found 337.1198



6b

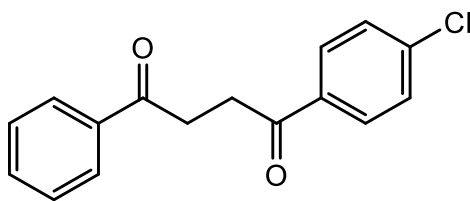
21.5 mg, 84 % yield. The spectra matched with the previous report.⁵

¹H NMR (500 MHz, CDCl₃) δ 8.10 – 8.01 (m, 4H), 7.58 (t, *J* = 7.1 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.15 (t, *J* = 8.5 Hz, 2H), 3.44 (dd, *J* = 14.7, 6.2 Hz, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 198.6, 197.1, 165.8 (d, *J* = 254.6 Hz), 136.7, 133.2 (d, *J* = 7.8 Hz), 133.2, 130.8 (d, *J* = 9.4 Hz), 128.6, 128.1, 115.7 (d, *J* = 22.0 Hz), 32.6, 32.4.

¹⁹F NMR (376 MHz, CDCl₃) δ -105.24.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₃FO₂ 257.0972, found 257.0974



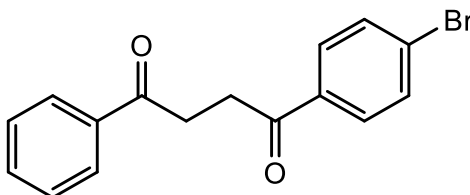
7b

26.2 mg, 96% yield. The spectra matched with the previous report.⁵

¹H NMR (500 MHz, CDCl₃) δ 8.03 (d, *J* = 7.7 Hz, 2H), 7.97 (d, *J* = 7.6 Hz, 2H), 7.57 (d, *J* = 7.2 Hz, 1H), 7.51 – 7.42 (m, 4H), 3.45 (d, *J* = 4.3 Hz, 2H), 3.42 (d, *J* = 5.8 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 198.5, 197.5, 139.6, 136.7, 135.1, 133.2, 129.6, 128.9, 128.6, 128.1, 32.6, 32.5.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₃ClO₂ 273.0677, found 273.0675



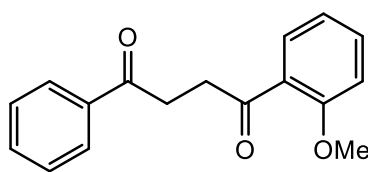
8b

29.8 mg, 94% yield. The spectra matched with the previous report.⁷

¹H NMR (500 MHz, CDCl₃) δ 8.03 (d, *J* = 7.7 Hz, 2H), 7.90 (d, *J* = 8.1 Hz, 2H), 7.62 (d, *J* = 8.6 Hz, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 3.45 (d, *J* = 5.9 Hz, 2H), 3.41 (d, *J* = 5.5 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 198.5, 197.7, 136.7, 135.5, 133.2, 131.9, 129.7, 128.6, 128.3, 128.1, 32.5, 32.5.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₃BrO₂ 317.0172, found 317.0172



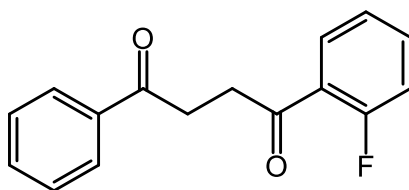
9b

23.6 mg, 88 % yield.

¹H NMR (500 MHz, CDCl₃) δ 8.03 (d, *J* = 7.6 Hz, 2H), 7.77 (d, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 3H), 7.04 – 6.95 (m, 2H), 3.92 (s, 3H), 3.46 (d, *J* = 6.0 Hz, 2H), 3.42 (d, *J* = 6.0 Hz, 2H).

¹³C NMR (125MHz, CDCl₃) δ 200.6, 199.1, 158.8, 137.0, 133.6, 133.0, 130.5, 128.6, 128.1, 127.9, 120.7, 111.6, 55.5, 38.0, 33.0.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₆O₃ 269.1172, found 269.1178



10b

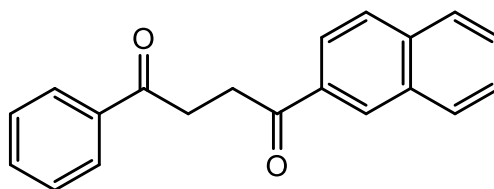
22.0 mg, 86% yield. The spectra matched with the previous report.⁷

¹H NMR (500 MHz, CDCl₃) δ 8.16 – 7.97 (m, 2H), 7.90 (td, *J* = 7.6, 1.9 Hz, 1H), 7.61 – 7.48 (m, 4H), 7.26 – 7.19 (m, 1H), 7.19 – 7.09 (m, 1H), 3.45 (d, *J* = 2.0 Hz, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 198.6, 197.1 (d, *J* = 4.0 Hz), 162.1 (d, *J* = 254.8 Hz), 136.9, 134.7 (d, *J* = 9.0 Hz), 133.2, 130.8 (d, *J* = 2.6 Hz), 128.7, 128.2, 125.6, 125.5, 124.5 (d, *J* = 3.4 Hz), 116.7 (d, *J* = 23.9 Hz), 37.5, 37.5, 32.7 (d, *J* = 2.2 Hz).

¹⁹F NMR (376 MHz, CDCl₃) δ -108.96.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₃FO₂ 257.0972, found 257.0976



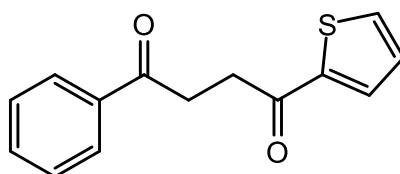
11b

19.3 mg, 67% yield. The spectra matched with the previous report.⁵

¹H NMR (500 MHz, CDCl₃) δ 8.59 (s, 1H), 8.08 (t, *J* = 9.3 Hz, 3H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.93 – 7.85 (m, 2H), 7.63 – 7.53 (m, 3H), 7.49 (t, *J* = 7.6 Hz, 2H), 3.61 (t, *J* = 6.3 Hz, 2H), 3.52 (t, *J* = 6.4 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 198.8, 198.6, 136.8, 135.7, 134.1, 133.2, 132.6, 129.9, 129.6, 128.6, 128.5, 128.2, 127.8, 126.8, 123.9, 32.7, 32.7.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₂₀H₁₆O₂ 289.1223, found 289.1224



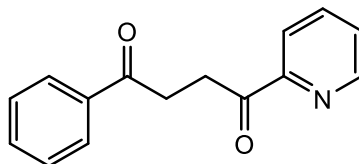
12b

13.2 mg, 54% yield. The spectra matched with the previous report.⁵

¹H NMR (500 MHz, CDCl₃) δ 8.02 (d, *J* = 7.7 Hz, 2H), 7.83 (d, *J* = 3.8 Hz, 1H), 7.64 (d, *J* = 5.0 Hz, 1H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.15 (t, *J* = 4.4 Hz, 1H), 3.45 (d, *J* = 5.9 Hz, 2H), 3.41 (d, *J* = 5.8 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 198.6, 191.7, 144.1, 136.8, 133.7, 133.3, 132.2, 128.8, 128.3, 33.3, 32.8.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₄H₁₂O₂S 245.0631, found 245.0631



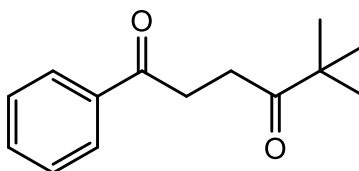
13b

15.3 mg, 64% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.71 (d, *J* = 4.7 Hz, 1H), 8.04 (t, *J* = 6.7 Hz, 3H), 7.83 (t, *J* = 7.5 Hz, 1H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 3H), 3.69 (t, *J* = 6.3 Hz, 2H), 3.47 (t, *J* = 6.3 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 198.5, 196.6, 151.3, 147.0, 134.9, 134.8, 131.0, 126.5, 126.1, 125.1, 119.8, 30.7, 30.0.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₅H₁₃NO₂ 240.1019, found 240.1019



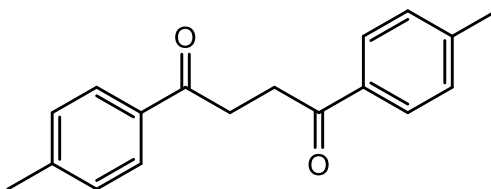
14b

18.8 mg, 86% yield. The spectra matched with the previous report.⁷

¹H NMR (500 MHz, CDCl₃) δ 7.98 (d, *J* = 7.0 Hz, 2H), 7.54 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 3.24 (t, *J* = 6.3 Hz, 2H), 2.96 (t, *J* = 6.3 Hz, 2H), 1.21 (s, 9H).

¹³C NMR (125 MHz, CDCl₃) δ 214.7, 199.0, 136.9, 133.1, 128.6, 128.1, 44.1, 32.4, 30.8, 26.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₄H₁₈O₂ 219.1380, found 219.1383



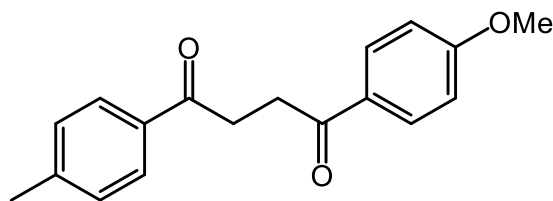
15b

20.8 mg, 78 % yield. The spectra matched with the previous report.⁴

¹H NMR (500 MHz, CDCl₃) δ 7.97 (d, *J* = 8.0 Hz, 4H), 7.30 (d, *J* = 8.1 Hz, 4H), 3.46 (s, 4H), 2.45 (s, 6 H).

¹³C NMR (125 MHz, CDCl₃) δ 198.5, 143.9, 134.3, 129.3, 128.3, 32.5, 21.7.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₈H₁₈O₂ 267.1380, found 267.1380



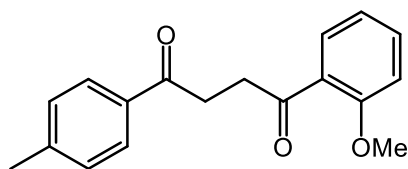
16b

22.0 mg, 78% yield. The spectra matched with the previous report.⁴

¹H NMR (500 MHz, CDCl₃) δ 8.02 (d, *J* = 8.7 Hz, 2H), 7.94 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 6.97 – 6.92 (m, 2H), 3.87 (s, 3H), 3.46 – 3.37 (m, 4H), 2.41 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 198.5, 197.3, 163.5, 143.9, 134.4, 130.4, 130.0, 129.3, 128.2, 113.7, 55.5, 32.6, 32.3, 21.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₈H₁₈O₃ 283.1329, found 283.1331



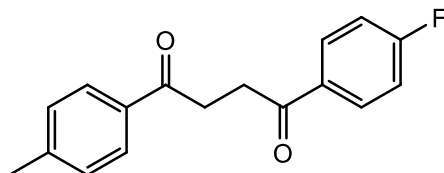
17b

22.0 mg, 78% yield.

¹H NMR (500 MHz, CDCl₃) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.79 – 7.74 (m, 1H), 7.49 – 7.43 (m, 1H), 7.29 – 7.24 (m, 2H), 7.04 – 6.95 (m, 2H), 3.92 (s, 3H), 3.44 (d, *J* = 6.2 Hz, 2H), 3.39 (d, *J* = 5.8 Hz, 2H), 2.41 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 200.8, 198.7, 158.8, 143.7, 134.5, 133.5, 130.5, 129.2, 128.2, 128.0, 120.6, 111.6, 55.5, 38.0, 32.9, 21.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₈H₁₈O₃ 283.1329, found 283.1327



18b

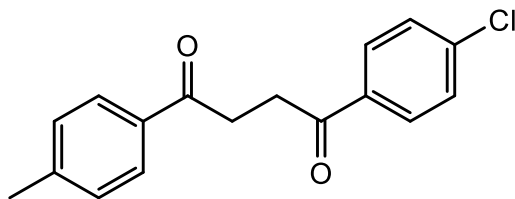
19.2 mg, 71% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.13 – 8.06 (m, 2H), 7.96 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.17 (t, *J* = 8.6 Hz, 2H), 3.46 – 3.44 (m, 4H), 2.44 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 196.2, 195.2, 163.8 (d, *J* = 254.5 Hz), 142.0, 132.3, 131.3 (d, *J* = 3.1 Hz), 128.7 (d, *J* = 9.4 Hz), 127.3, 126.2, 113.6 (d, *J* = 21.9 Hz), 30.5, 19.6.

¹⁹F NMR (376 MHz, CDCl₃) δ -105.33.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₅FO₂ 271.1129, found 271.1130



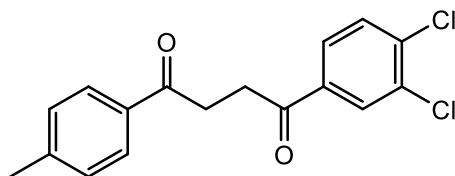
19b

22.1 mg, 77% yield. The spectra matched with the previous report.⁸

¹H NMR (500 MHz, CDCl₃) δ 7.98 (d, *J* = 8.5 Hz, 2H), 7.93 (d, *J* = 8.2 Hz, 2H), 7.45 (d, *J* = 8.6 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 3.47 – 3.37 (m, 4H), 2.42 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 197.1, 196.6, 143.0, 138.5, 134.1, 133.2, 128.5, 128.3, 127.9, 127.2, 31.5, 31.4, 20.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₅ClO₂ 287.0833, found 287.0830



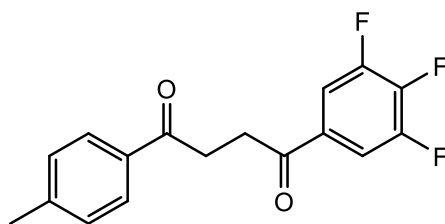
20b

26.3 mg, 82% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.11 (d, *J* = 2.0 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 2H), 7.86 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.44 (d, *J* = 6.2 Hz, 2H), 3.38 (d, *J* = 6.2 Hz, 2H), 2.42 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 197.9, 196.7, 144.1, 137.7, 136.4, 134.1, 133.3, 130.7, 130.2, 129.3, 128.2, 127.2, 32.6, 32.4, 21.7.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₄Cl₂O₂ 321.0444, found 321.0444



21b

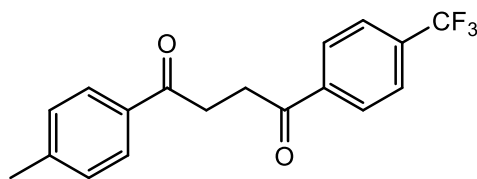
25.7 mg, 84% yield.

¹H NMR (500 MHz, CDCl₃) δ 7.91 (d, *J* = 8.0 Hz, 2H), 7.68 (t, *J* = 7.1 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.45 (t, *J* = 6.1 Hz, 2H), 3.33 (t, *J* = 6.2 Hz, 2H), 2.42 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 197.7, 195.4, 152.2 (d, *J* = 10.9 Hz), 150.2 (d, *J* = 7.0 Hz), 144.2, 142.2 (d, *J* = 15.5 Hz), 134.0, 132.4 (d, *J* = 4.9 Hz), 129.3, 128.2, 125.6, 112.8 (d, *J* = 5.4 Hz), 112.6 (d, *J* = 5.3 Hz), 32.5, 32.3, 21.7.

¹⁹F NMR (376 MHz, CDCl₃) δ -137.01, -137.07, -157.18, -157.24, -157.29.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₃F₃O₂ 307.0940, found 307.0946



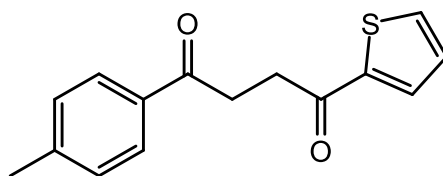
22b

22.4 mg, 70% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.14 (d, *J* = 8.0 Hz, 2H), 7.93 (d, *J* = 7.9 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.47 – 3.45 (m, 4H), 2.42 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 198.0, 144.1, 139.5, 134.5, 134.3, 134.1, 129.3, 128.5, 128.2, 125.7, 125.7, 124.7, 122.5, 32.8, 32.5, 21.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₈H₁₅F₃O₂ 321.1097, found 321.1096



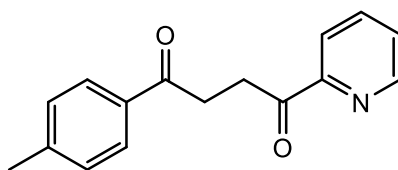
23b

17.6 mg, 68% yield. The spectra matched with the previous report.⁵

¹H NMR (500 MHz, CDCl₃) δ 7.95 (d, *J* = 8.0 Hz, 2H), 7.86 (d, *J* = 3.8 Hz, 1H), 7.66 (d, *J* = 5.0 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.17 (t, *J* = 4.4 Hz, 1H), 3.44 (dd, *J* = 16.9, 5.5 Hz, 4H), 2.44 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 198.1, 191.8, 144.0, 144.0, 134.2, 133.5, 132.0, 129.3, 128.2, 128.1, 33.2, 32.5, 21.7.

HRMS (ESI-TOF, [M + H⁺]): calcd for, found C₁₅H₁₄O₂S 259.0787, found 259.0787



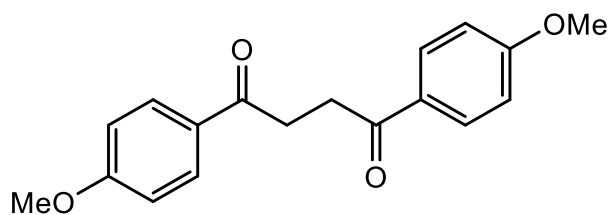
24b

22.0 mg, 87% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.71 (d, *J* = 4.4 Hz, 1H), 8.04 (d, *J* = 7.8 Hz, 1H), 7.93 (d, *J* = 8.2 Hz, 2H), 7.85 – 7.82 (m, 1H), 7.49 – 7.46 (m, 1H), 7.27 (d, *J* = 5.8 Hz, 2H), 3.68 (t, *J* = 6.3 Hz, 2H), 3.45 (t, *J* = 6.3 Hz, 2H), 2.42 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 200.6, 198.3, 153.3, 149.0, 143.8, 136.8, 134.4, 129.2, 128.2, 127.1, 121.8, 32.6, 32.0, 21.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₅NO₂ 254.1176, found 254.1176



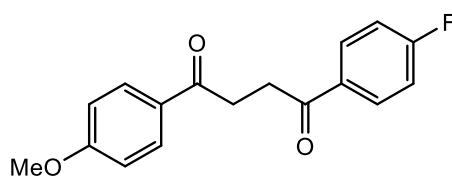
25b

19.7 mg, 66 % yield. The spectra matched with the previous report.³

¹H NMR (500 MHz, CDCl₃) δ 8.02 (d, *J* = 8.5 Hz, 4H), 6.95 (d, *J* = 8.5 Hz, 4H), 3.87 (s, 6H), 3.40 (s, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 197.4, 163.5, 130.4, 130.0, 113.7, 55.5, 32.3.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₈H₁₈O₄ 299.1278, found 299.1282



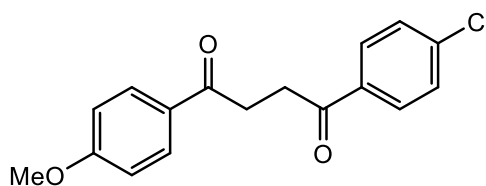
26b

22.0 mg, 77% yield. The spectra matched with the previous report.⁴

¹H NMR (500 MHz, CDCl₃) δ 8.09 – 8.04 (m, 2H), 8.01 (d, *J* = 8.9 Hz, 2H), 7.18 – 7.10 (m, 2H), 6.99 – 6.90 (m, 2H), 3.87 (s, 3H), 3.41 (d, *J* = 2.4 Hz, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 197.3, 197.1, 166.8, 164.8, 163.6, 133.3, 130.8, 130.7, 130.4, 129.8, 115.8, 115.6, 113.8, 55.5, 32.5, 32.2.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₅FO₃ 287.1078, found 287.1078



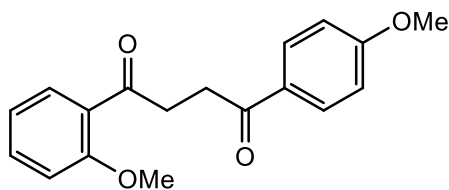
27b

22.4 mg, 74% yield. The spectra matched with the previous report.⁴

¹H NMR (500 MHz, CDCl₃) δ 8.02 – 7.97 (m, 4H), 7.45 (d, *J* = 8.6 Hz, 2H), 6.95 (d, *J* = 9.0 Hz, 2H), 3.88 (s, 3H), 3.42 – 3.40 (m, 1H).

¹³C NMR (125 MHz, CDCl₃) δ 197.7, 197.0, 163.6, 139.5, 135.2, 130.4, 129.8, 129.6, 128.9, 113.8, 55.5, 32.6, 32.2.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₅ClO₃ 303.0782, found 303.0785



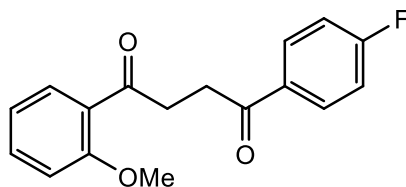
28b

23.0 mg, 77% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.01 (d, *J* = 8.8 Hz, 2H), 7.77 – 7.75 (m, 1H), 7.48 – 7.44 (m, 1H), 7.03 – 6.91 (m, 4H), 3.91 (s, 3H), 3.86 (s, 3H), 3.44 (t, *J* = 6.3 Hz, 2H), 3.36 (t, *J* = 6.1 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 200.9, 197.6, 163.4, 158.8, 133.5, 130.5, 130.4, 130.1, 128.0, 120.6, 113.7, 111.6, 55.5, 55.5, 38.0, 32.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₈H₁₈O₄ 299.1278, found 299.1282



29b

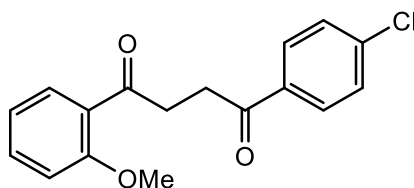
21.8 mg, 76% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.07 – 8.04 (m, 2H), 7.77 – 7.75 (m, 1H), 7.49 – 7.45 (m, 1H), 7.13 (t, *J* = 8.6 Hz, 2H), 7.02 – 6.97 (m, 2H), 3.92 (s, 3H), 3.46 (t, *J* = 6.3 Hz, 2H), 3.37 (t, *J* = 6.3 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 200.5, 197.5, 165.7 (d, *J* = 254.3 Hz), 158.9, 133.7, 133.4 (d, *J* = 3.1 Hz), 130.7 (d, *J* = 9.4 Hz), 130.5, 127.7, 120.7, 115.6 (d, *J* = 21.8 Hz), 111.6, 55.5, 37.9, 32.9.

¹⁹F NMR (376 MHz, CDCl₃) δ -104.51.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₅FO₃ 287.1078, found 287.1180



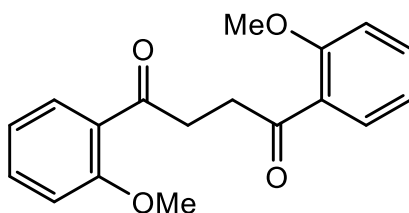
30b

23.0 mg, 76% yield.

¹H NMR (500 MHz, CDCl₃) δ 7.97 (d, *J* = 8.5 Hz, 2H), 7.77 – 7.75 (m, 1H), 7.51 – 7.45 (m, 1H), 7.44 (d, *J* = 8.5 Hz, 2H), 7.04 – 6.95 (m, 2H), 3.92 (s, 3H), 3.46 (t, *J* = 6.3 Hz, 2H), 3.36 (t, *J* = 6.4 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 200.4, 197.9, 158.9, 139.4, 135.3, 133.7, 130.5, 129.6, 128.9, 127.7, 120.7, 111.6, 55.5, 37.9, 32.9.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₇H₁₅ClO₃ 303.0782, found 303.0792



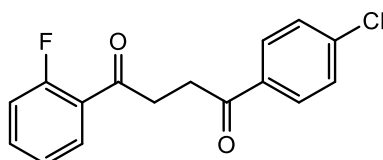
31b

26.0 mg, 87 % yield. The spectra matched with the previous report.¹²

¹H NMR (500 MHz, CDCl₃) δ 7.75 (d, *J* = 7.7 Hz, 2H), 7.46 (t, *J* = 7.1 Hz, 2H), 6.99 (q, *J* = 9.6, 9.1 Hz, 4H), 3.91 (s, 6H), 3.41 (s, 4H).

¹³C NMR (125MHz, CDCl₃) δ 201.2, 158.7, 133.4, 130.4, 128.2, 120.6, 111.5, 55.5, 38.3.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₈H₁₈O₄ 299.1278, found 299.1281



32b

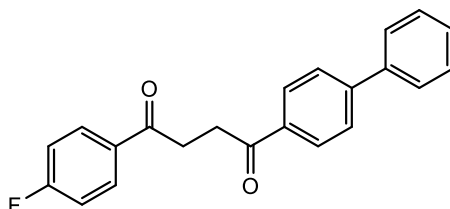
20.9 mg, 76% yield. The spectra matched with the previous report.¹³

¹H NMR (500 MHz, CDCl₃) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.91 – 7.88 (m, 1H), 7.57 – 7.49 (m, 1H), 7.45 (d, *J* = 8.4 Hz, 1H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.16 (dd, *J* = 11.3, 8.3 Hz, 1H), 3.45 (dd, *J* = 6.3, 3.2 Hz, 1H), 3.41 (d, *J* = 5.9 Hz, 1H).

¹³C NMR (125 MHz, CDCl₃) δ 197.3, 196.8 (d, *J* = 4.1 Hz), 162.1 (d, *J* = 254.9 Hz), 139.5, 135.1, 134.7 (d, *J* = 9.1 Hz), 130.7 (d, *J* = 2.7 Hz), 129.6, 128.9, 125.4, 125.2, 124.5 (d, *J* = 3.4 Hz), 116.7 (d, *J* = 23.8 Hz), 37.4 (d, *J* = 8.5 Hz), 32.5.

¹⁹F NMR (376 MHz, CDCl₃) δ -108.87.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₂ClFO₂ 291.0583, found 291.0583



33b

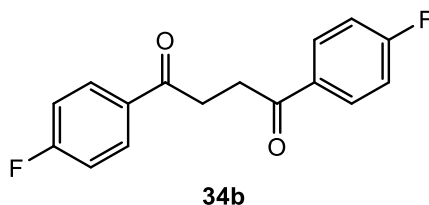
25.3 mg, 76% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.12 – 8.06 (m, 4H), 7.71 (d, *J* = 8.1 Hz, 2H), 7.64 (d, *J* = 7.2 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.3 Hz, 1H), 7.15 (t, *J* = 8.5 Hz, 2H), 3.48 (d, *J* = 5.5 Hz, 2H), 3.45 (d, *J* = 5.4 Hz, 2H).

^{13}C NMR (125 MHz, CDCl_3) δ 198.2, 197.1, 165.8 (d, $J = 254.7$ Hz), 145.9, 139.9, 135.4, 133.3 (d, $J = 3.1$ Hz), 130.8 (d, $J = 9.2$ Hz), 129.0, 128.7, 128.3, 127.3 (d, $J = 2.4$ Hz), 115.7 (d, $J = 21.9$ Hz), 32.6, 32.5.

^{19}F NMR (376 MHz, CDCl_3) δ -105.22.

HRMS (ESI-TOF, $[\text{M} + \text{H}^+]$): calcd for $\text{C}_{22}\text{H}_{17}\text{FO}_2$ 333.1285, found 333.1289



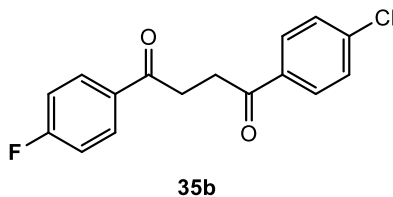
22.5 mg, 82% yield. The spectra matched with the previous report.⁴

^1H NMR (500 MHz, CDCl_3) δ 8.07 – 8.05 (m, 4H), 7.15 (t, $J = 8.5$ Hz, 4H), 3.42 (s, 4H).

^{13}C NMR (125 MHz, CDCl_3) δ 197.0, 165.8 (d, $J = 254.9$ Hz), 133.2, 130.8 (d, $J = 9.4$ Hz), 115.7 (d, $J = 21.9$ Hz), 32.4.

^{19}F NMR (376 MHz, CDCl_3) δ -105.11.

HRMS (ESI-TOF, $[\text{M} + \text{H}^+]$): calcd for $\text{C}_{16}\text{H}_{12}\text{F}_2\text{O}_2$ 275.0878, found 275.0878



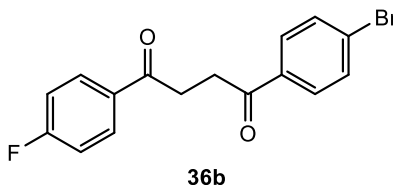
23.3 mg, 80% yield.

^1H NMR (500 MHz, CDCl_3) δ 8.07 – 8.04 (m, 2H), 7.97 (d, $J = 8.6$ Hz, 2H), 7.45 (d, $J = 8.6$ Hz, 2H), 7.15 (t, $J = 8.6$ Hz, 2H), 3.42 (s, 4H).

^{13}C NMR (125 MHz, CDCl_3) δ 197.4, 196.9, 165.9 (d, $J = 254.8$ Hz), 139.7, 135.0, 133.1, 130.8 (d, $J = 9.2$ Hz), 129.5, 128.9, 115.7 (d, $J = 22.0$ Hz), 32.5, 32.4.

^{19}F NMR (376 MHz, CDCl_3) δ -105.06.

HRMS (ESI-TOF, $[\text{M} + \text{H}^+]$): calcd for $\text{C}_{16}\text{H}_{12}\text{ClFO}_2$ 291.0583, found 291.0588



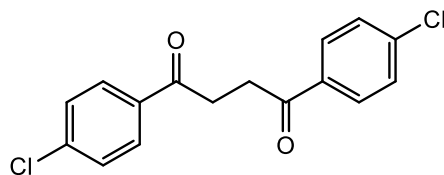
29.8 mg, 89% yield.

^1H NMR (500 MHz, CDCl_3) δ 8.05 (dd, $J = 8.7, 5.6$ Hz, 2H), 7.89 (d, $J = 8.5$ Hz, 2H), 7.62 (d, $J = 8.6$ Hz, 2H), 7.14 (t, $J = 8.6$ Hz, 2H), 3.41 (s, 4H).

^{13}C NMR (125 MHz, CDCl_3) δ 197.6, 196.9, 165.9 (d, $J = 254.8$ Hz), 135.4, 133.1 (d, $J = 3.1$ Hz), 131.9, 130.8 (d, $J = 9.2$ Hz), 129.7, 128.4, 115.7 (d, $J = 22.0$ Hz), 32.5, 32.4.

^{19}F NMR (376 MHz, CDCl_3) δ -105.04.

HRMS (ESI-TOF, $[\text{M} + \text{H}^+]$): calcd for $\text{C}_{16}\text{H}_{12}\text{BrFO}_2$ 335.0077, found 335.0076



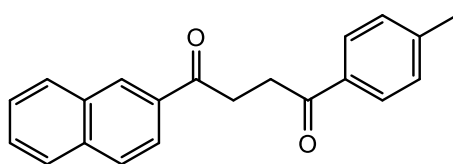
37b

29.5 mg, 96% yield. The spectra matched with the previous report.⁴

¹H NMR (500 MHz, CDCl₃) δ 7.97 (d, *J* = 8.6 Hz, 4H), 7.45 (d, *J* = 8.6 Hz, 4H), 3.42 (s, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 197.3, 139.7, 135.0, 129.5, 129.0, 32.5.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₂Cl₂O₂ 307.0287, found 307.0287



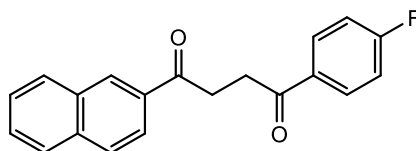
38b

21.2 mg, 70% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.59 (s, 1H), 8.08 (d, *J* = 8.4 Hz, 1H), 7.99 – 7.96 (m, 3H), 7.90 (dd, *J* = 12.4, 8.3 Hz, 2H), 7.58 (dt, *J* = 22.7, 7.0 Hz, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 3.60 (t, *J* = 6.4 Hz, 2H), 3.50 (t, *J* = 6.4 Hz, 2H), 2.43 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 198.8, 198.4, 144.0, 135.7, 134.3, 134.2, 132.6, 129.9, 129.6, 129.3, 128.4, 128.3, 127.8, 126.7, 123.9, 32.7, 32.6, 21.7.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₂₁H₁₈O₂ 303.1380, found 303.1383



39b

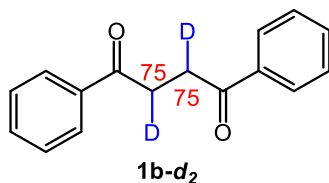
20.5 mg, 67% yield.

¹H NMR (500 MHz, CDCl₃) δ 8.59 (s, 1H), 8.11 – 8.07 (m, 3H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.92 – 7.88 (m, 2H), 7.63 – 7.55 (m, 2H), 7.16 (t, *J* = 8.6 Hz, 2H), 3.61 (t, *J* = 6.3 Hz, 2H), 3.48 (t, *J* = 6.3 Hz, 2H).

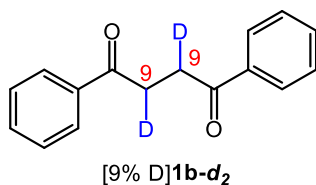
¹³C NMR (125 MHz, CDCl₃) δ 198.5, 197.2, 165.8 (d, *J* = 254.8 Hz), 135.7, 134.0, 133.2, 132.5, 130.8 (d, *J* = 9.3 Hz), 129.9, 129.6, 128.5 (d, *J* = 3.3 Hz), 127.8, 126.8, 123.8, 115.7 (d, *J* = 21.9 Hz), 32.7, 32.6.

¹⁹F NMR (376 MHz, CDCl₃) δ -105.20.

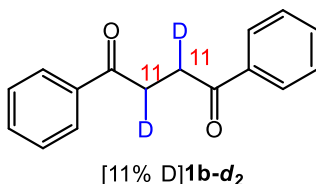
HRMS (ESI-TOF, [M + H⁺]): calcd for C₂₀H₁₅FO₂ 307.1129, found 307.1133



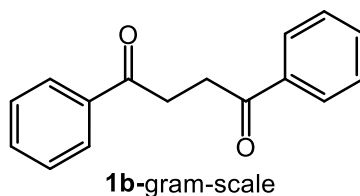
¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 7.2 Hz, 4H), 7.58 (t, *J* = 7.4 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 4H), 3.47 – 3.43 (m, 2.66H).



¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 7.8 Hz, 4H), 7.58 (t, *J* = 7.4 Hz, 2H), 7.48 (t, *J* = 7.4 Hz, 4H), 3.47 (s, 3.83H).



¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 7.4 Hz, 4H), 7.58 (t, *J* = 7.4 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 4H), 3.47 (s, 3.79H).



¹H NMR (500 MHz, CDCl₃) δ 8.08 (d, *J* = 7.4 Hz, 4H), 7.61 (t, *J* = 7.3 Hz, 2H), 7.52 (t, *J* = 7.8 Hz, 4H), 3.51 (s, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 198.7, 136.8, 133.2, 128.6, 128.1, 32.6.

HRMS (ESI-TOF, [M + H⁺]): calcd for C₁₆H₁₄O₂ 239.1067, found 239.1066

13. References

- 1 X. Zhu, Y. Liu, C. Liu, H. Yang and H. Fu, *Green Chem.*, 2020, **22**, 4357–4363
- 2 Y. Dong, R. Li, J. Zhou, and Z. Sun, *Org. Lett.*, 2021, **23**, 6387–6390
- 3 P. Mizar and T. Wirth, *Angew. Chem. Int. Ed.*, 2014, **53**, 5993–5997
- 4 L. Xu, X. Liu, G. R. Alvey, A. Shatskiy, J. Q. Liu, M. D. Karkas and X. S. Wang, *Org. Lett.*, 2022, **24**, 4513–4518
- 5 L. H. Wang and J. Zhao, *Eur. J. Org. Chem.*, 2018, **31**, 4345–4348

- 6 J. Xuan, Z. J. Feng, J. R. Chen, L. Q. Lu and W. J. Xiao, *Chem. Eur. J.*, 2014, **20**, 3045 – 3049
- 7 Y. Liu, S. Liu, D. Li, N. Zhang, L. Peng, J. Ao, C. E. Song, Y. Lan and H. L. Yan, *J. Am. Chem. Soc.*, 2019, **141**, 1150–1159
- 8 Z. L. Shen, K. Goh, H. L. Cheong, C. Wong, Y. C. Lai, Y. S. Yang, and T.P. Loh, *J. Am. Chem. Soc.*, 2010, **132**, 15852–15855
- 9 D. Ba, S. Wen, Q. Tian, Y. Chen, W. W. Lv and G. Cheng, *Nat. Commun.*, 2020, **11**, 4219-4225
- 10 W. Kong, C. Yu, H. An, and Q. Song, *Org. Lett.*, 2018, **20**, 349–352
- 11 S. R. Chowdhury, D. Singh, I. U. Hoque, and S. Maity, *J. Org. Chem.*, 2020, **85**, 13939–13950
- 12 D. H. Yu, W. P. To, G. S. Mingtong, L. L. Wu, K.T. Chan, L. L. Du, D. L. Phillips, Y. G. Liu and C. M. Che, *Chem. Sci.*, 2020, **11**, 6370–6382
- 13 Y. Zhang, X. Li, L. Xu, X. Xie, Y. Lu and Z. Zhang, *Org. Lett.*, 2022, **24**, 7512–7516
- 14 Q. Liu, R.-G. Wang, H.-J. Song, Y.-X. Liu and Q.-M. Wang, *Adv. Synth. Catal.*, 2020, **362**, 4391-4396
- 15 J. A. Clement, A. K. Mohanakrishnan, *Tetrahedron*, 2010, **66**, 2340-2350
- 16 M. Periasamy, M. Seenivasaperumal, V. D. Rao, *Synthesis*, 2003, **16**, 2507-2510
- 17 W.-L. Xu, W. Hu, W.-M. Zhao, M. Wang, J. Chen, and L. Zhou, *Org. Lett.*, 2020, **22**, 7169–7174
- 18 P. A. Cranwell and J. E. Saxton, *J. Chem. Soc.*, 1962, 3482-3487

The completed references in manuscript

Other citations of ref. 2 in the manuscript: (d) B. K. Peters, T. Zhou, J. Rujirawanich, A. Cadu, T. Singh, W. Rabten, S. Kerdphon and P. G. Andersson, *J. Am. Chem. Soc.*, 2014, **136**, 16557-16562; (e) K. Tokmic, C. R. Markus, L. Zhu and A. R. Fout, *J. Am. Chem. Soc.*, 2016, **138**, 11907-11913; (f) S. A. Green, S. W. M. Crossley, J. L. M. Matos, S. Vasquez-Cespedes, S. L. Shevick, and R. A. Shenvi, *Acc. Chem. Res.*, 2018, **51**, 2628-2640.

Other citations of ref. 6 in the manuscript: (d) T. Amaya, Y. Maegawa, T. Masuda, Y. Osafune and T. Hirao, *J. Am. Chem. Soc.*, 2015, **137**, 10072-10075; (e) C. Liu, Y. Deng, J. Wang, Y. Yang, S. Tang and A. Lei, *Angew. Chem. Int. Ed.*, 2011, **50**, 7337-7341; (f) P. Setzer, A. Beauseigneur, M. S. M. Pearson-Long and P. Bertus, *Angew. Chem. Int. Ed.*, 2010, **49**, 8691-8694; (g) H. Stetter, *Angew. Chem. Int. Ed.*, 1976, **15**, 639-647; (h) M. M. D. Wilde and M. Gravel, *Angew. Chem. Int. Ed.*, 2013, **52**, 12651-12654.

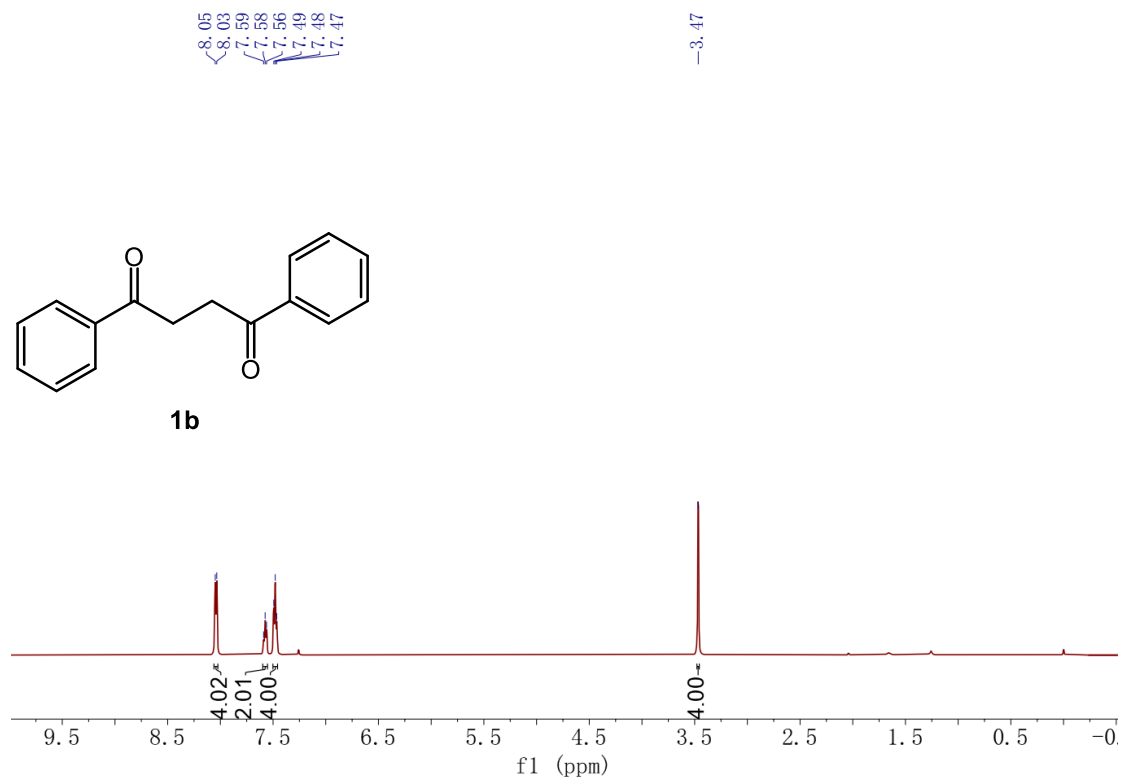
Other citations of ref. 7 in the manuscript: (c) D.-M. Cui, M. Kawamura, S. Shimada, T. Hayashi and M. Tanaka, *Tetrahedron Lett.*, 2003, **44**, 4007-4010; (d) Y. Y. Cheng, J. X. Yu, T. Lei, H. Y. Hou, B. Chen, C. H. Tung and L. Z. Wu, *Angew. Chem. Int. Ed.*, 2021, **60**, 26822-26828; (e) L. Wang, J. Sun, J. Xia, M. Li, L. Zhang, R. Ma, G. Zheng and Q. Zhang, *Sci. China Chem.*, 2022, **65**, 1938-1944.

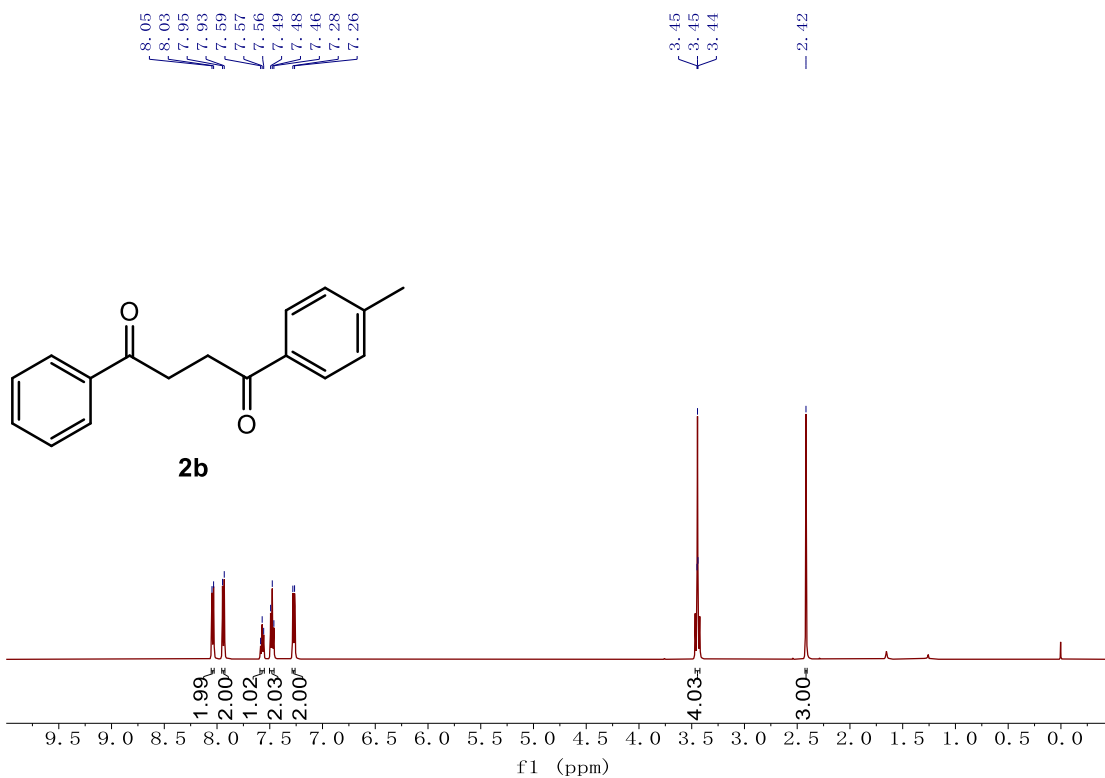
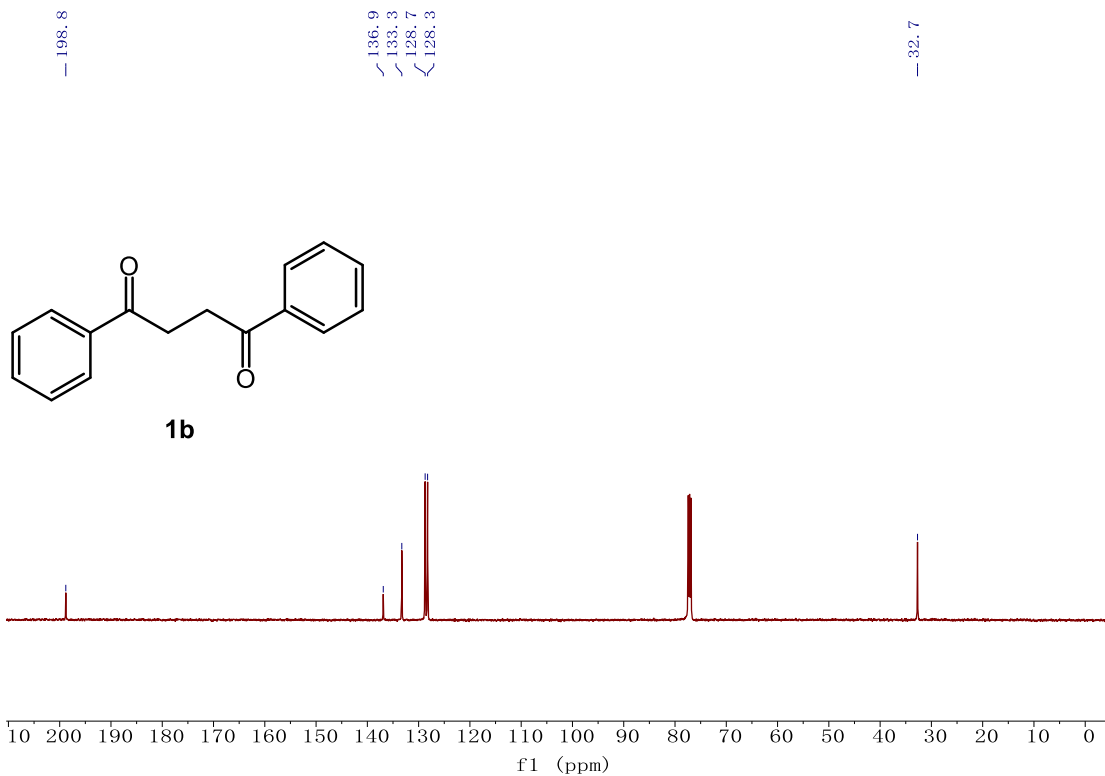
Other citations of ref. 8 in the manuscript: (d) K. J. Jiao, Y. K. Xing, Q. L. Yang, H. Qiu and T. S. Mei, *Acc. Chem. Res.*, 2020, **53**, 300-310; (e) L. Ackermann, *Acc. Chem. Res.*, 2020, **53**, 84-104; (f) L. F. T. Novaes, J. Liu, Y. Shen, L. Lu, J. M. Meinhardt and S. Lin, *Chem. Soc. Rev.*, 2021, **50**, 7941-8002; (g) Y. Yuan, J. Yang and A. Lei, *Chem. Soc. Rev.*, 2021, **50**, 10058-10086; (h) P. Xiong and H.-C. Xu, *Acc. Chem. Res.*, 2019, **52**, 3339-3350.

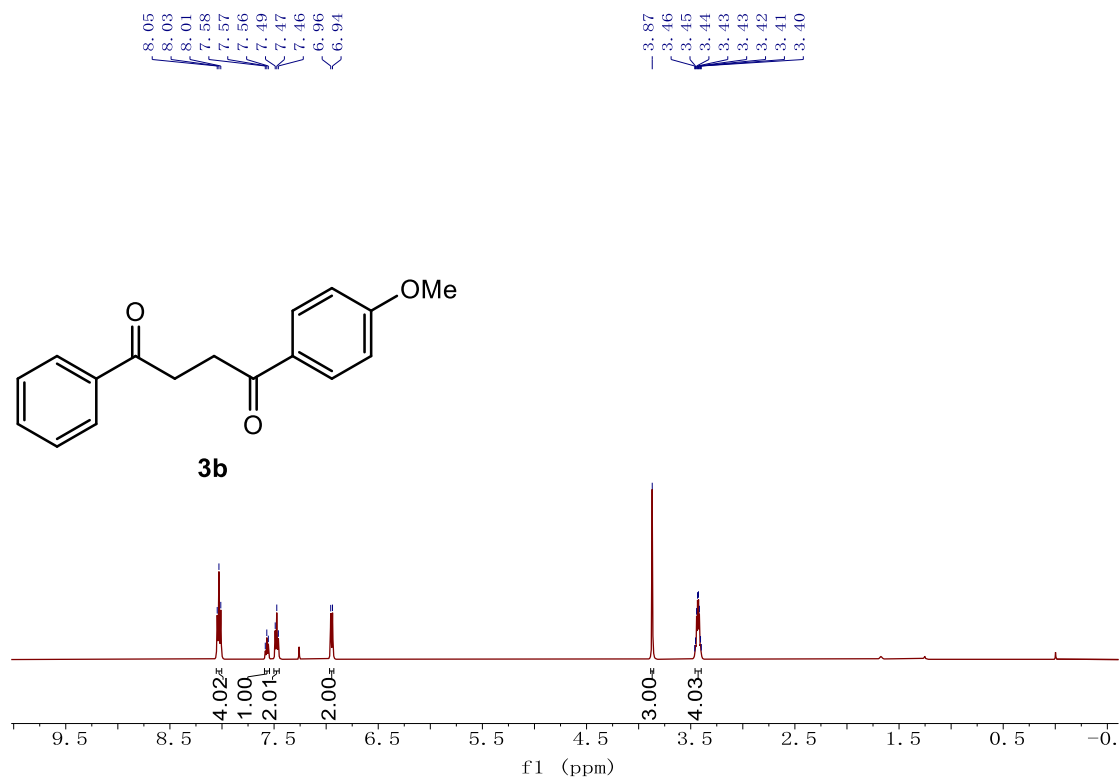
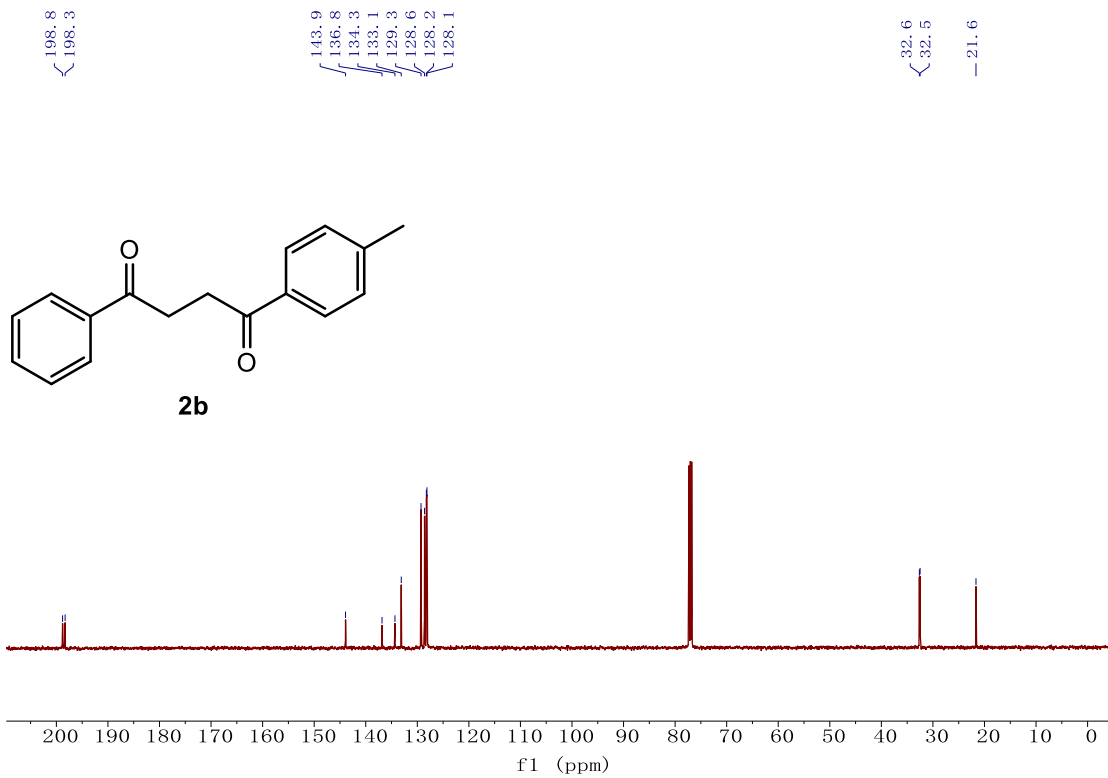
Other citations of ref. 9 in the manuscript: (e) D. F. Yang, Z. P. Guan, Y. N. Peng, S. X. Zhu, P. J. Wang, Z. L. Huang, H. Alhumade, D. Gu, H. Yi and A. Lei, *Nat. Commun.*, 2023, **14**, 1476-1484;

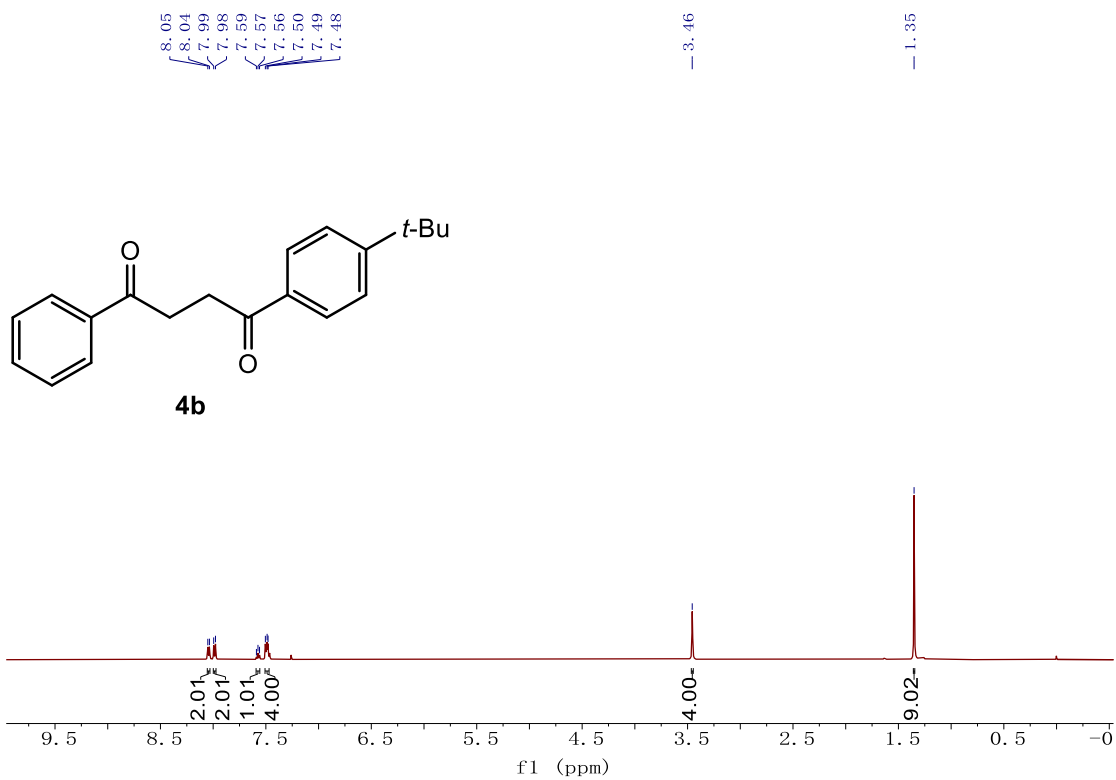
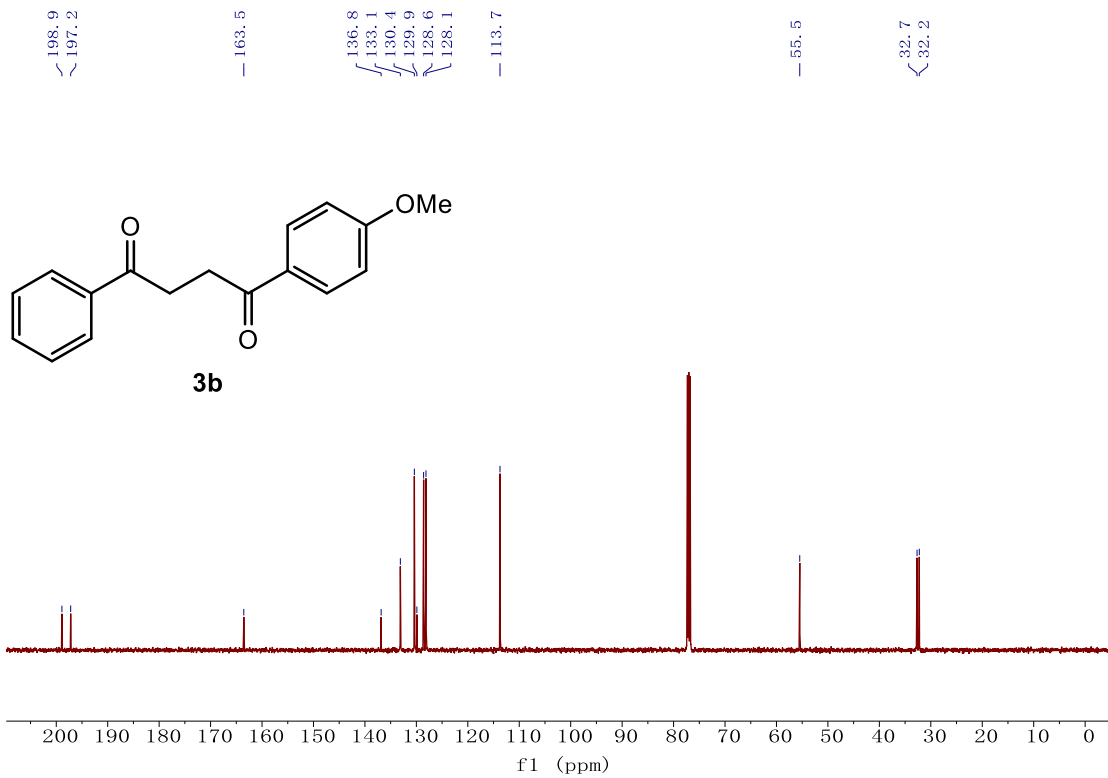
(f) L.-H. Jie, B. Guo, J. Song, and H.-C. Xu, *J. Am. Chem. Soc.*, 2022, **144**, 2343-2350; (g) Y. Gao, B. Zhang, J. He, and P. S. Baran, *J. Am. Chem. Soc.*, 2023, **145**, 11518-11523; (h) W. Zhang and S. Lin, *J. Am. Chem. Soc.*, 2020, **142**, 20661-20670.

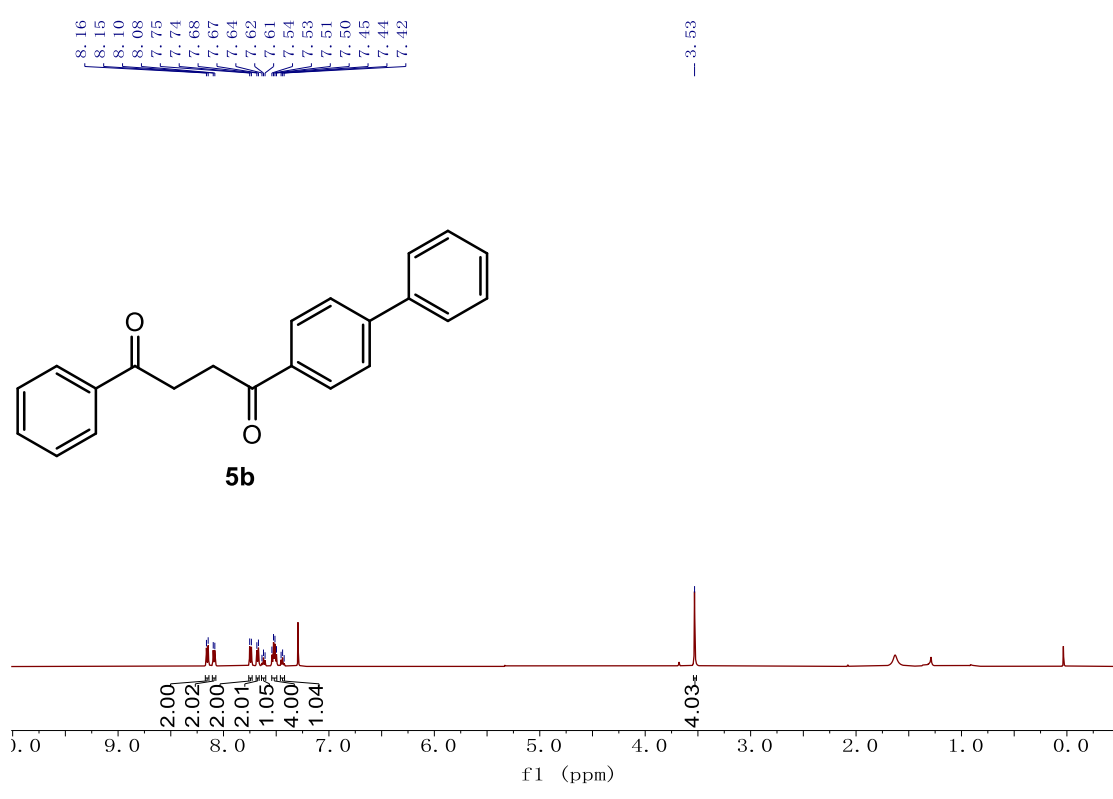
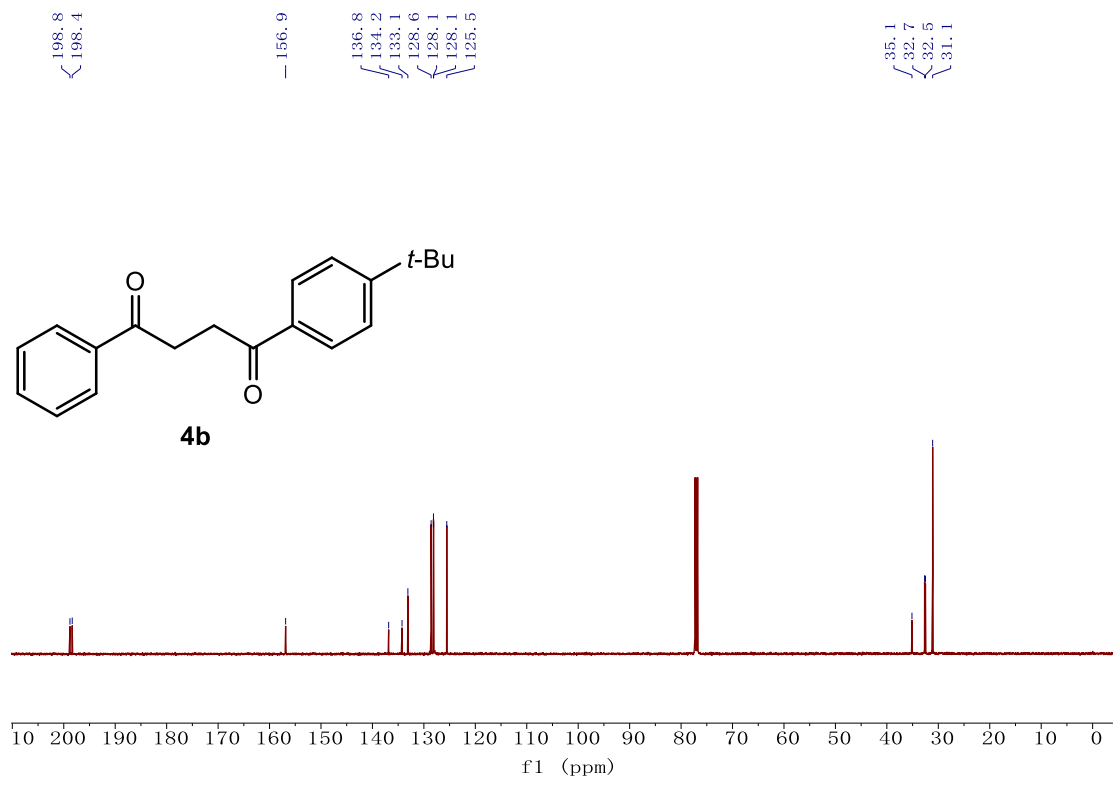
14. NMR spectra

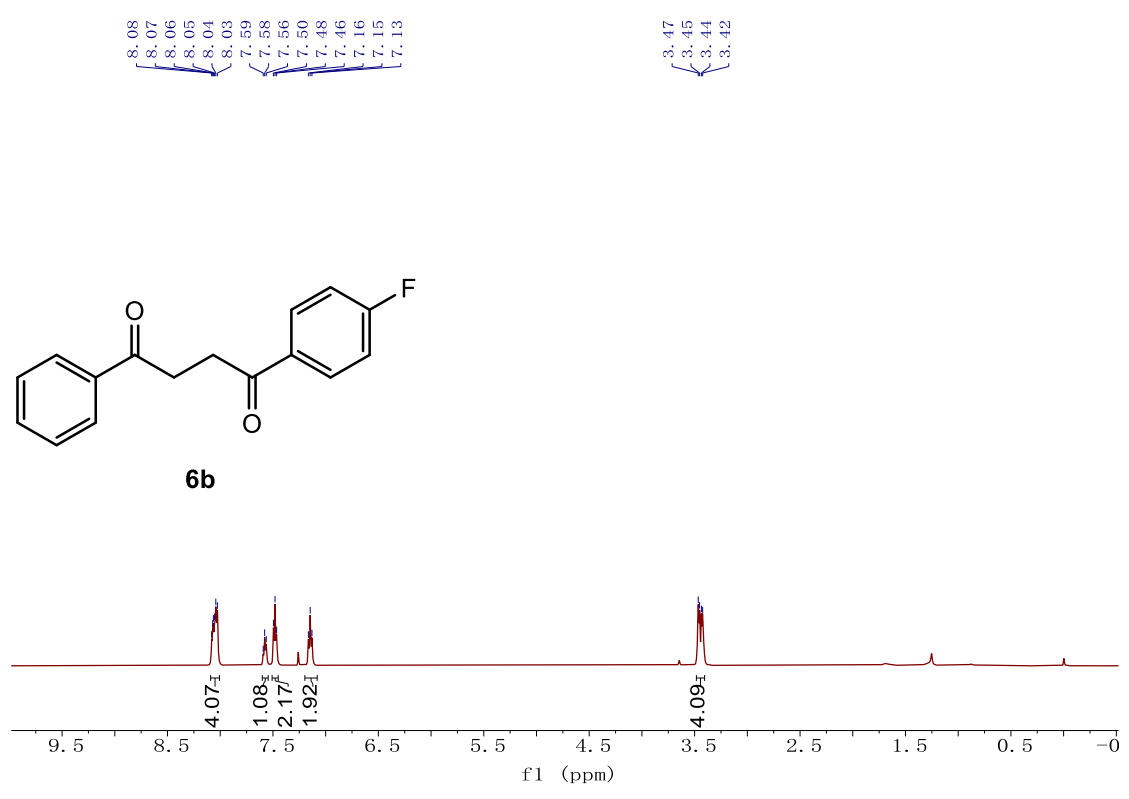
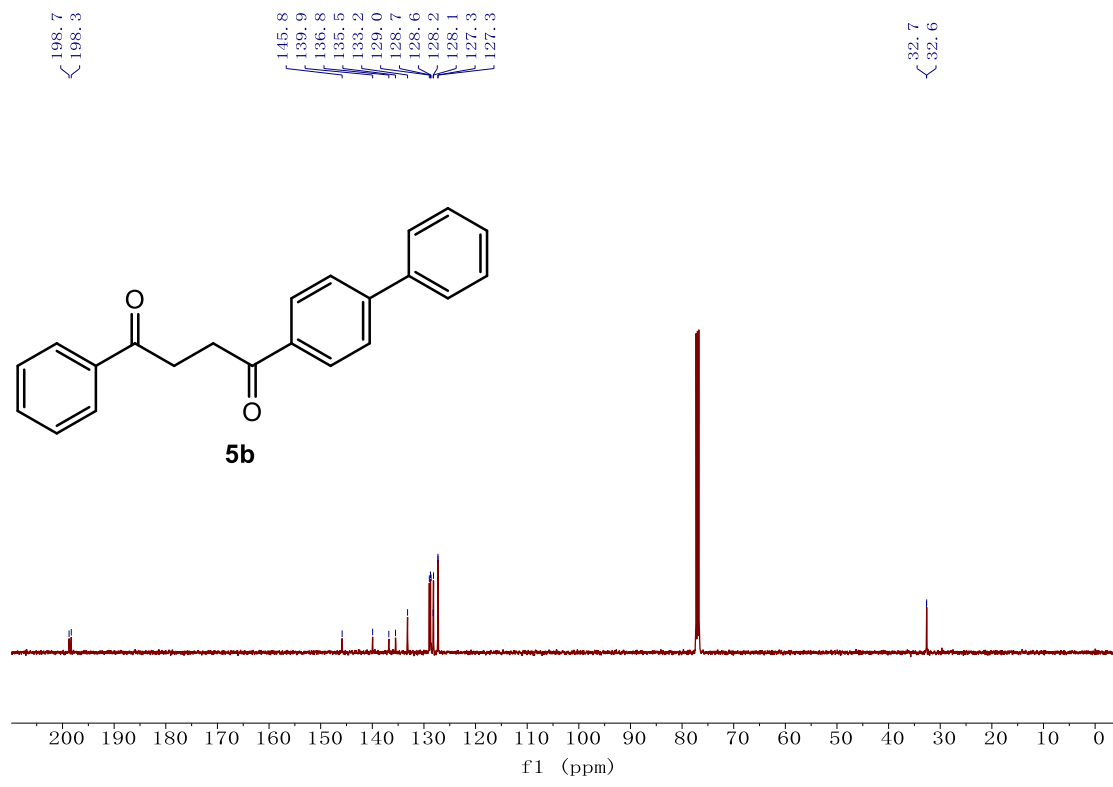


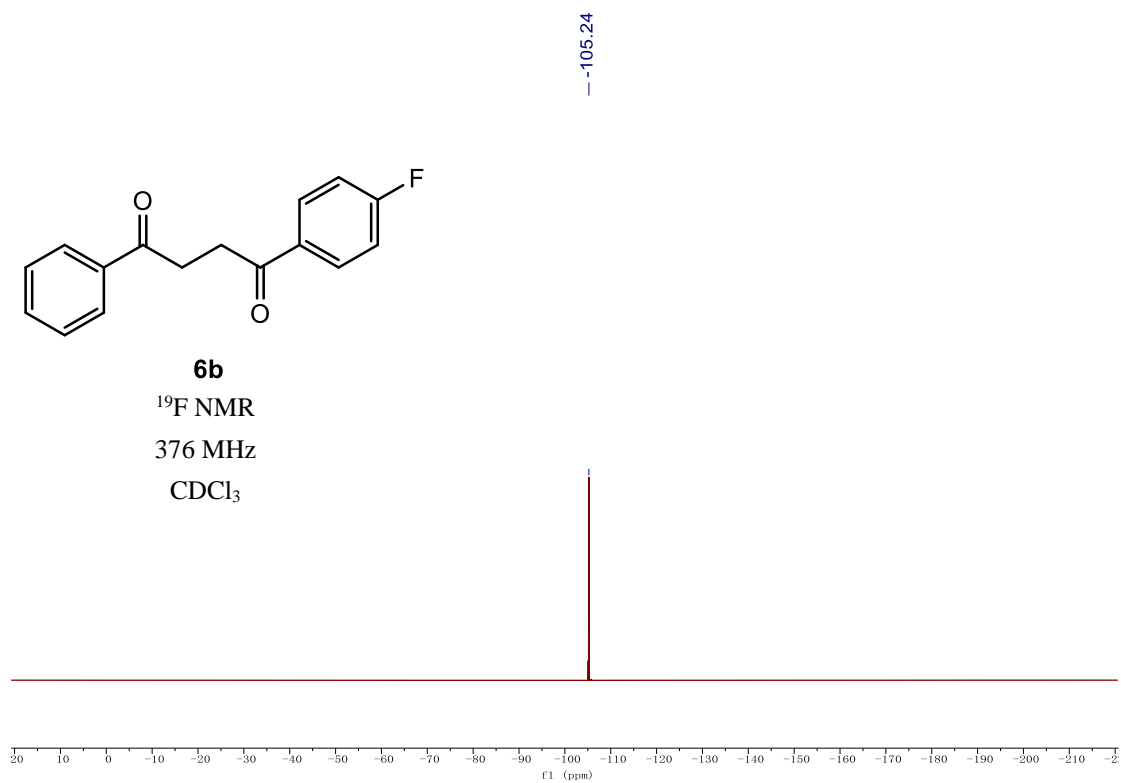
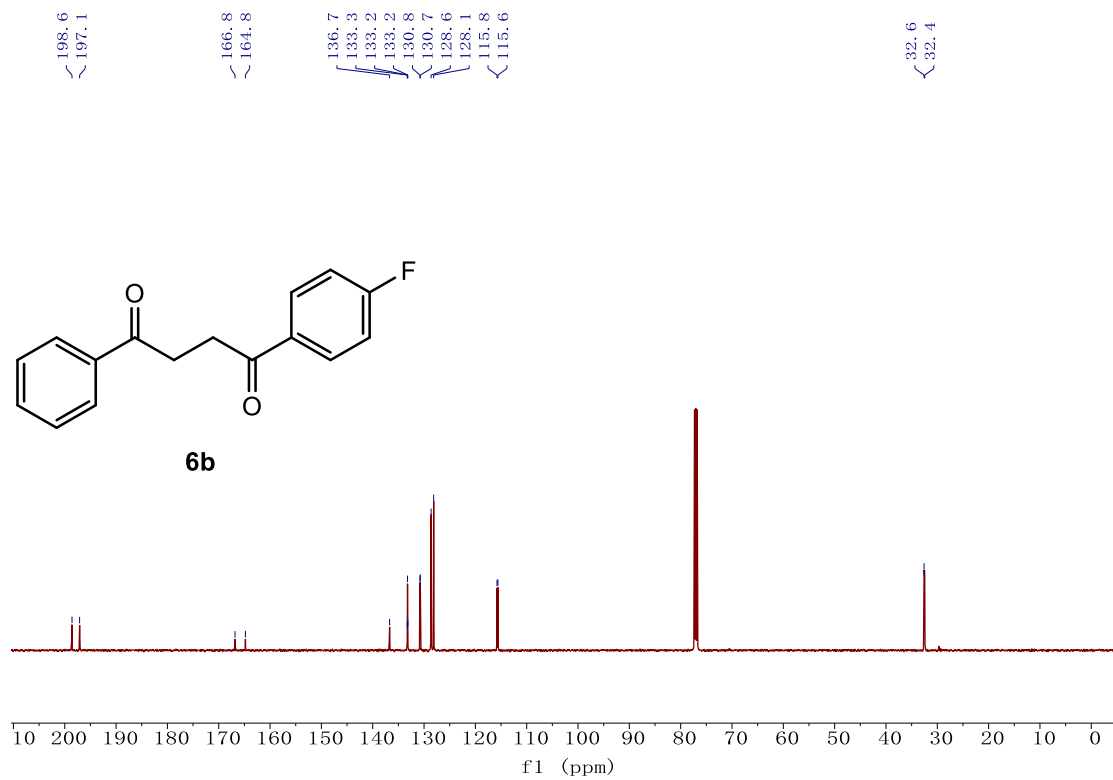


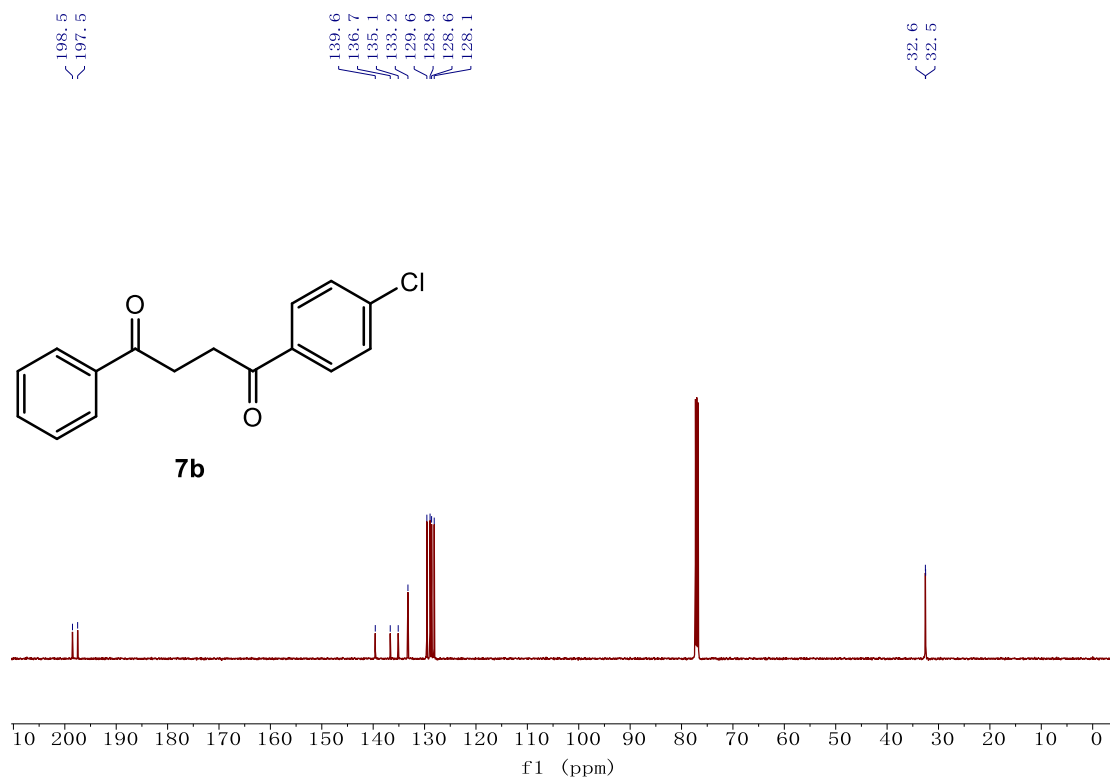
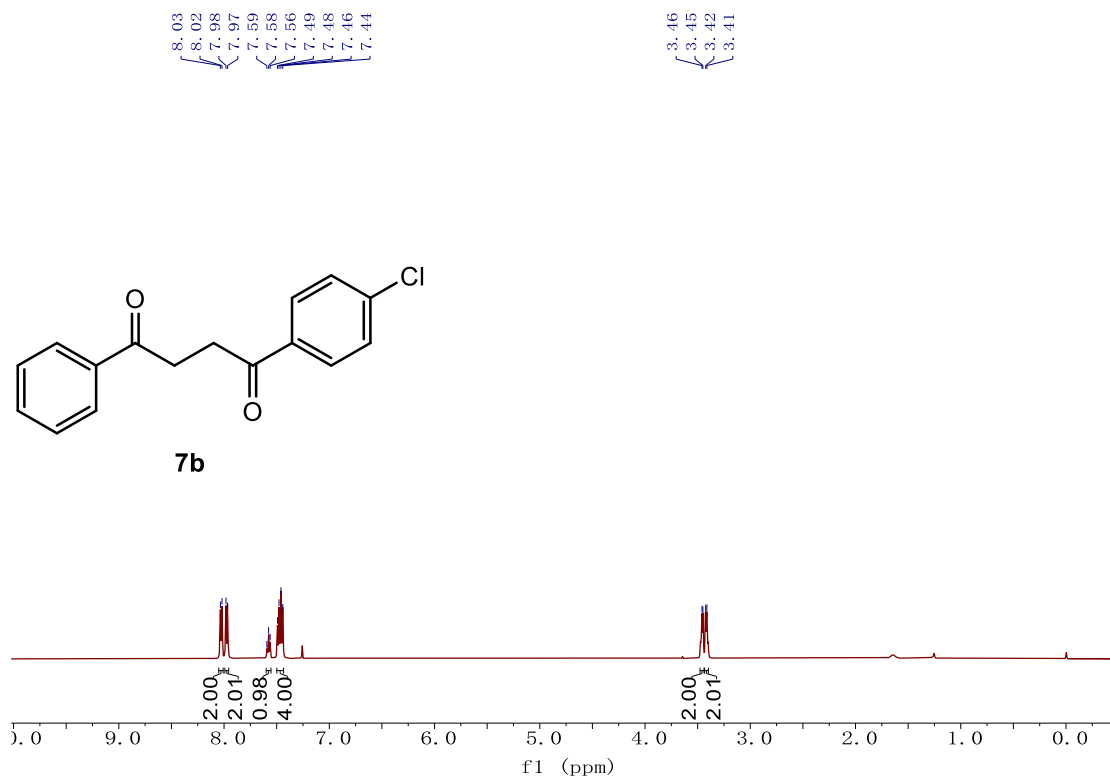


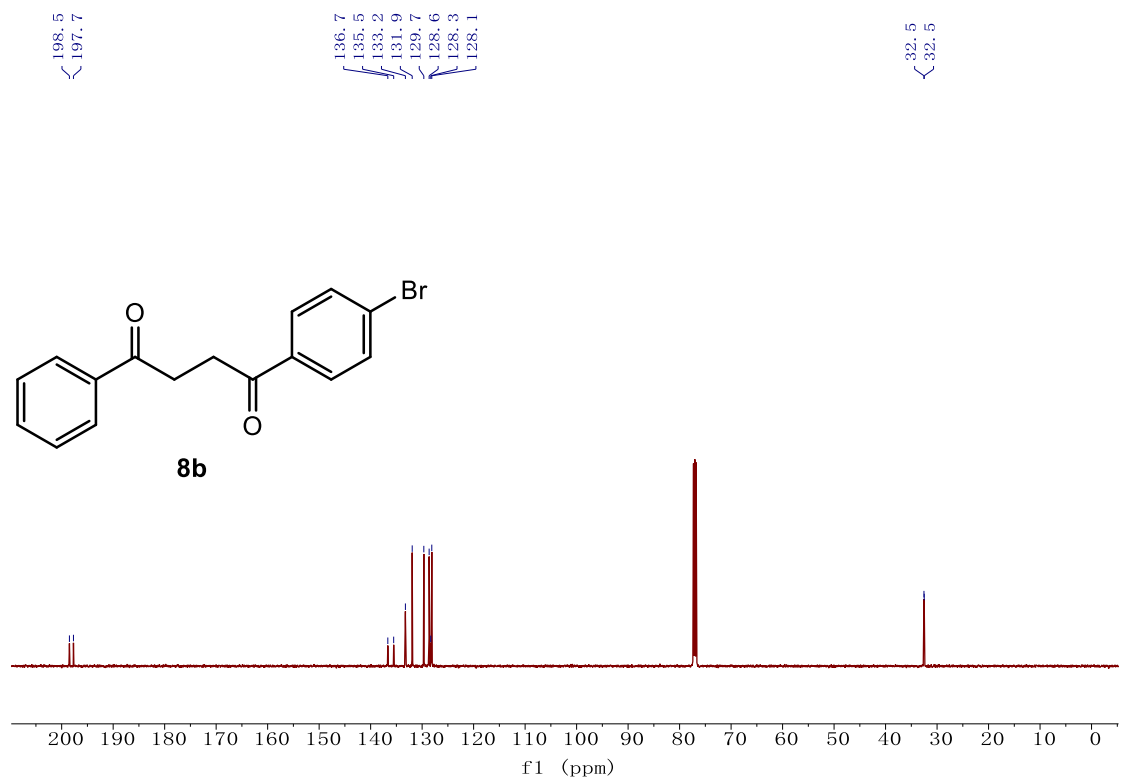
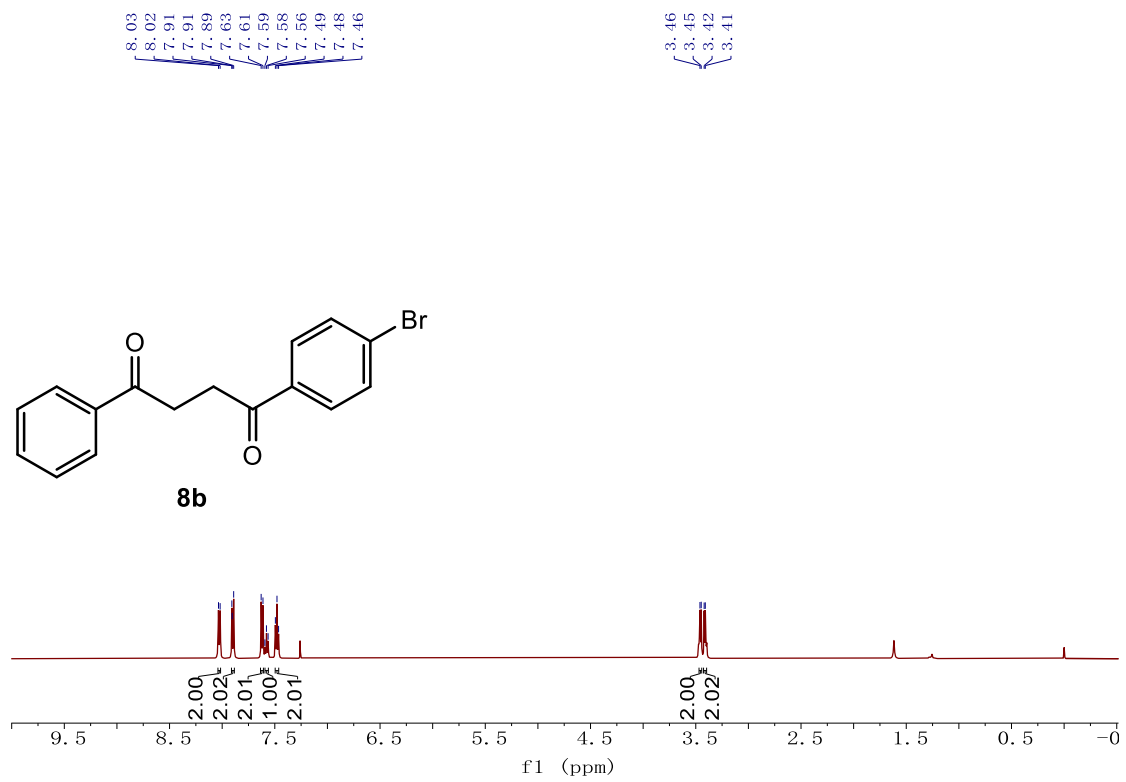


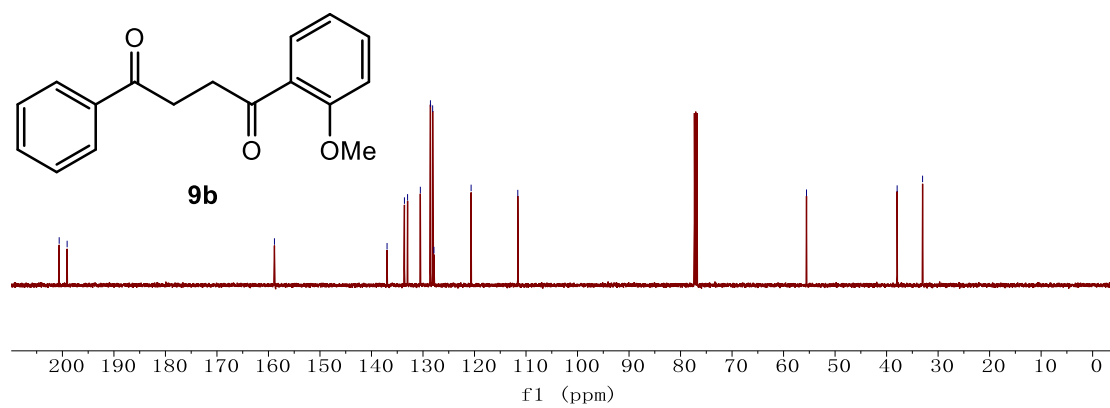
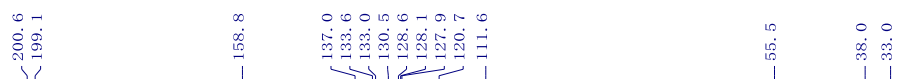
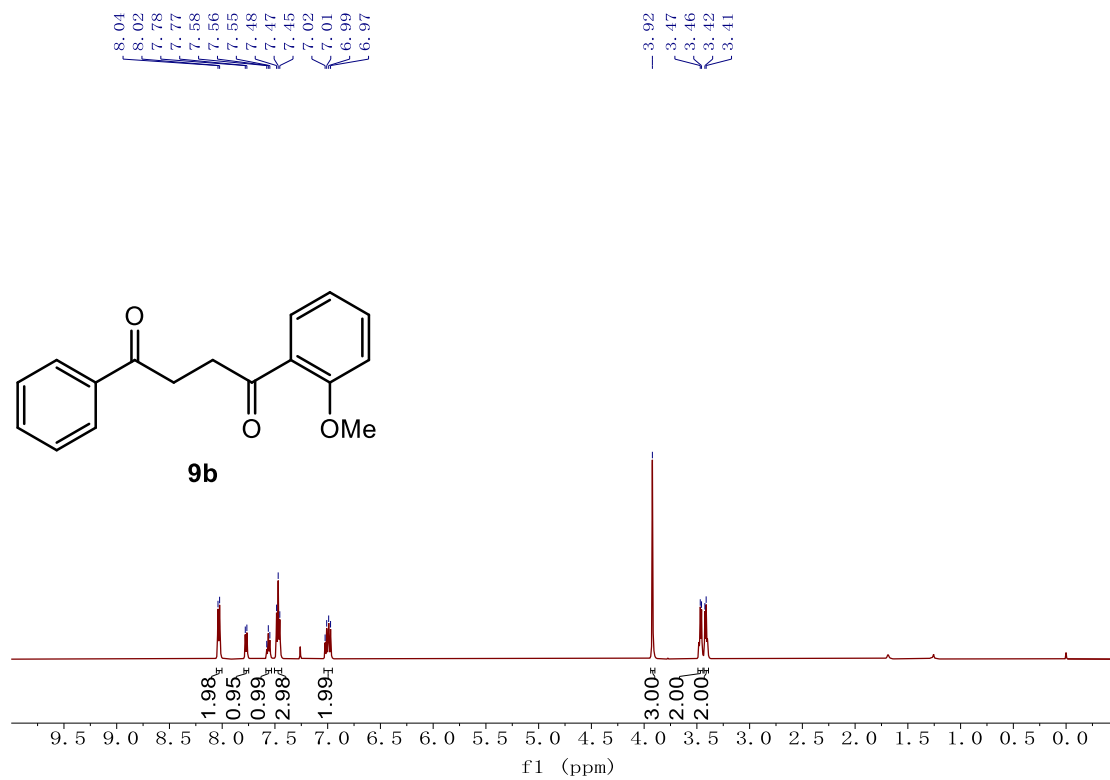




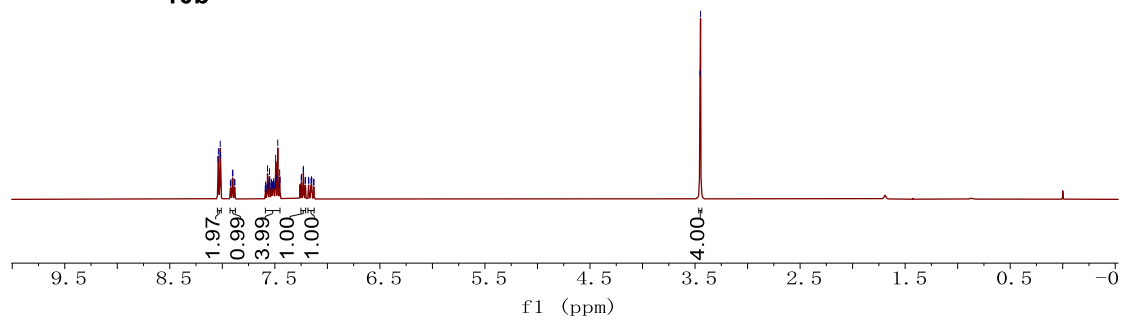
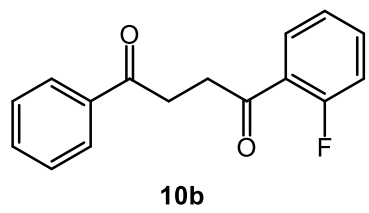




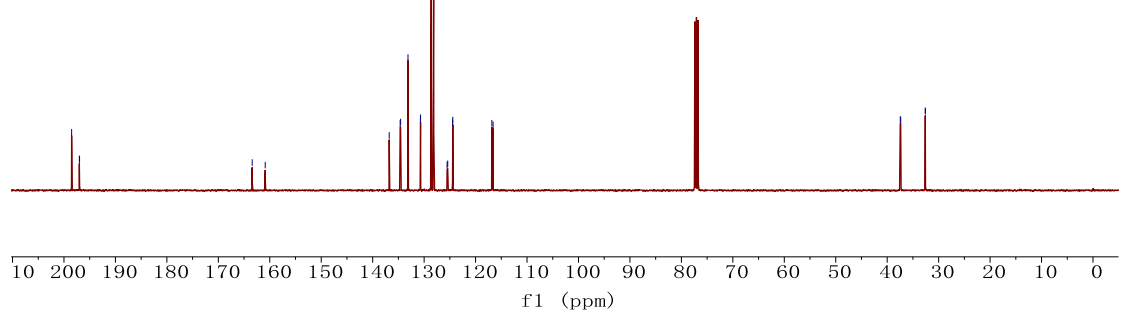
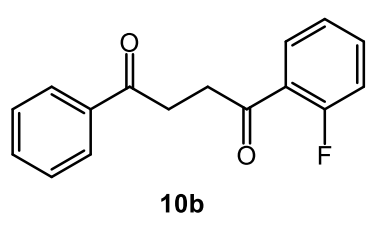


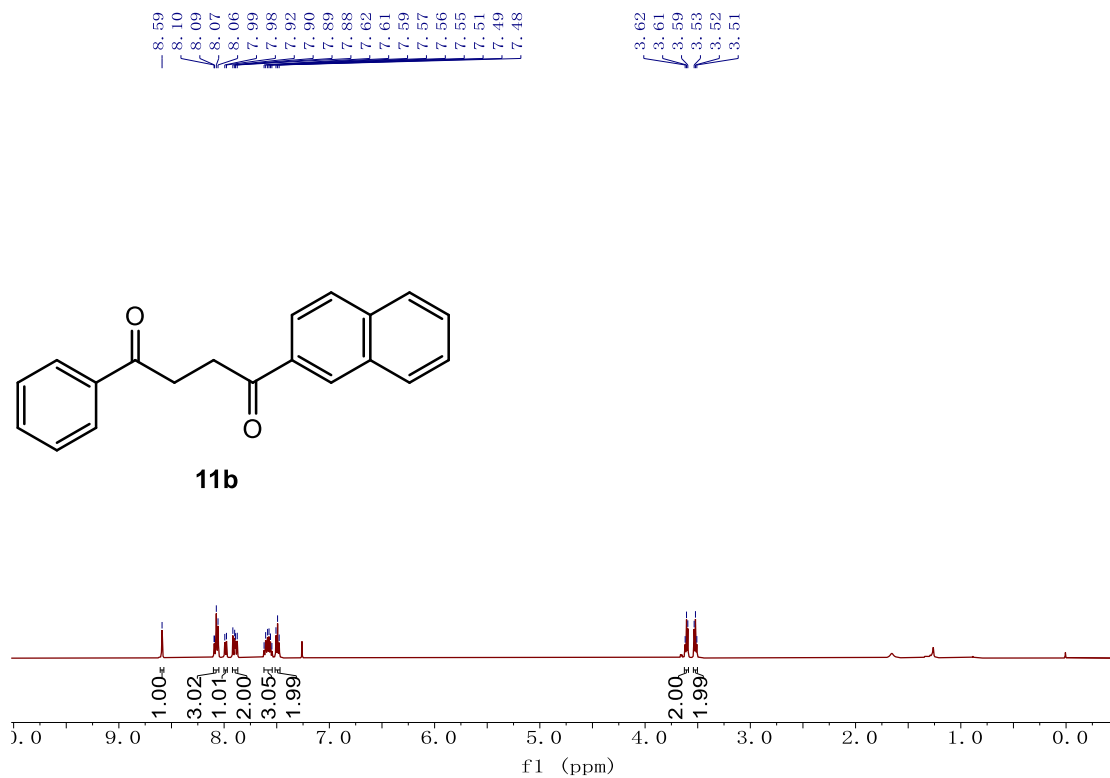
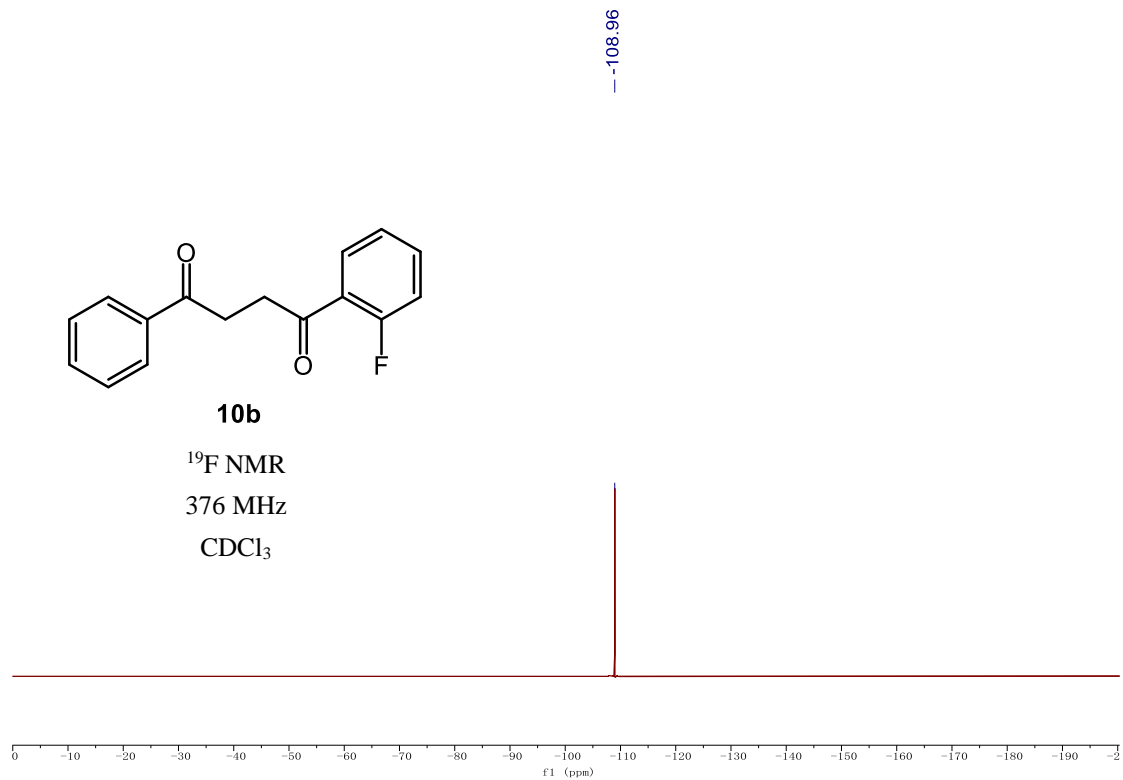


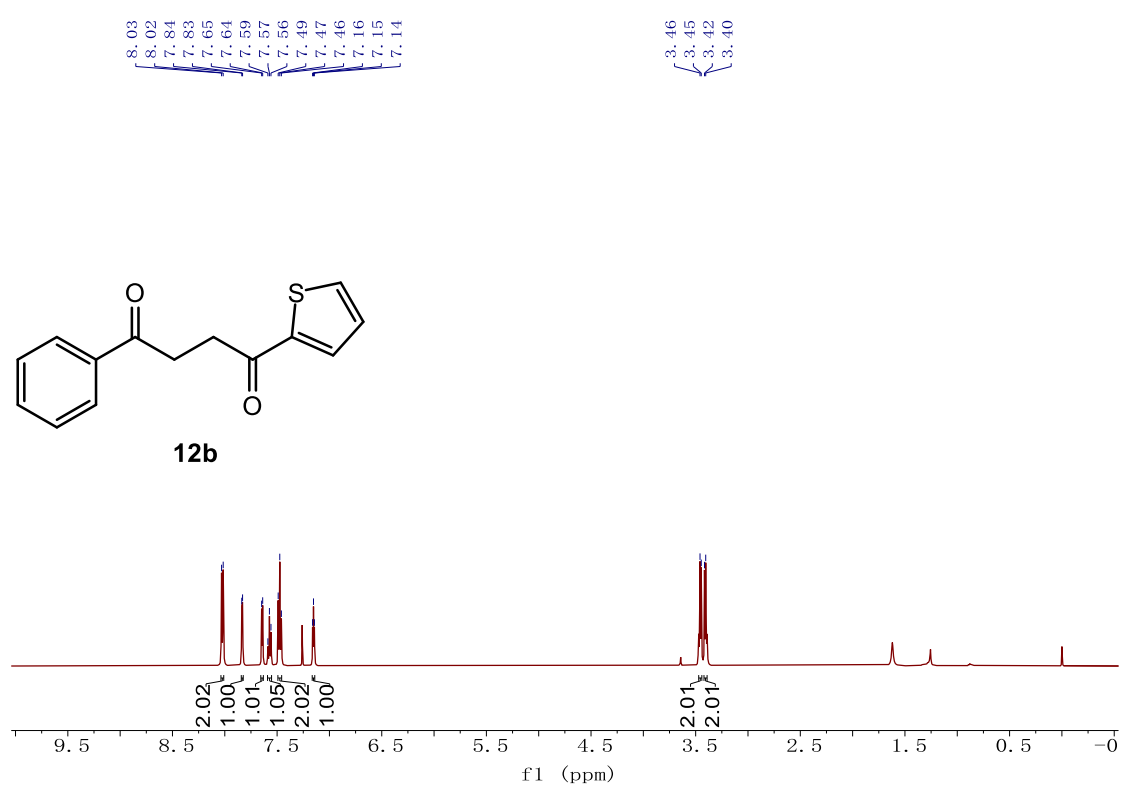
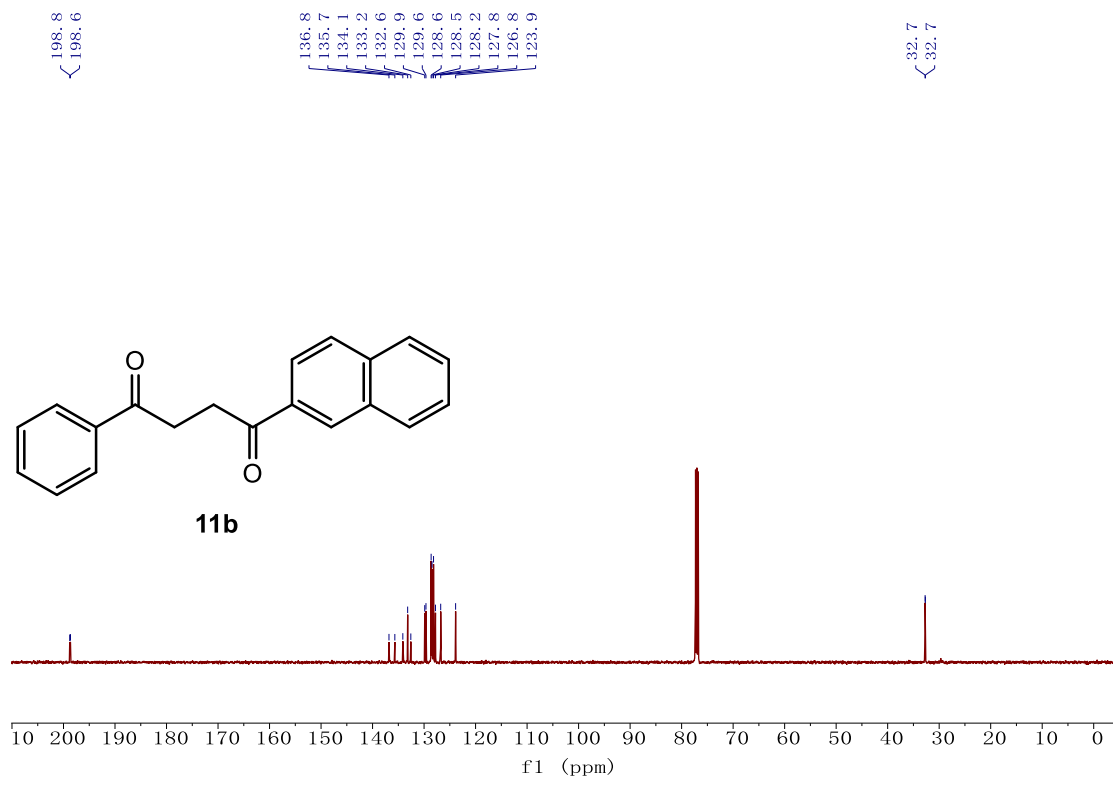
8.04
8.04
8.02
8.02
7.92
7.90
7.90
7.88
7.88
7.59
7.59
7.58
7.58
7.57
7.56
7.55
7.55
7.55
7.54
7.53
7.53
7.53
7.52
7.52
7.51
7.51
7.51
7.50
7.49
7.49
7.47
7.46
7.45
7.45
7.25
7.25
7.23
7.23
7.21
7.21
7.18
7.18
7.16
7.15
7.15
7.13
7.13
3.45
3.45

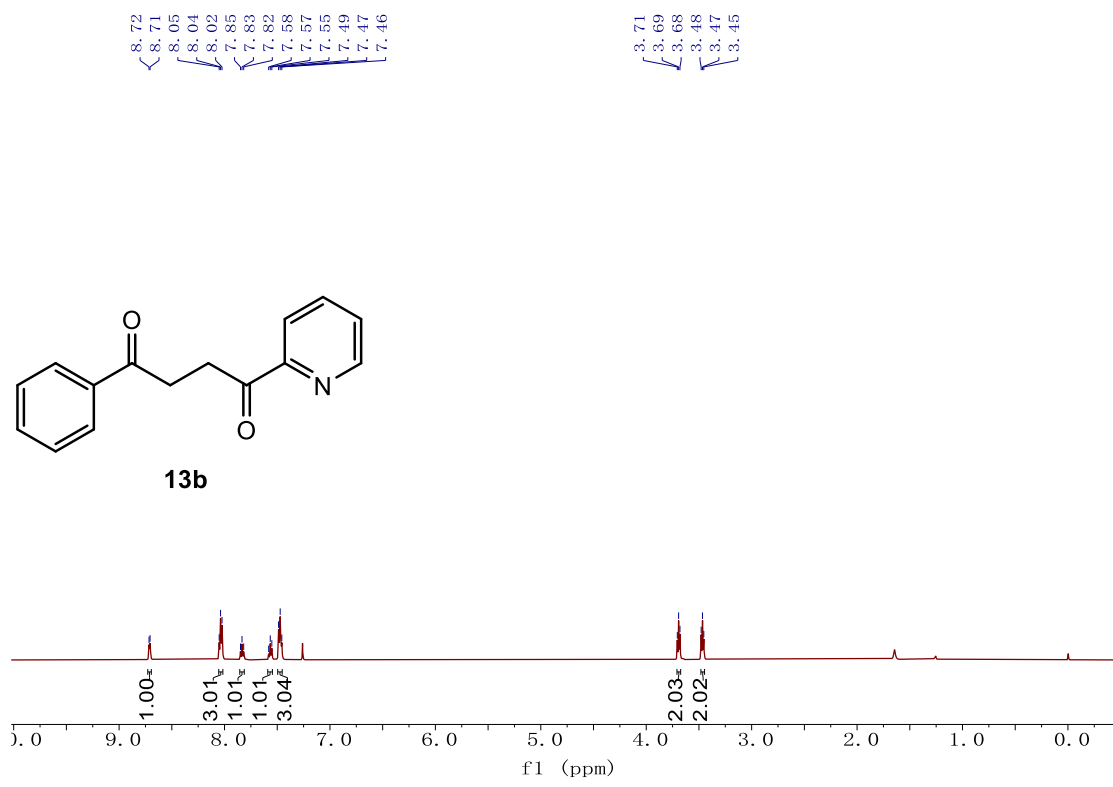
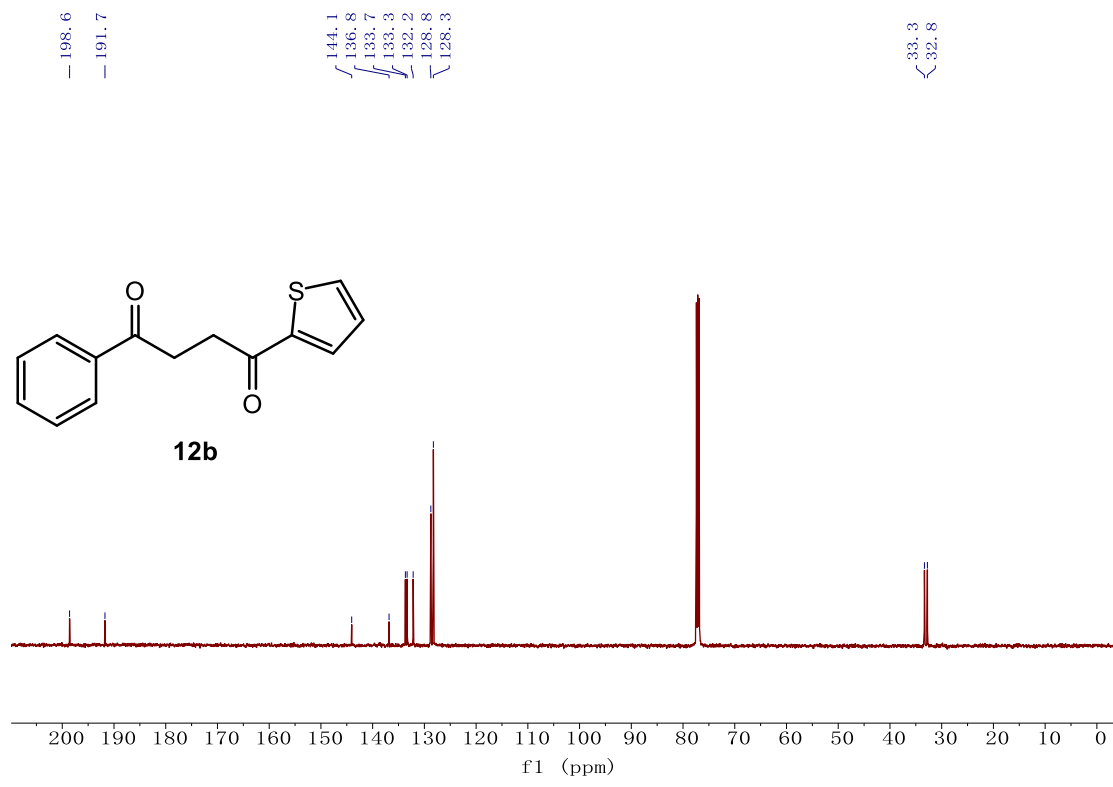


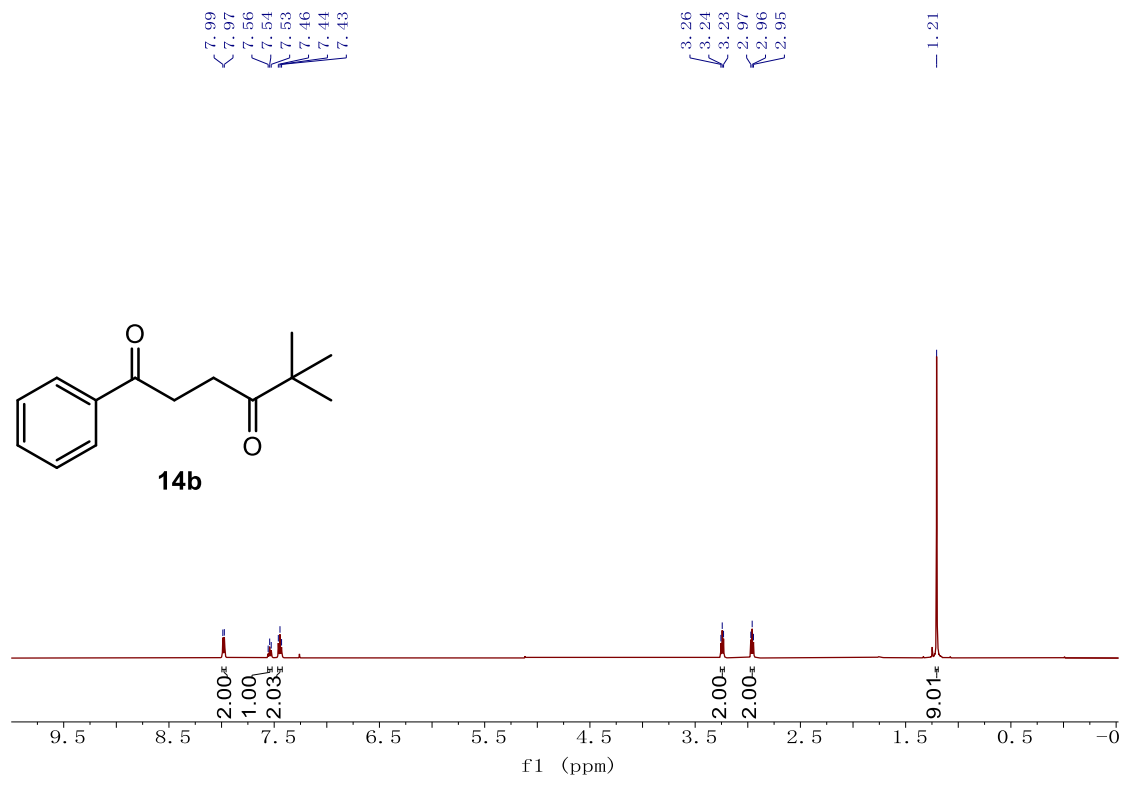
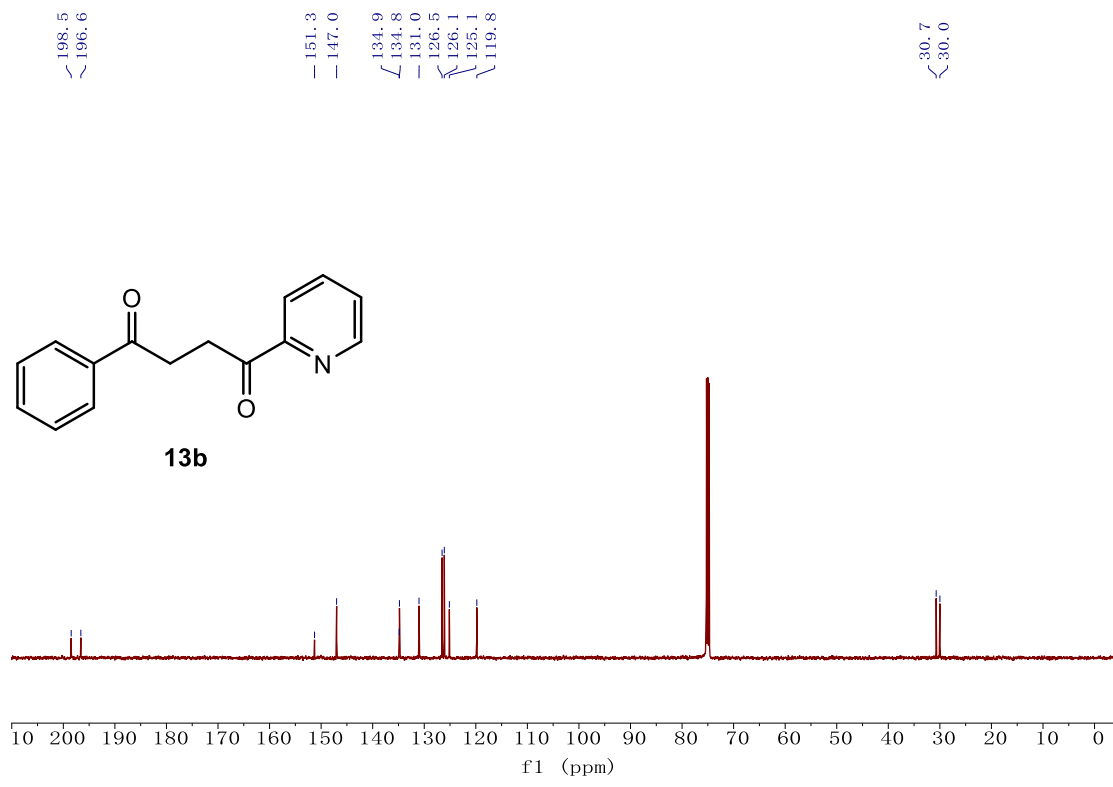
198.5
197.0
197.0
163.4
160.9
136.8
134.7
134.6
133.1
130.7
130.7
128.6
128.1
125.5
125.4
124.5
124.4
116.8
116.6
37.5
37.4
32.6
32.6

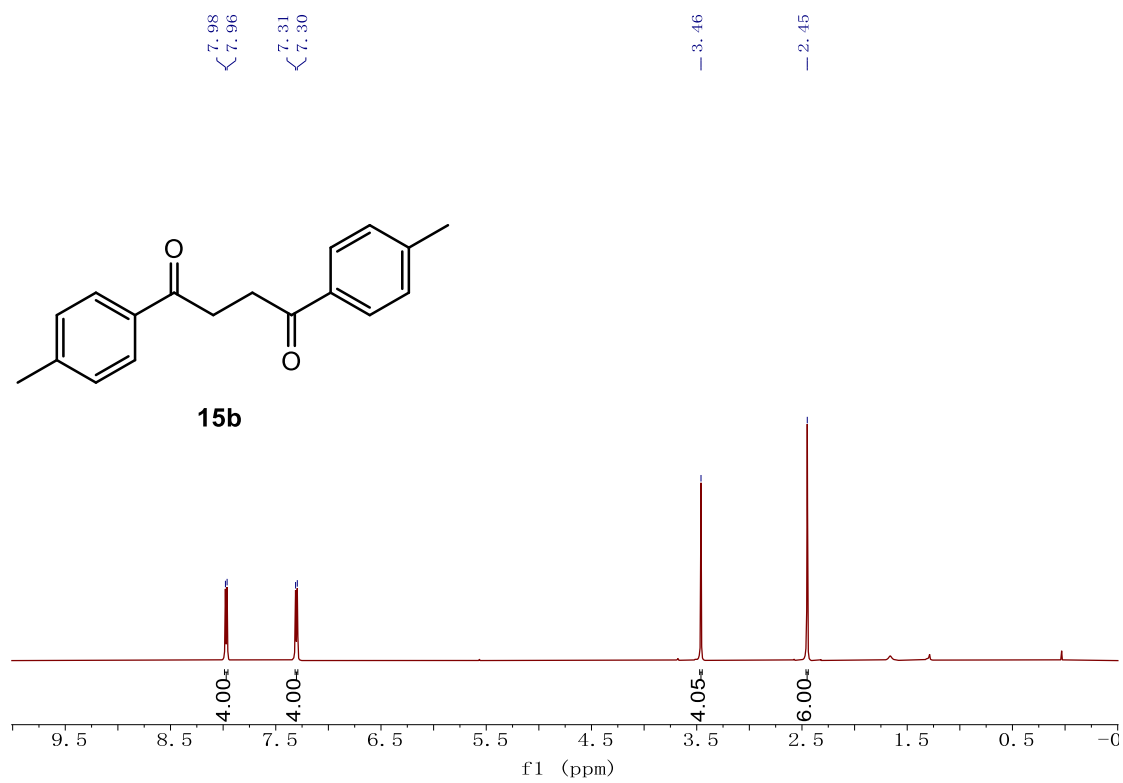
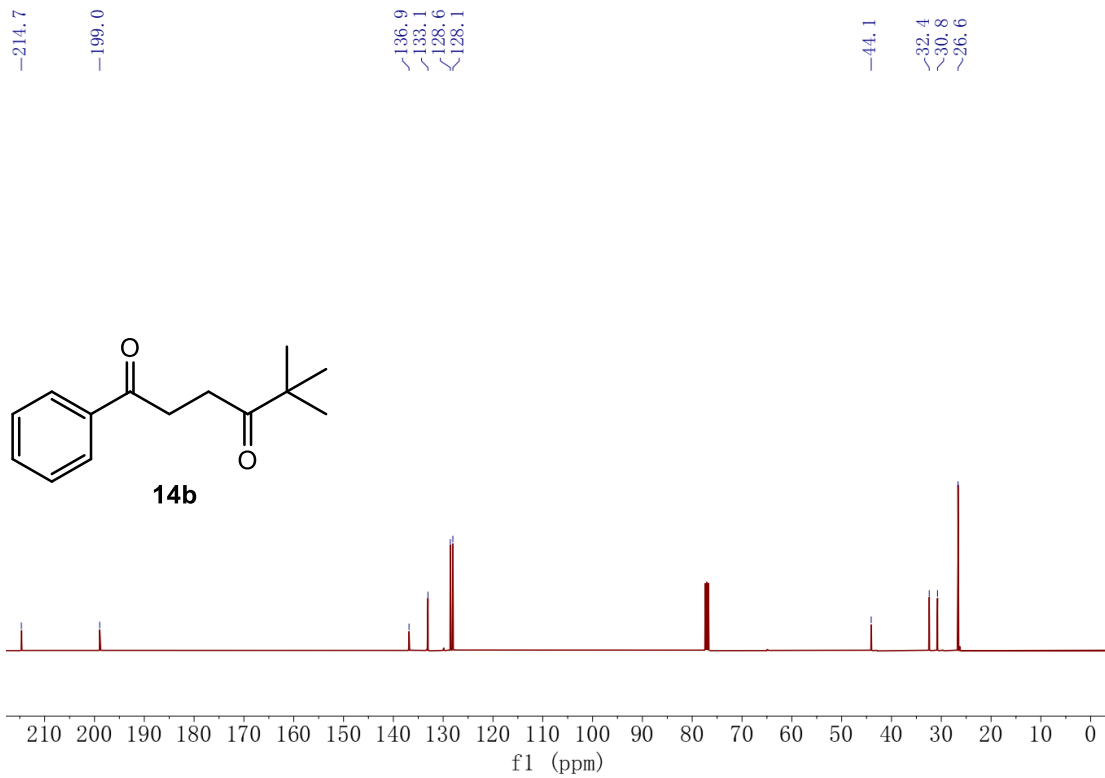


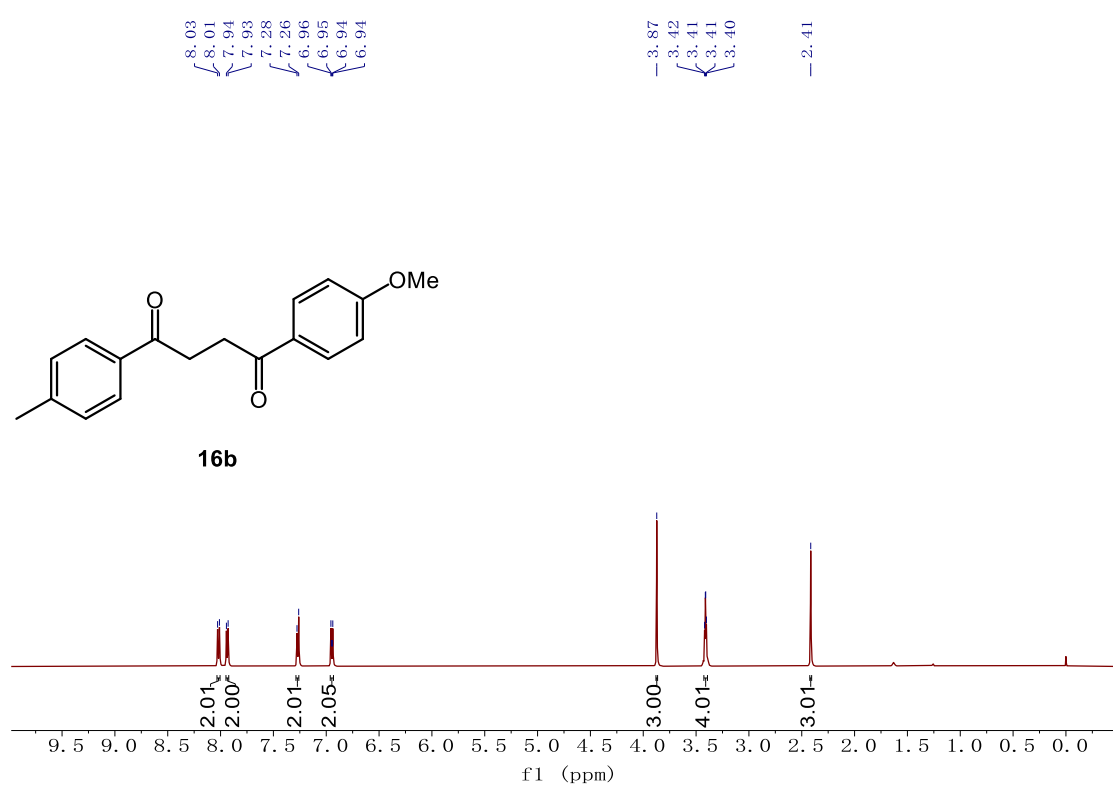
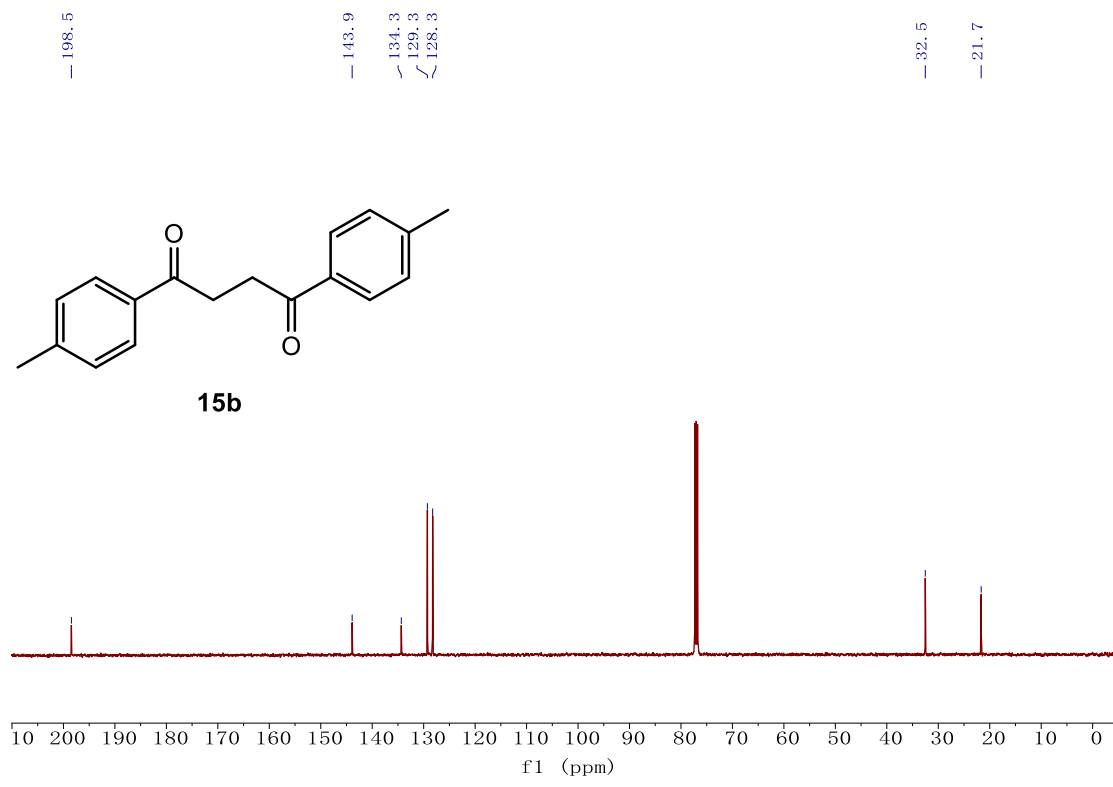


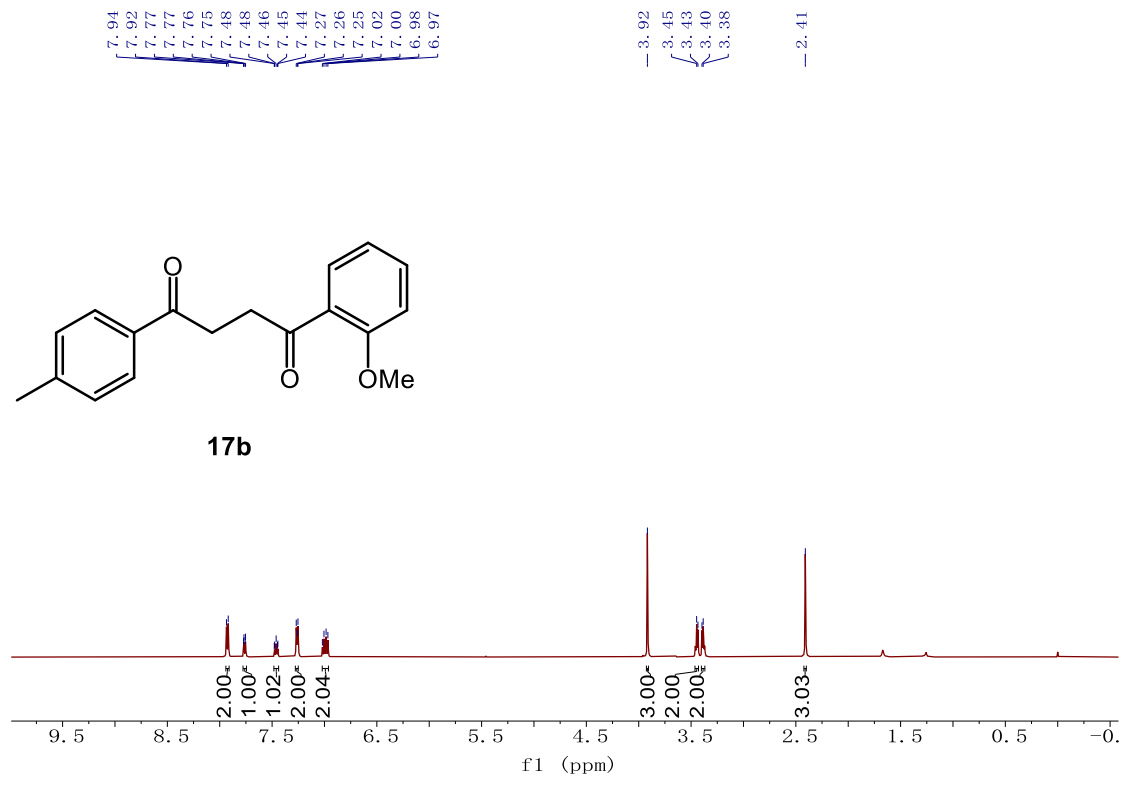
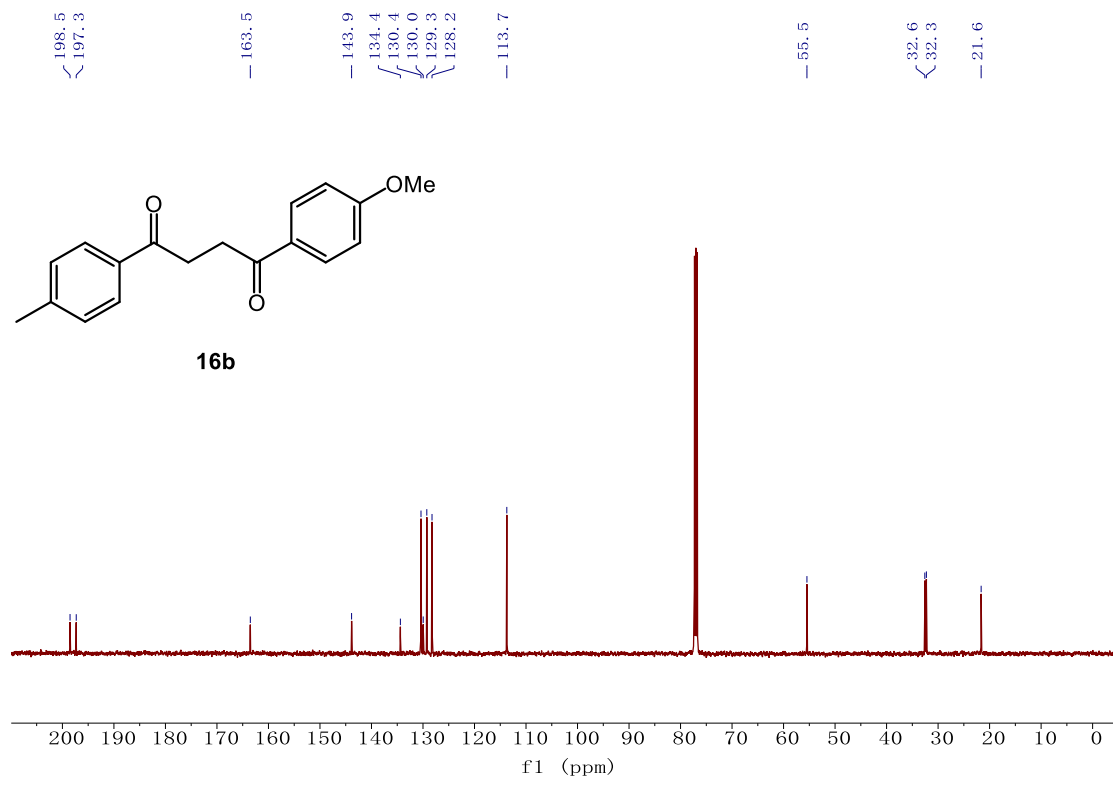


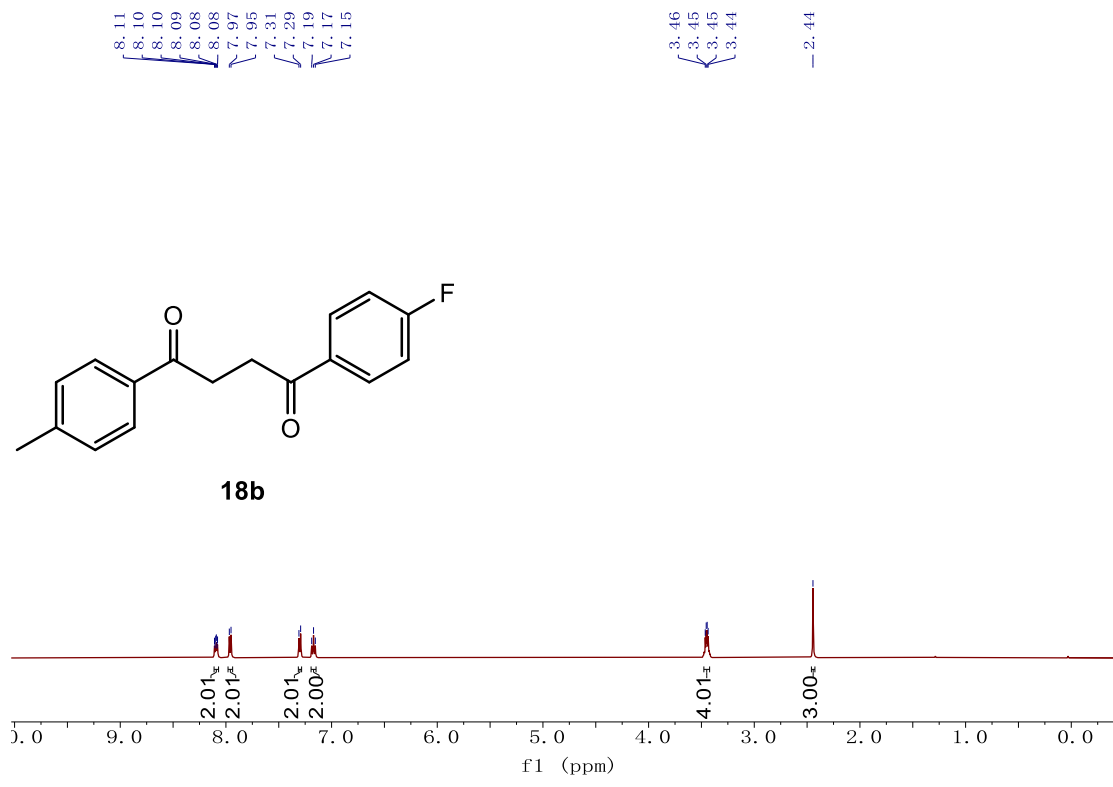
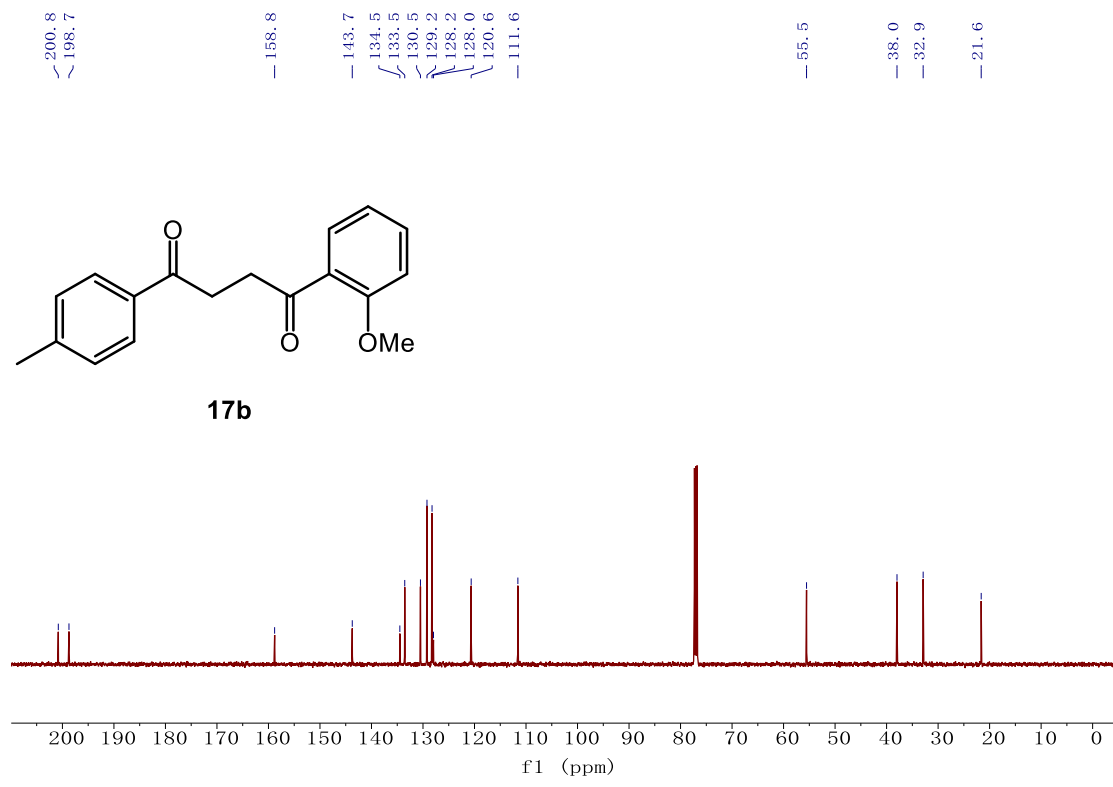


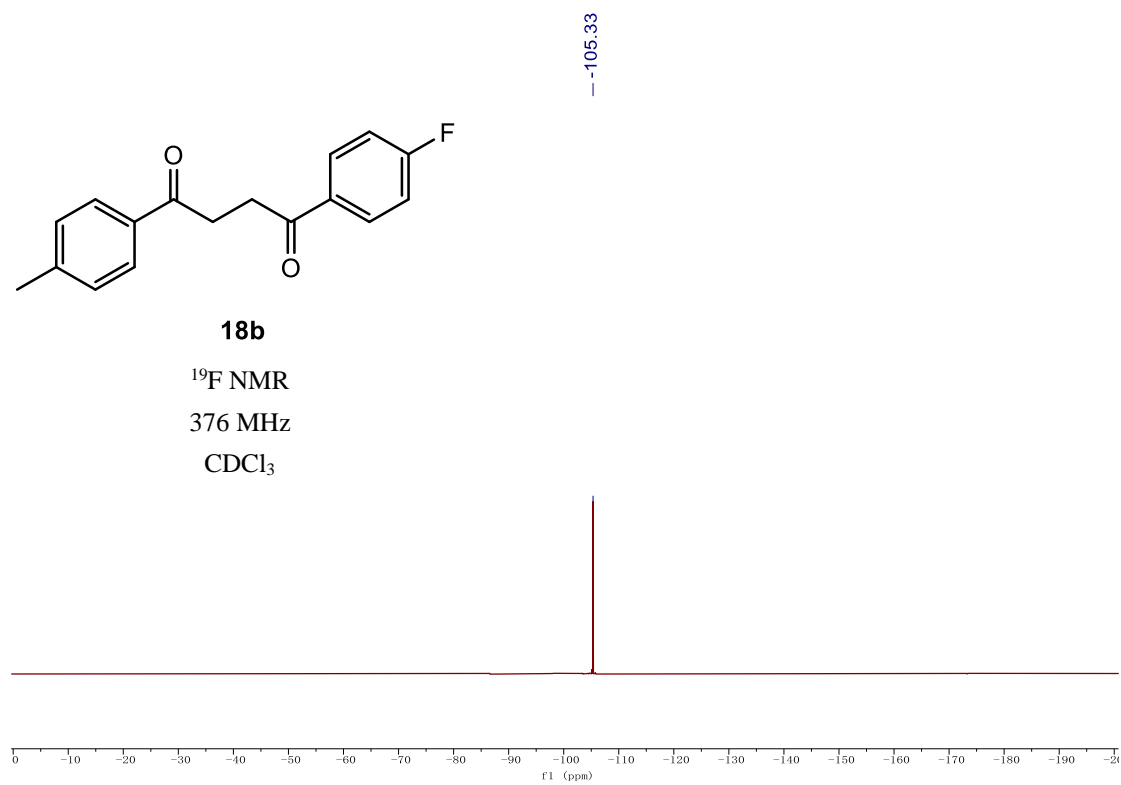
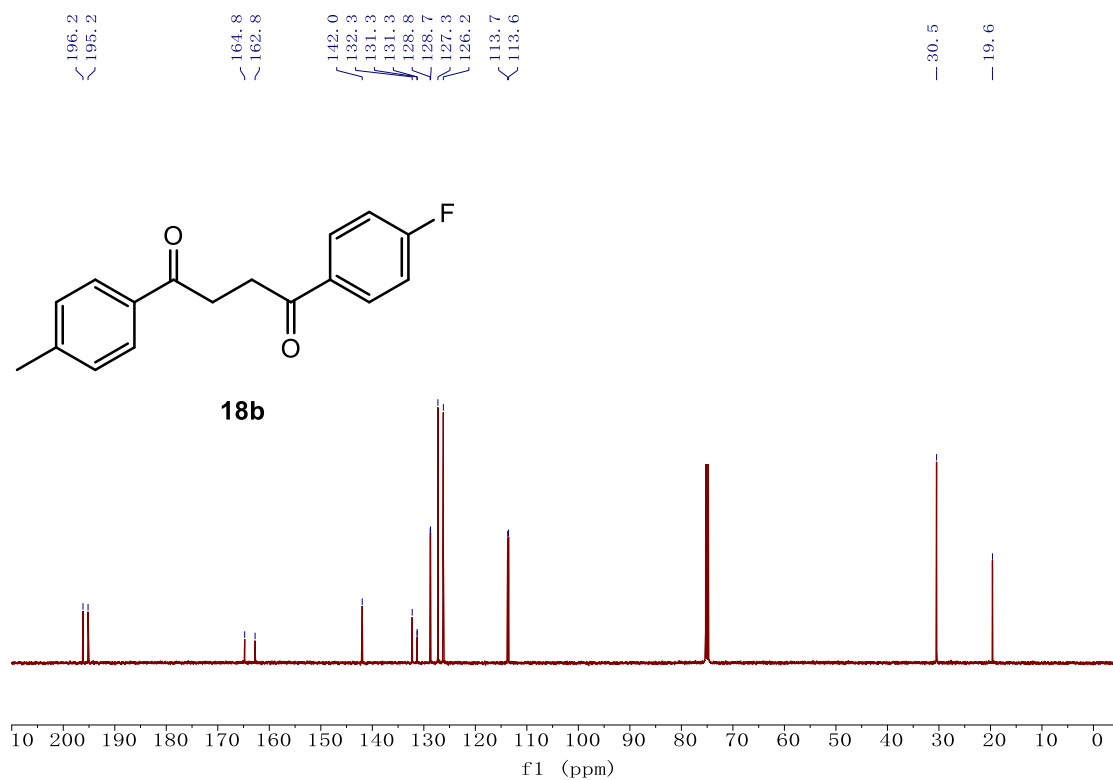


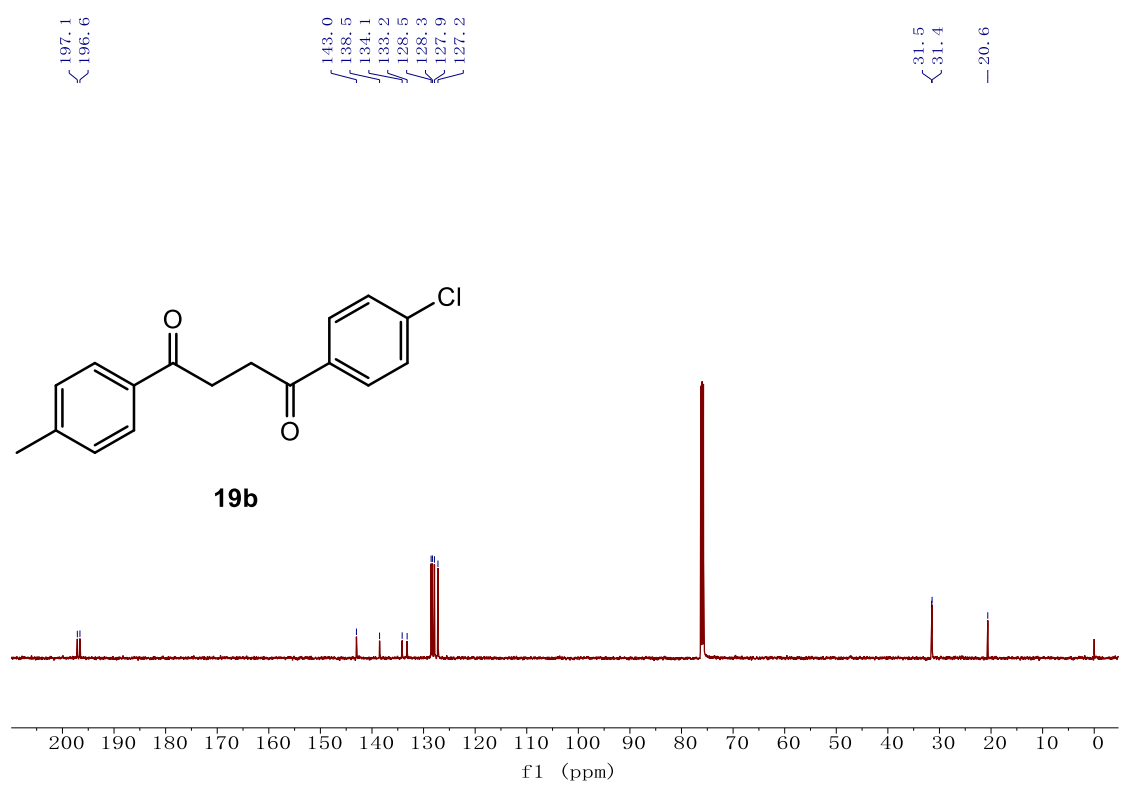
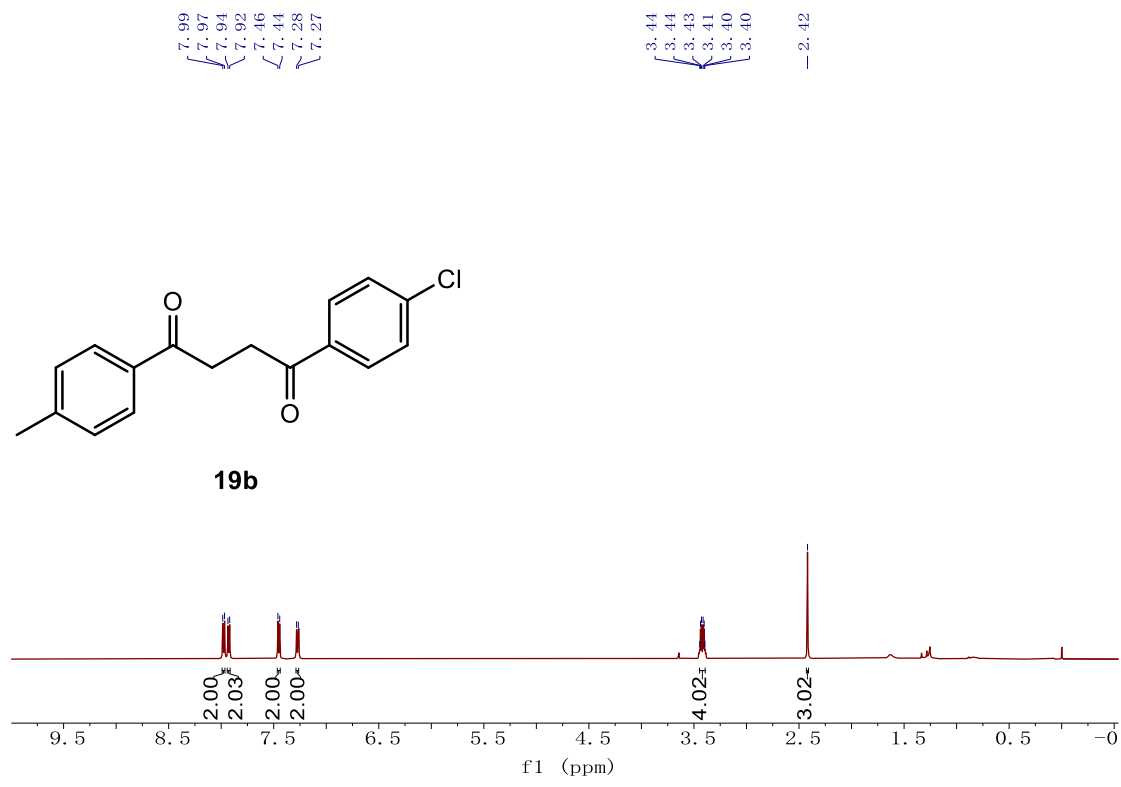


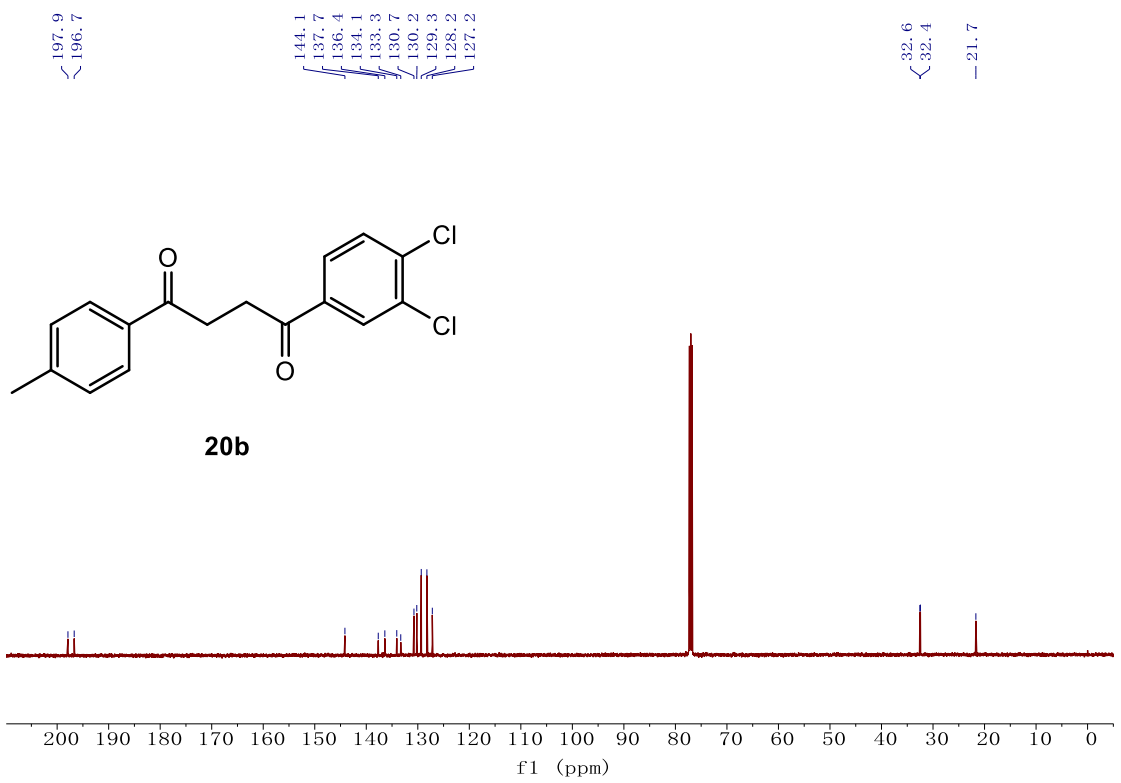
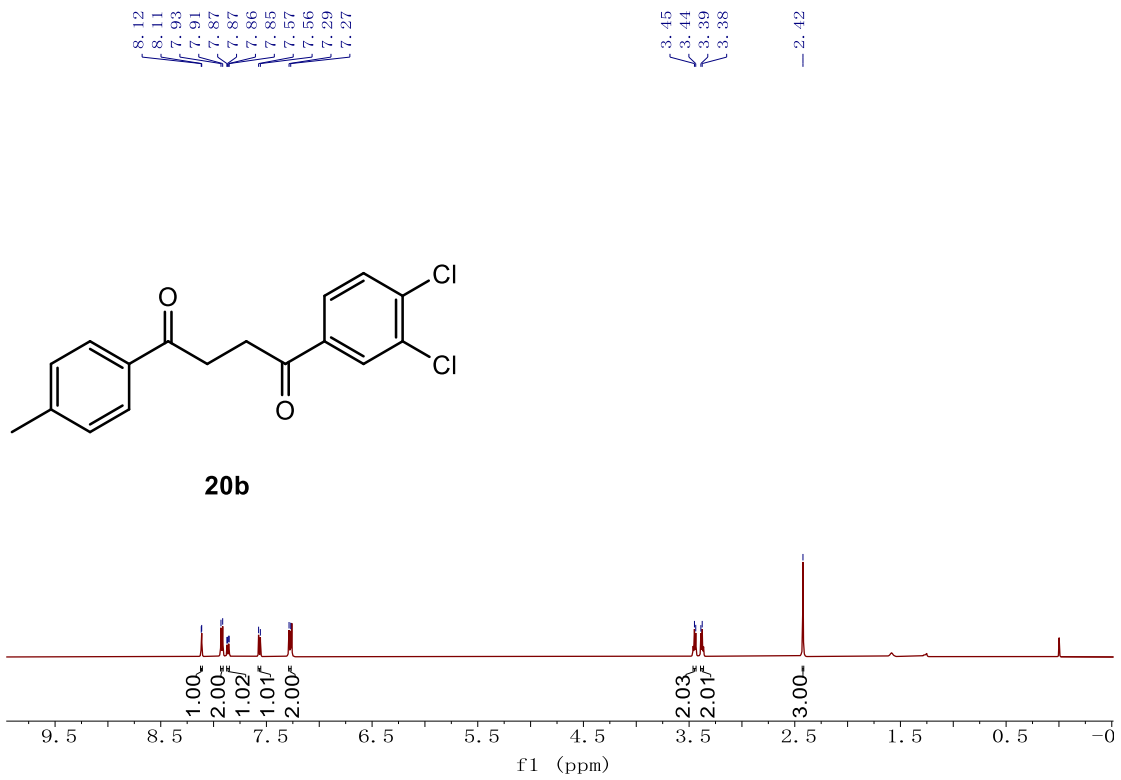


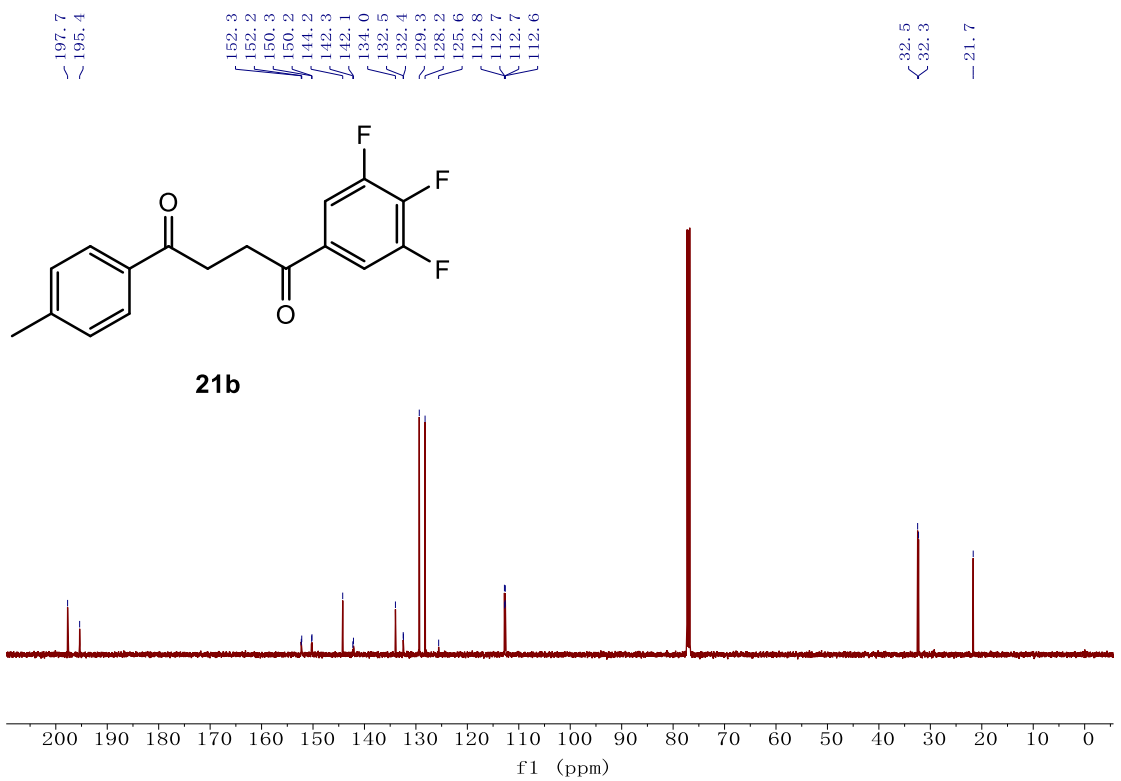
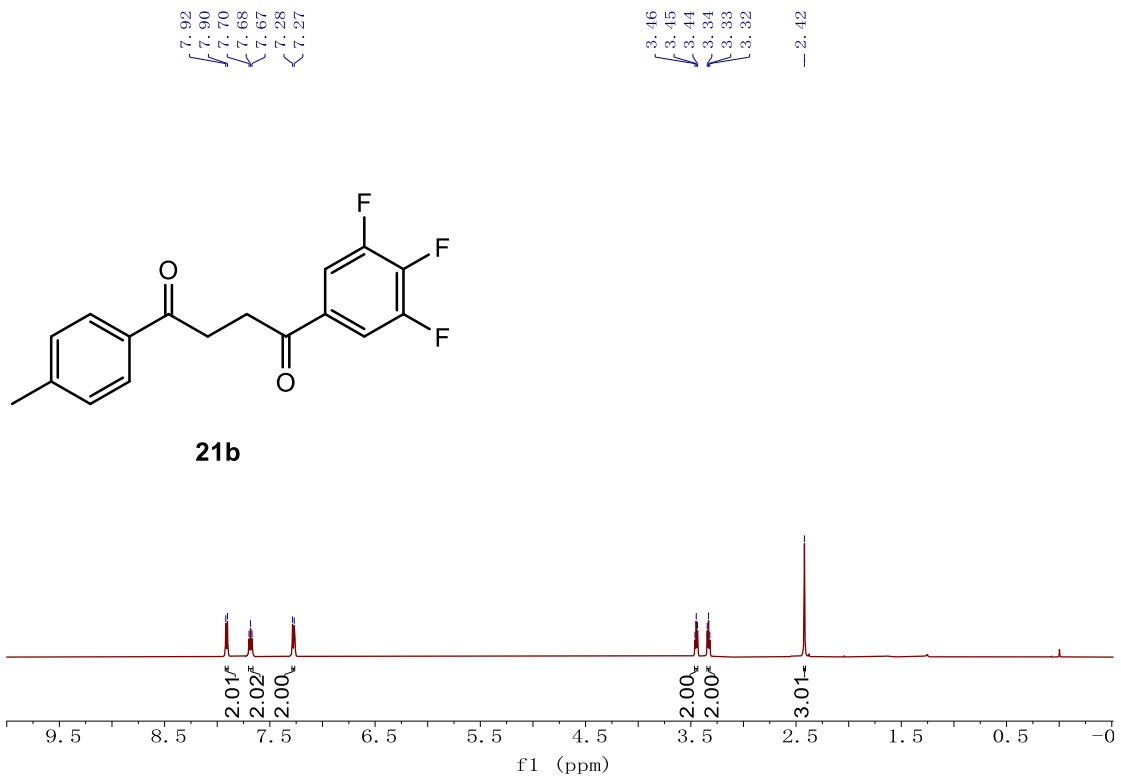


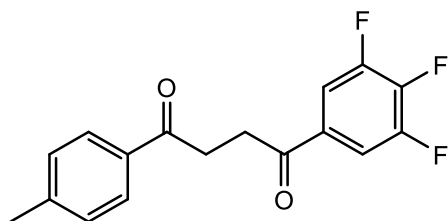






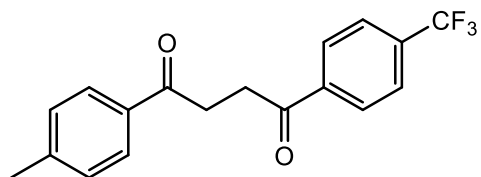
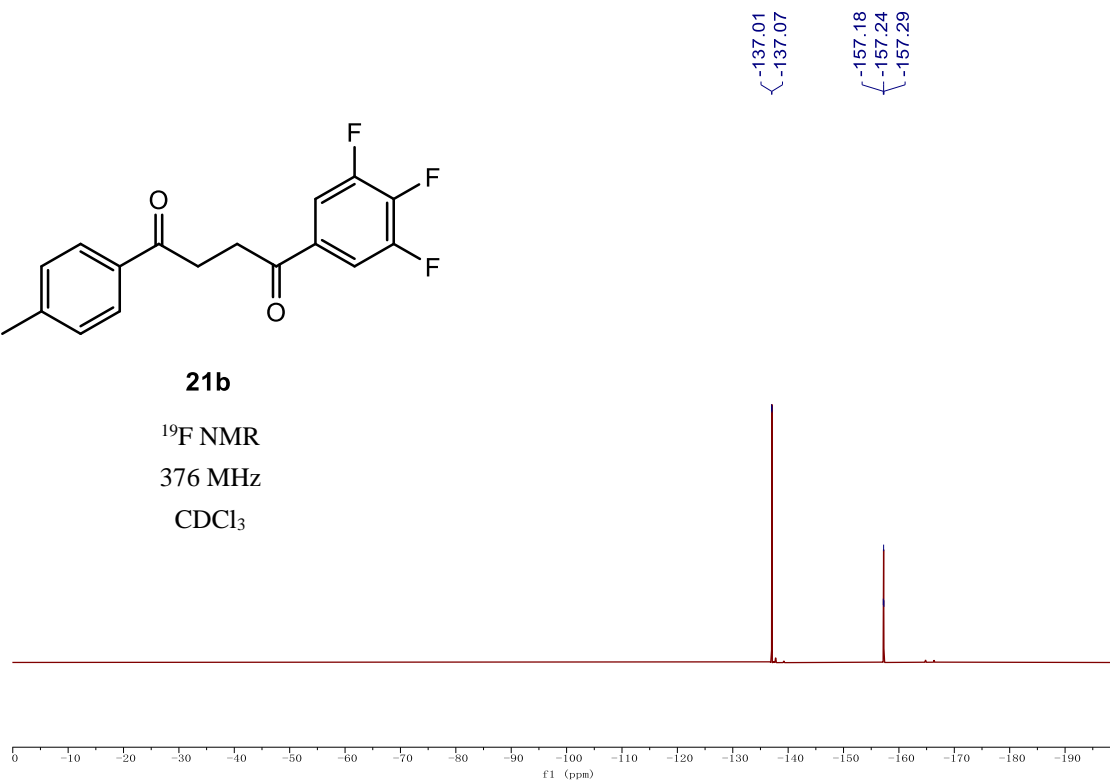




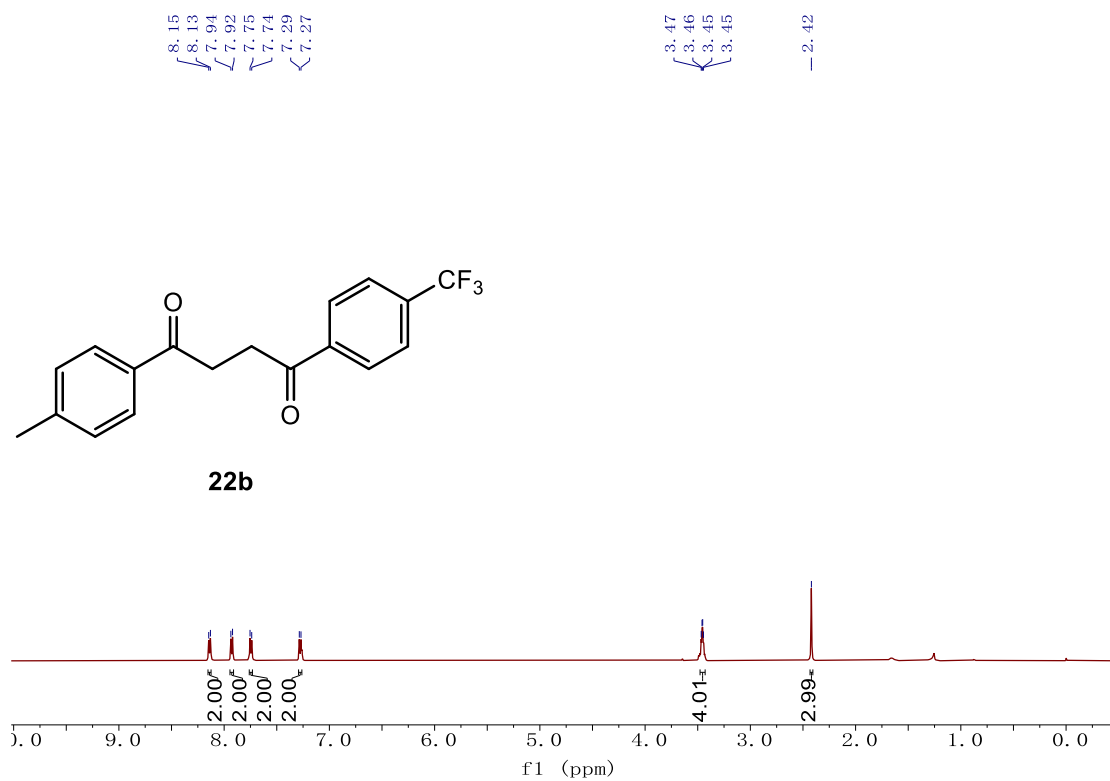


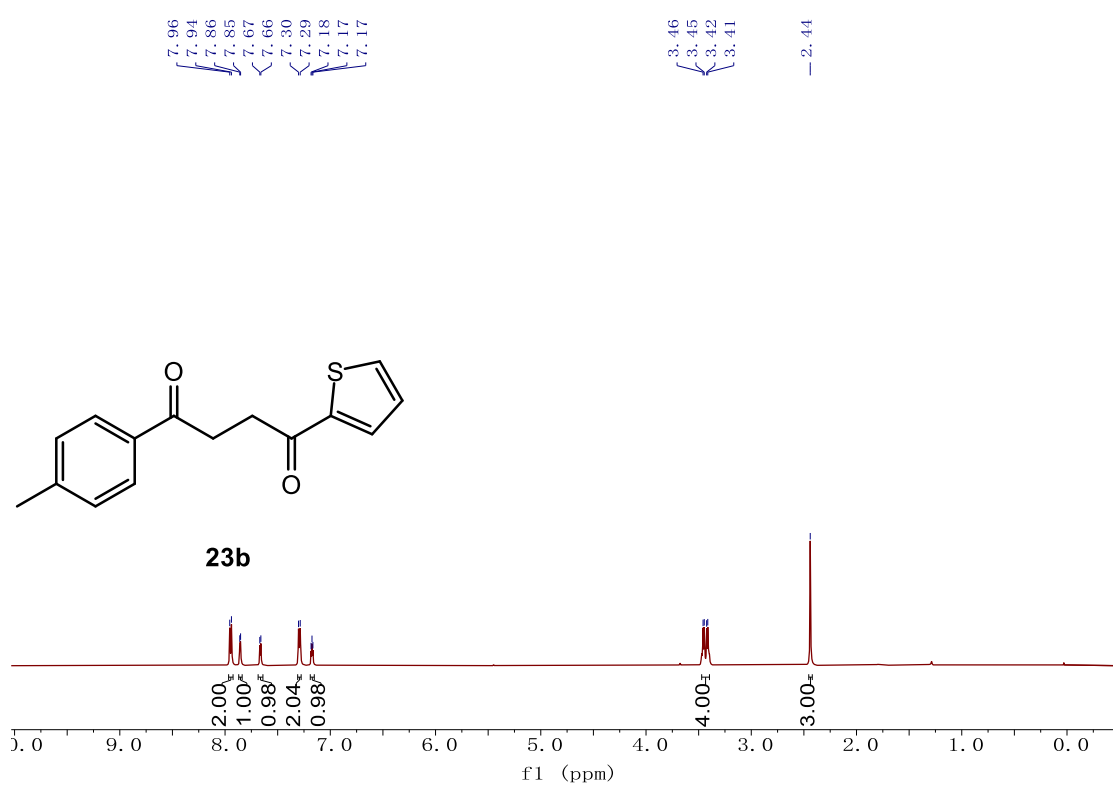
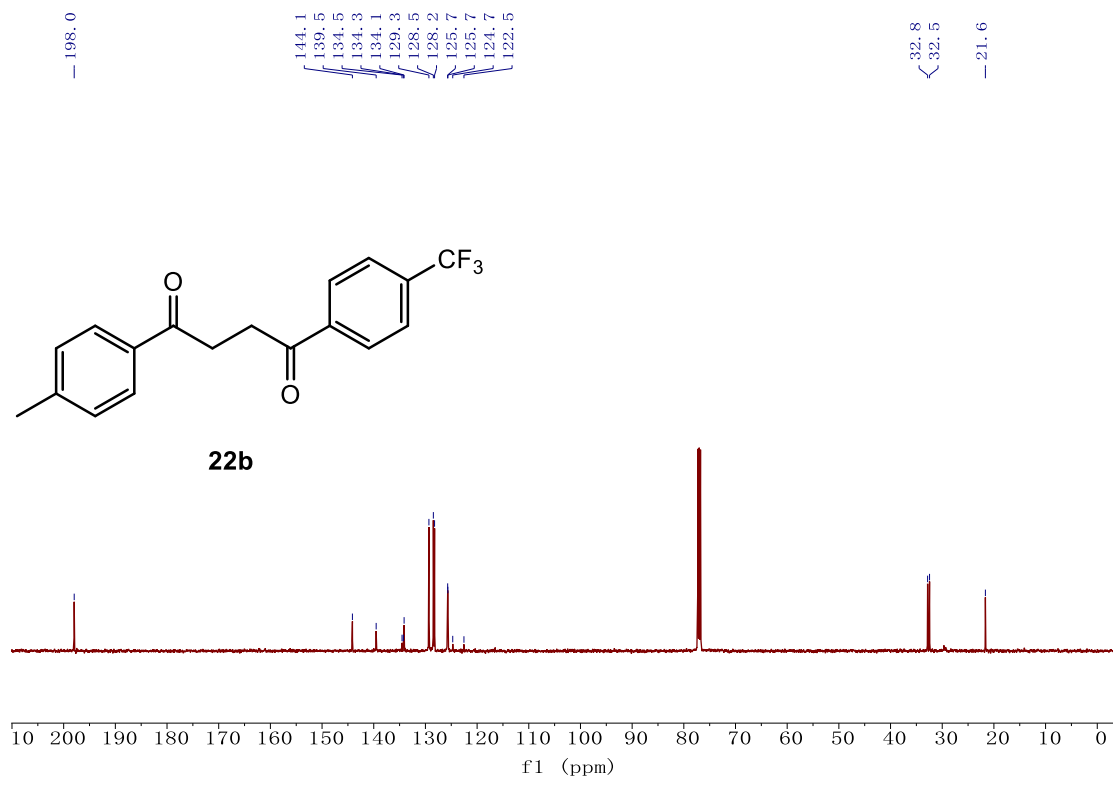
21b

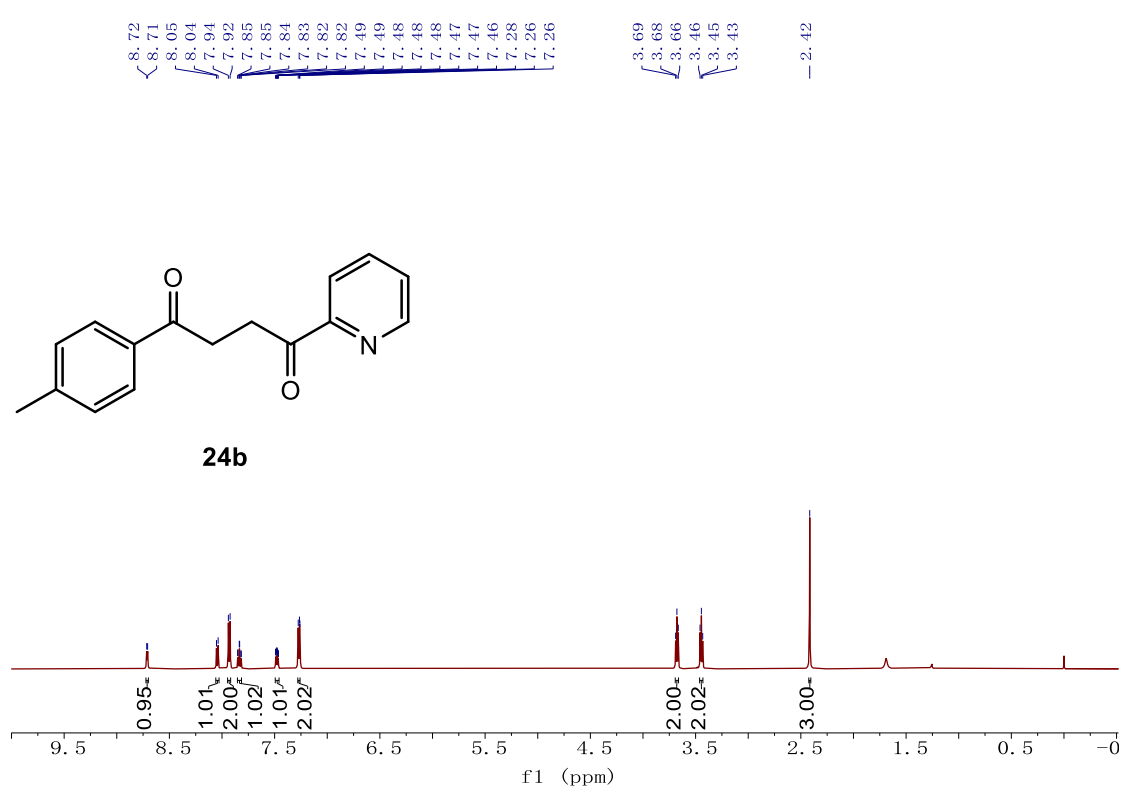
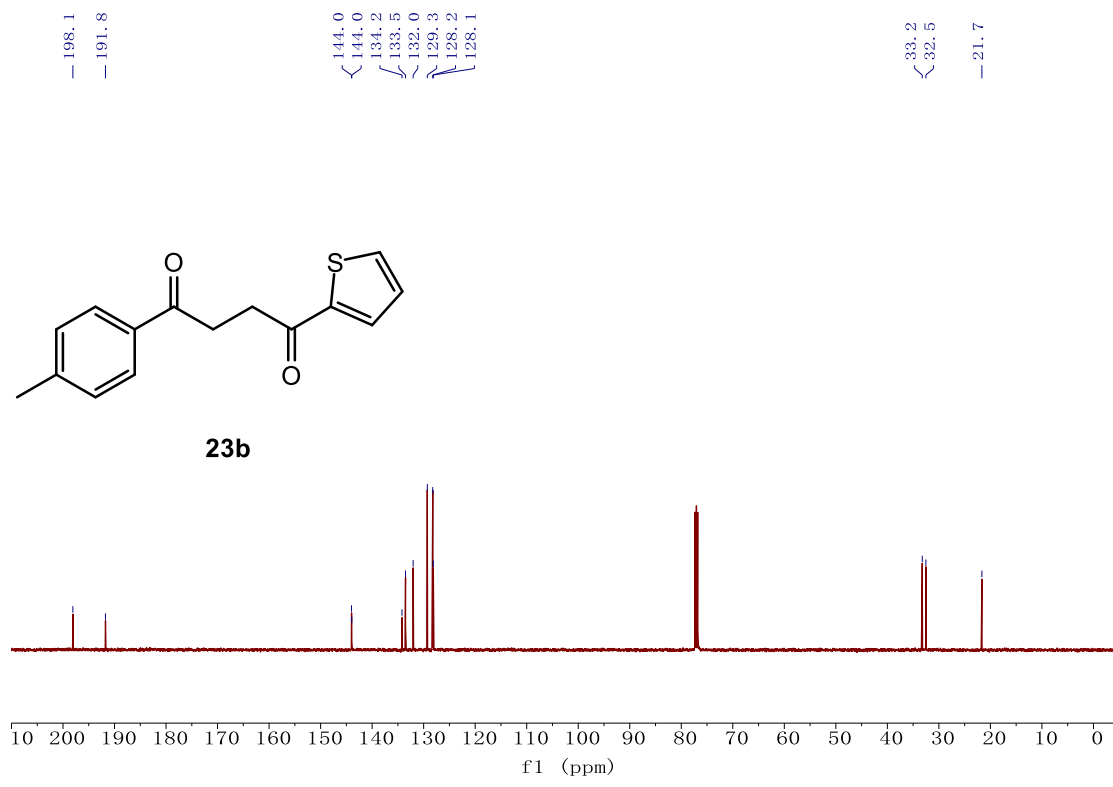
^{19}F NMR
376 MHz
 CDCl_3

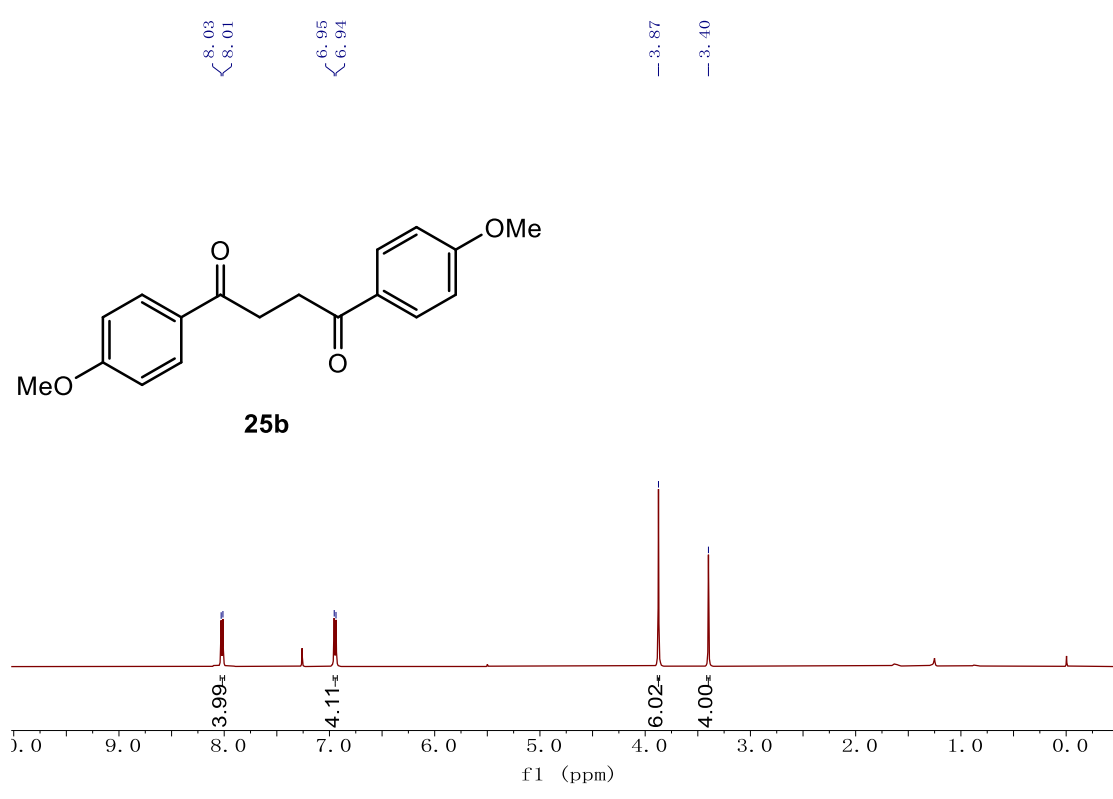
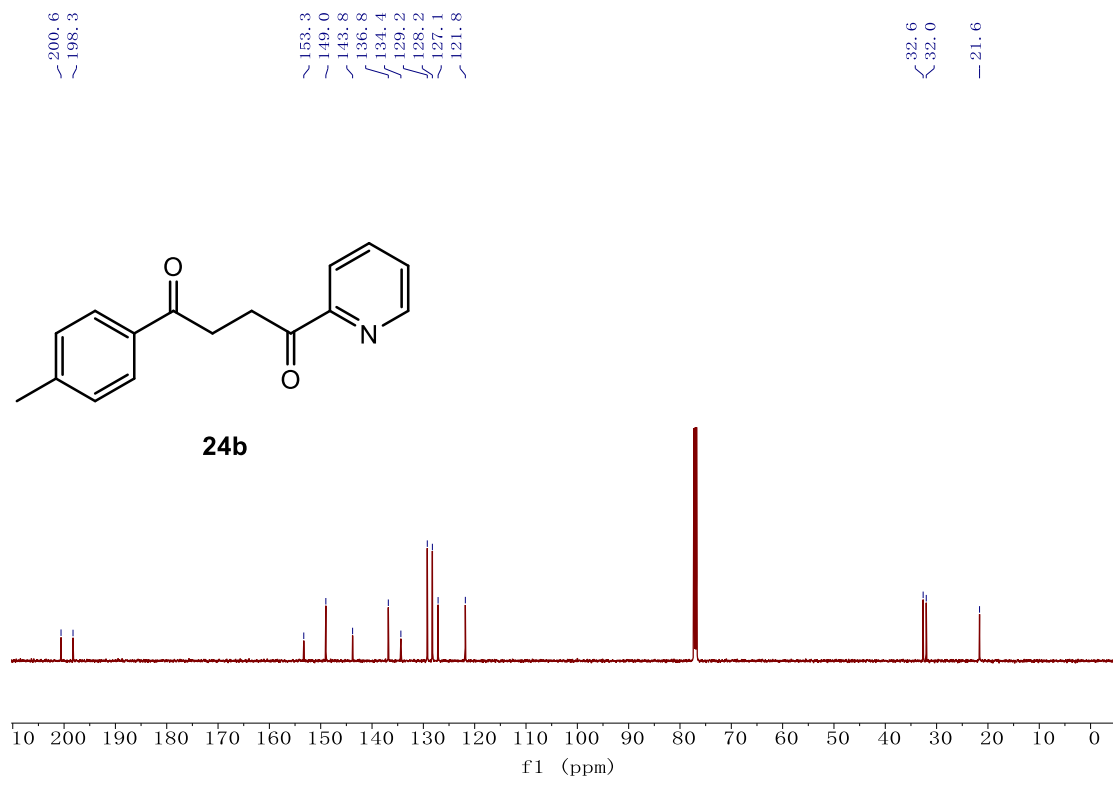


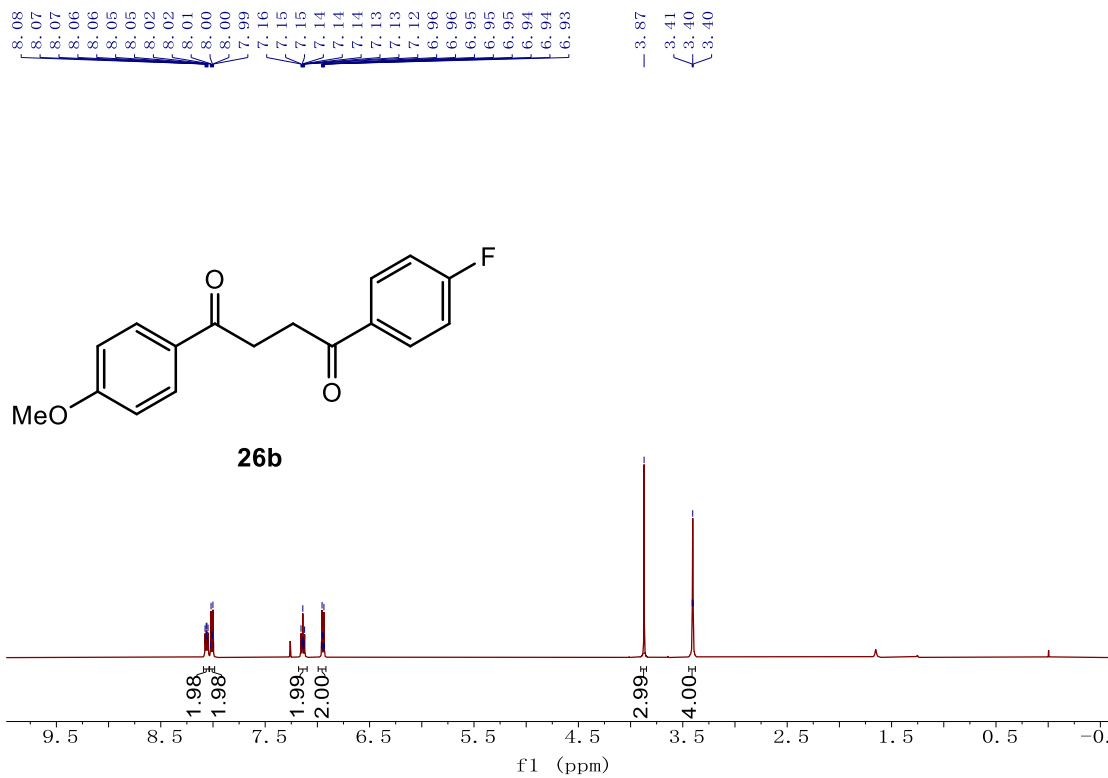
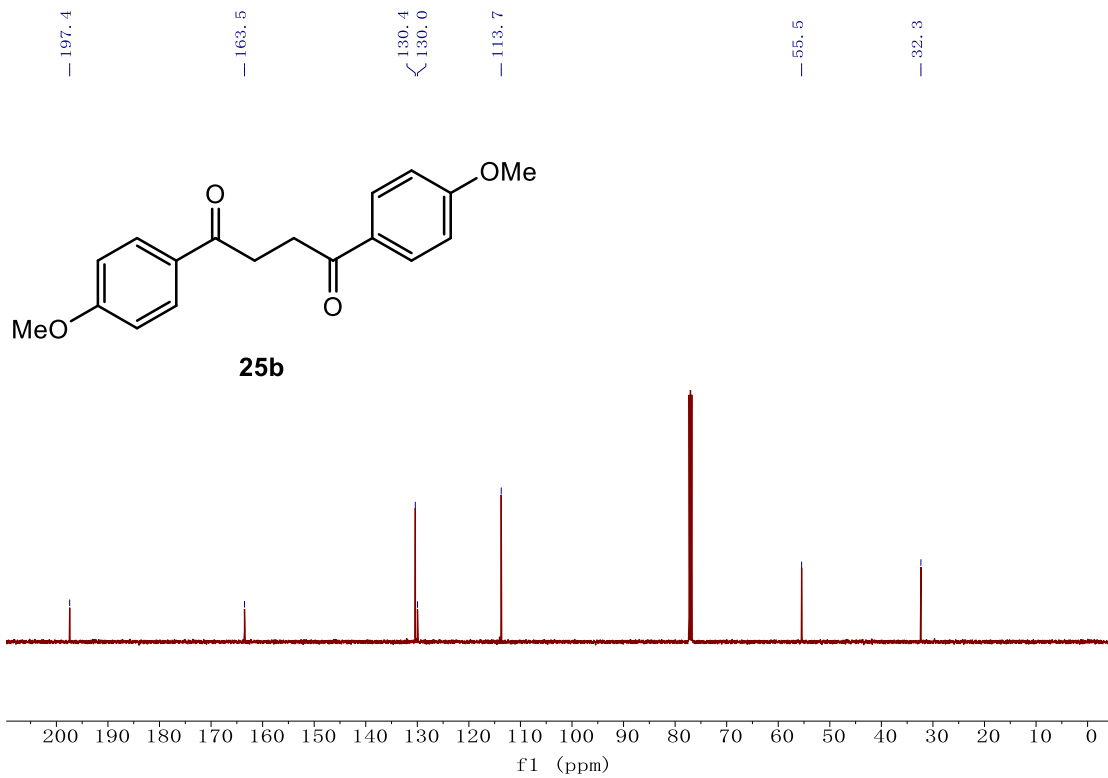
22b

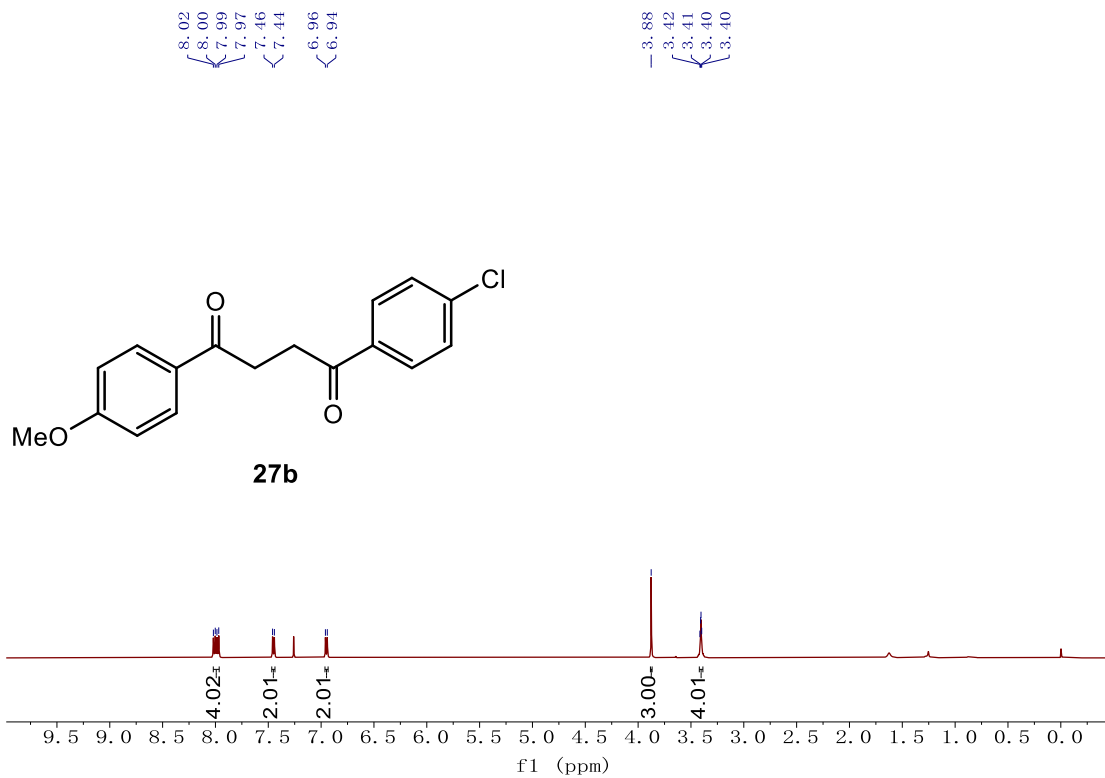
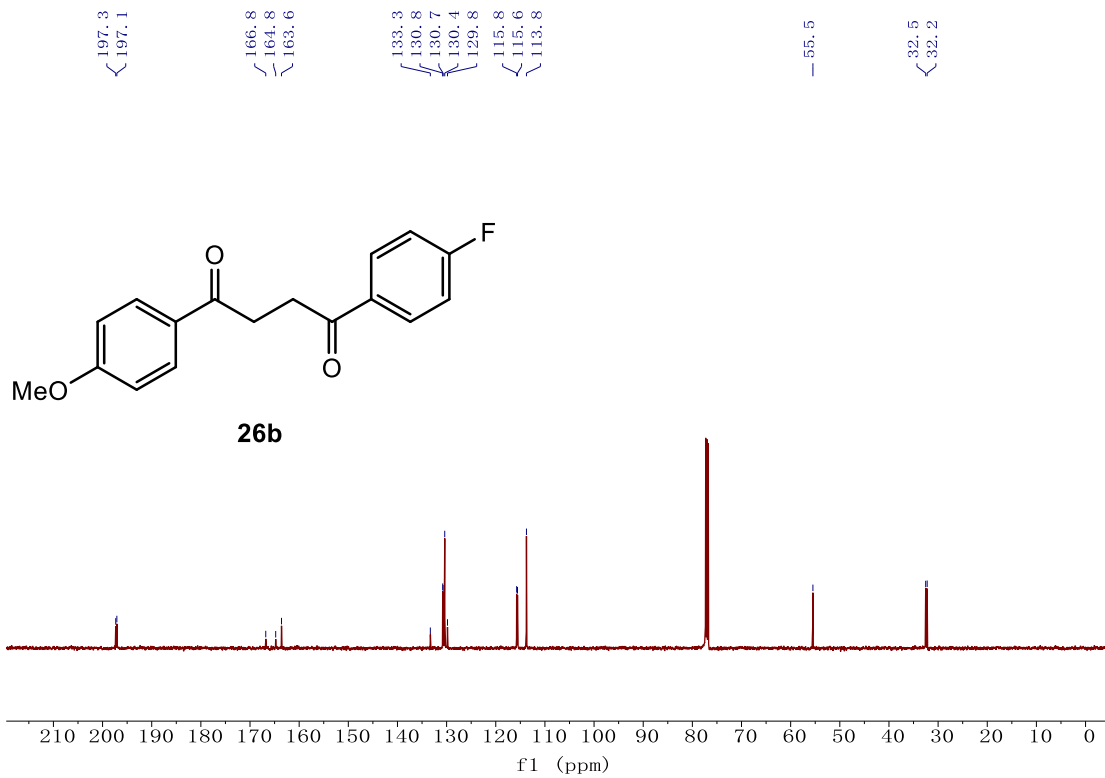


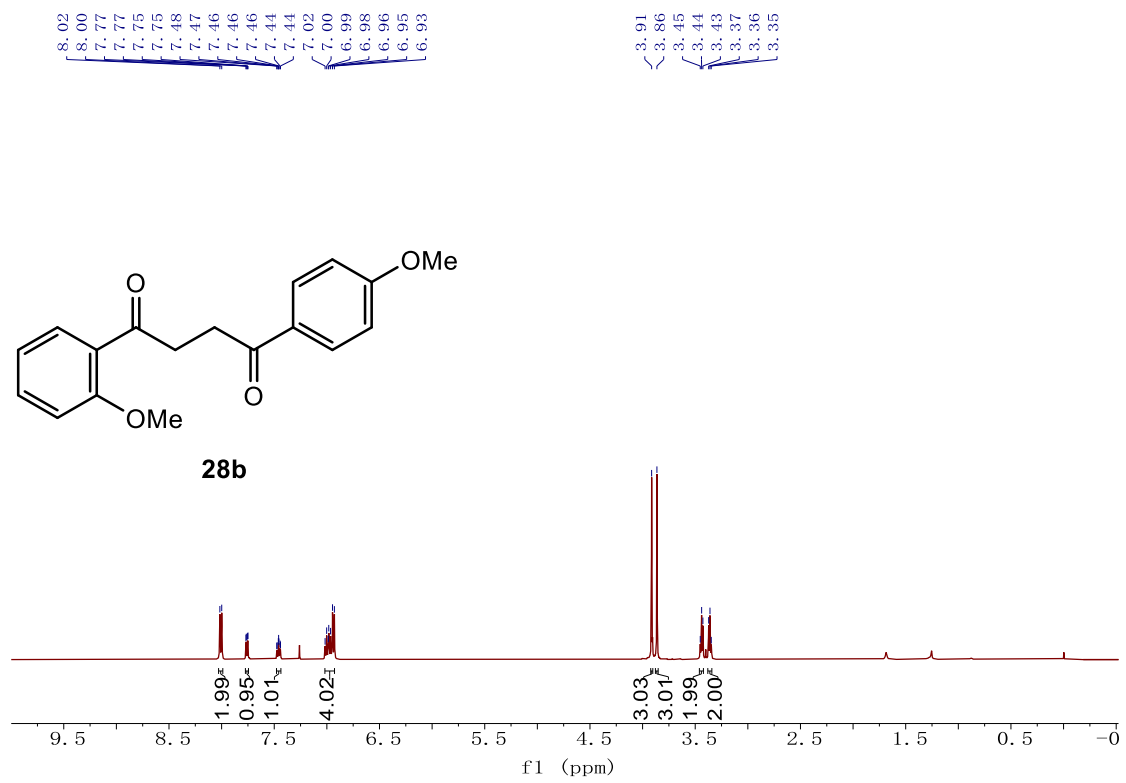
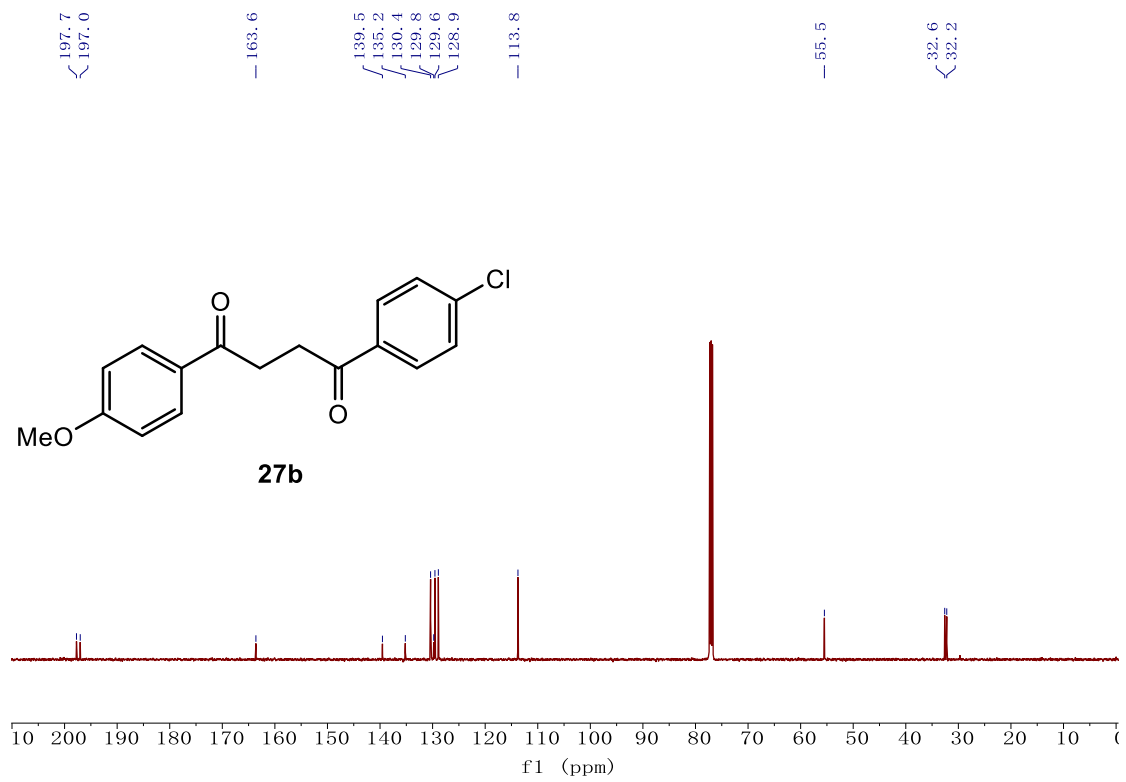


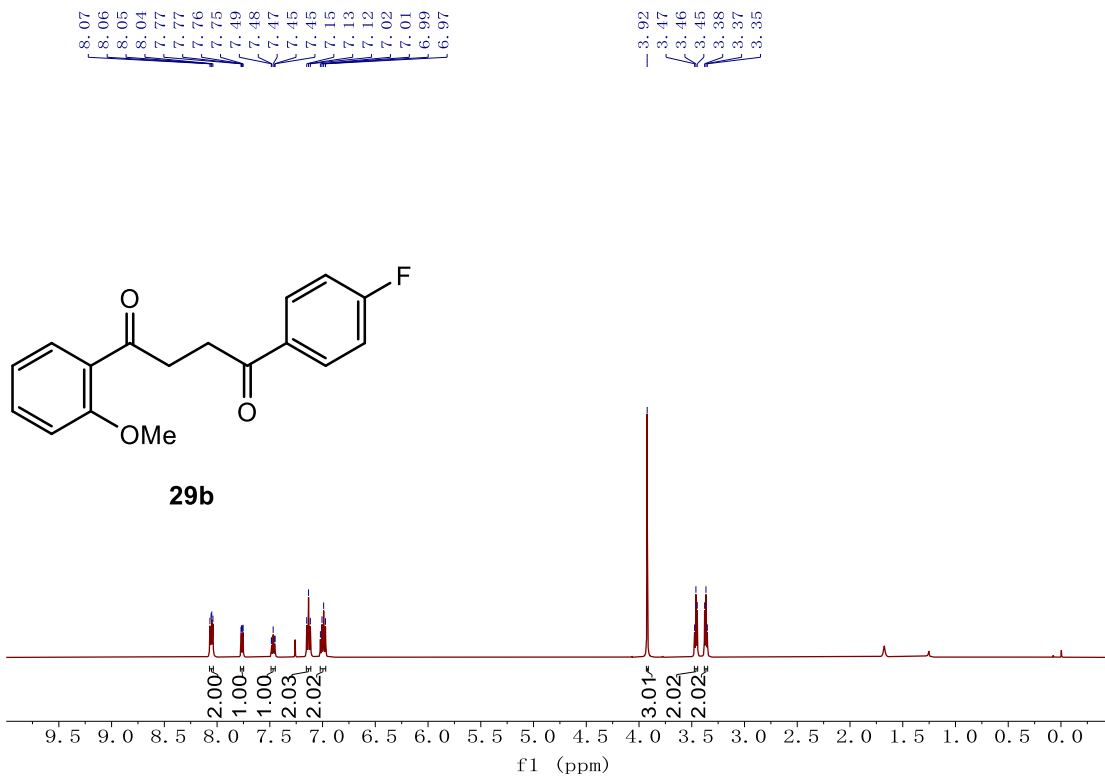
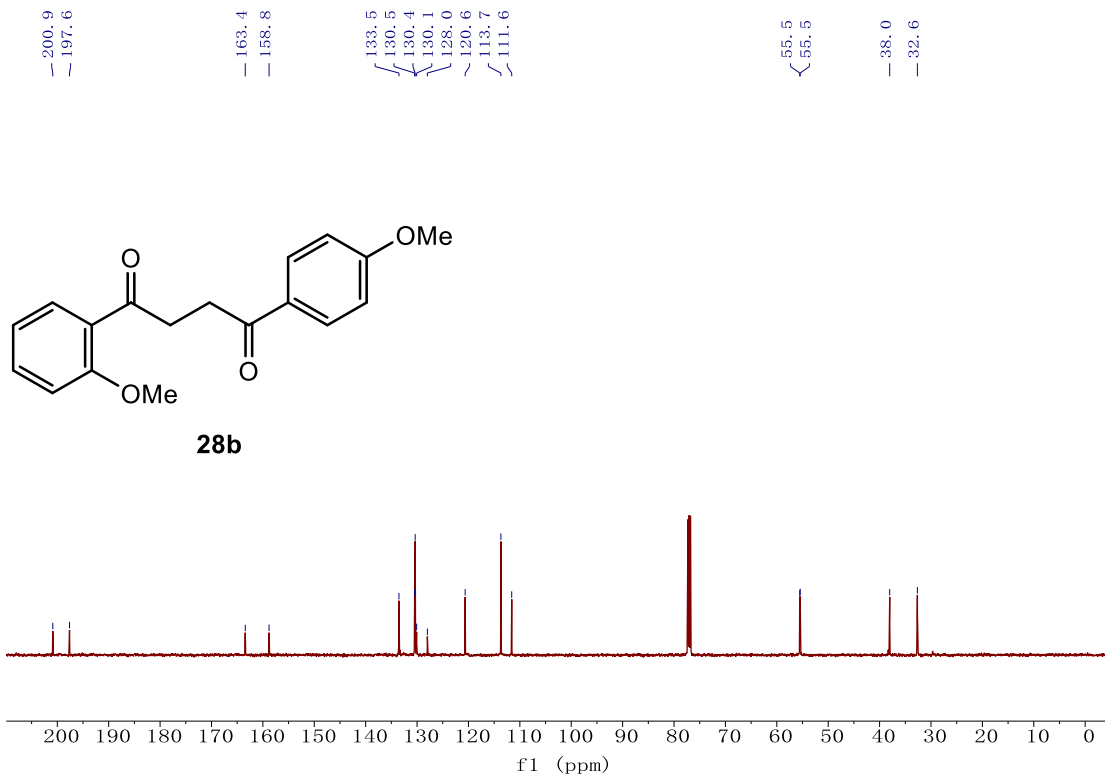


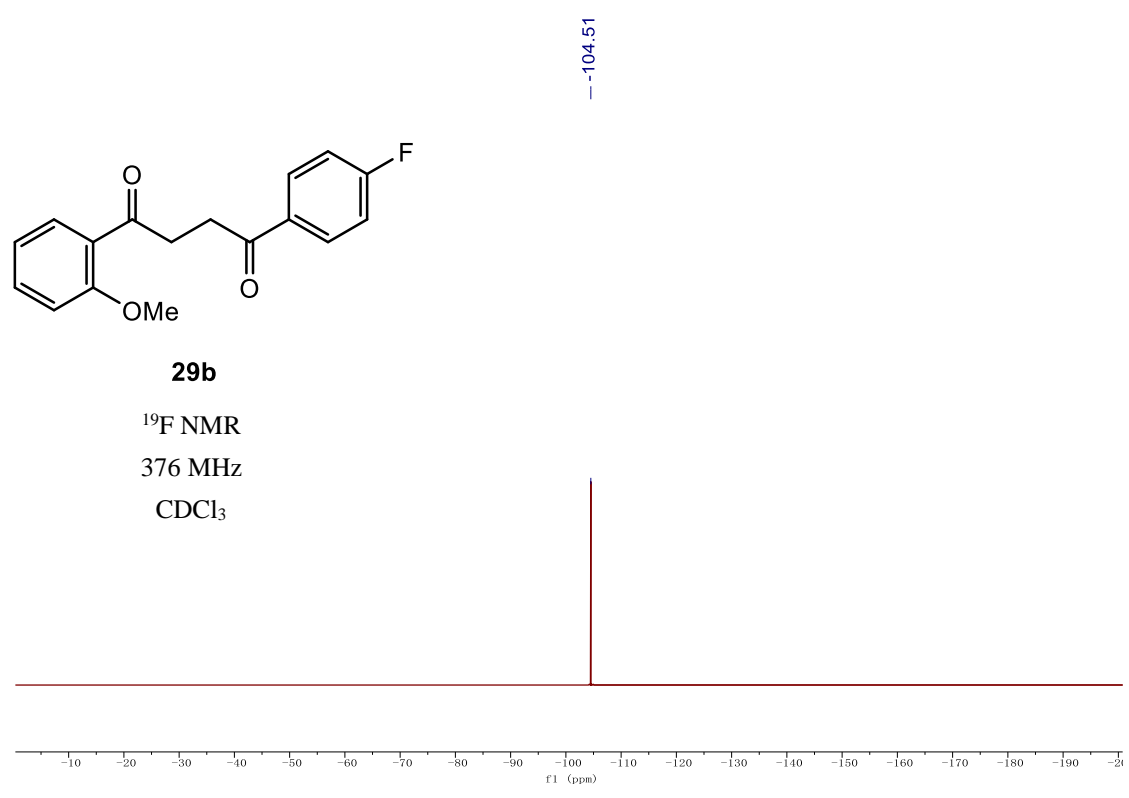
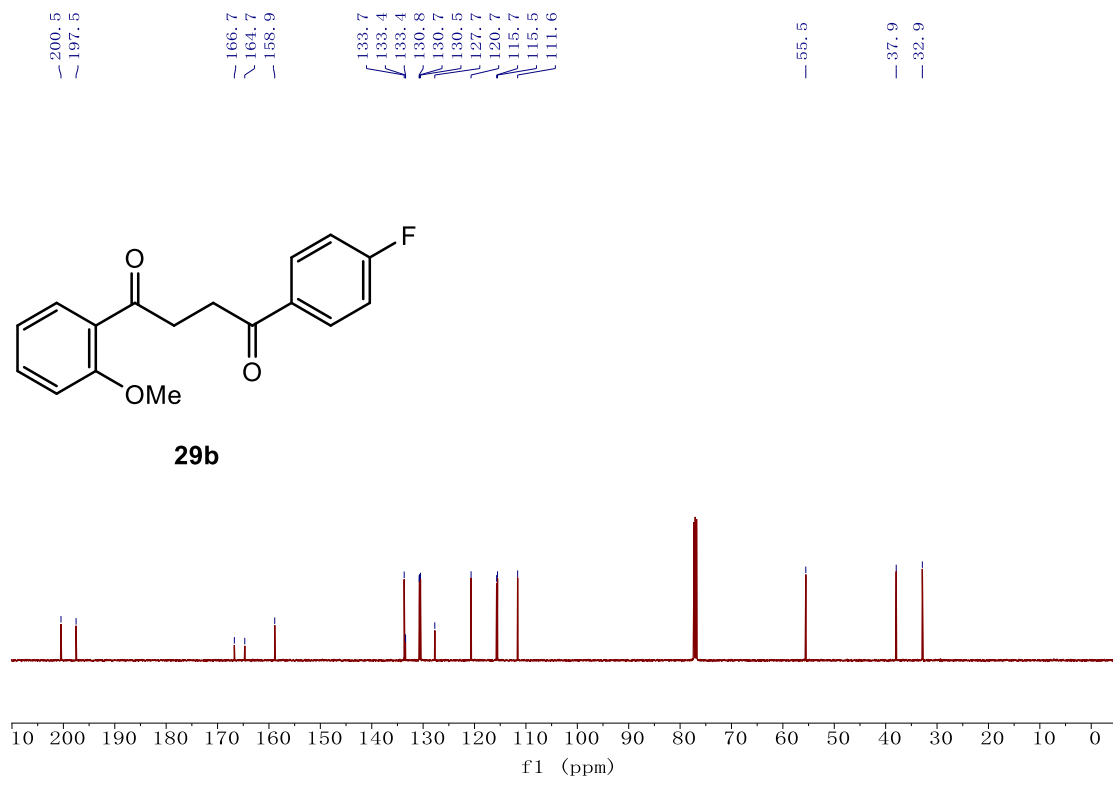


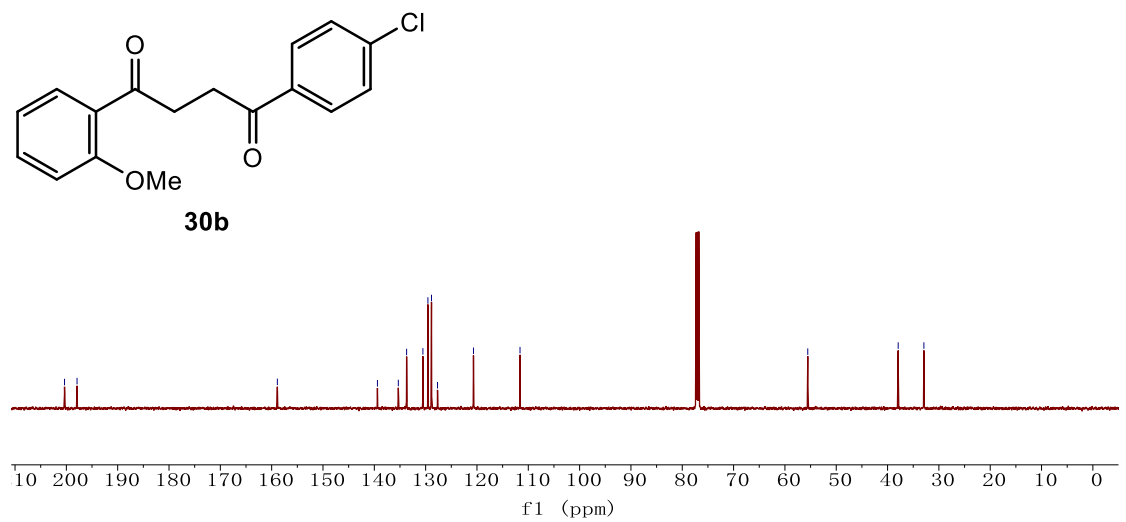
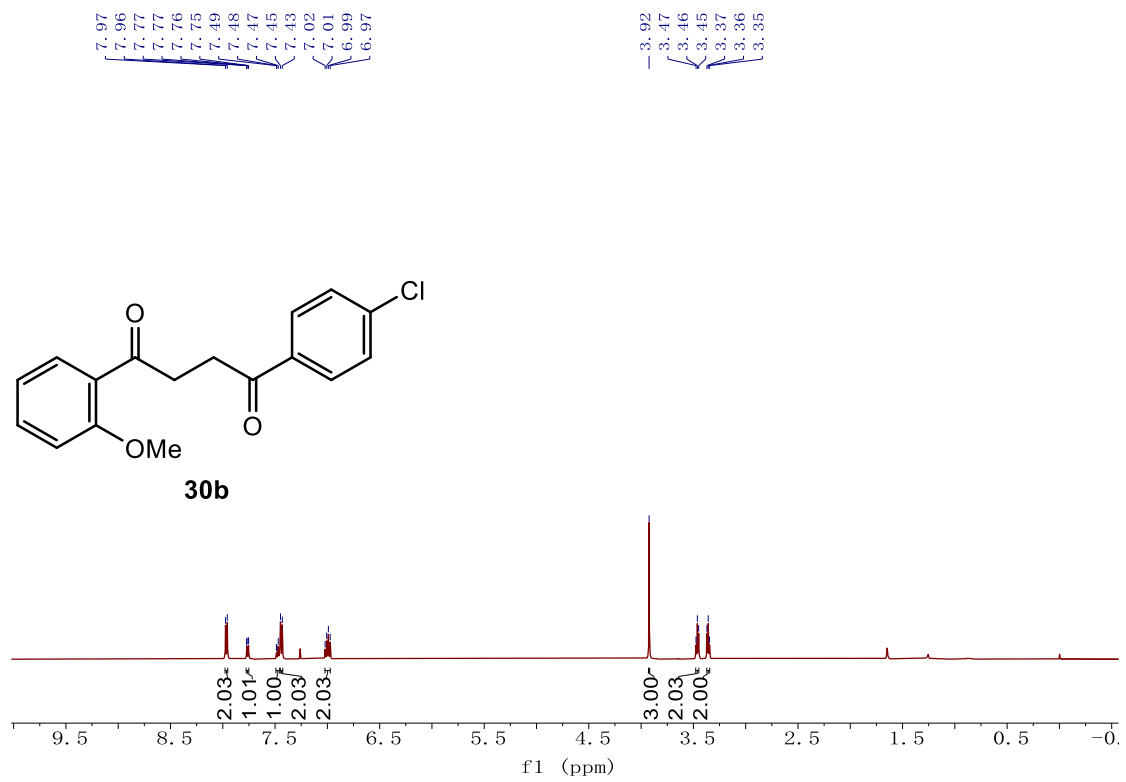


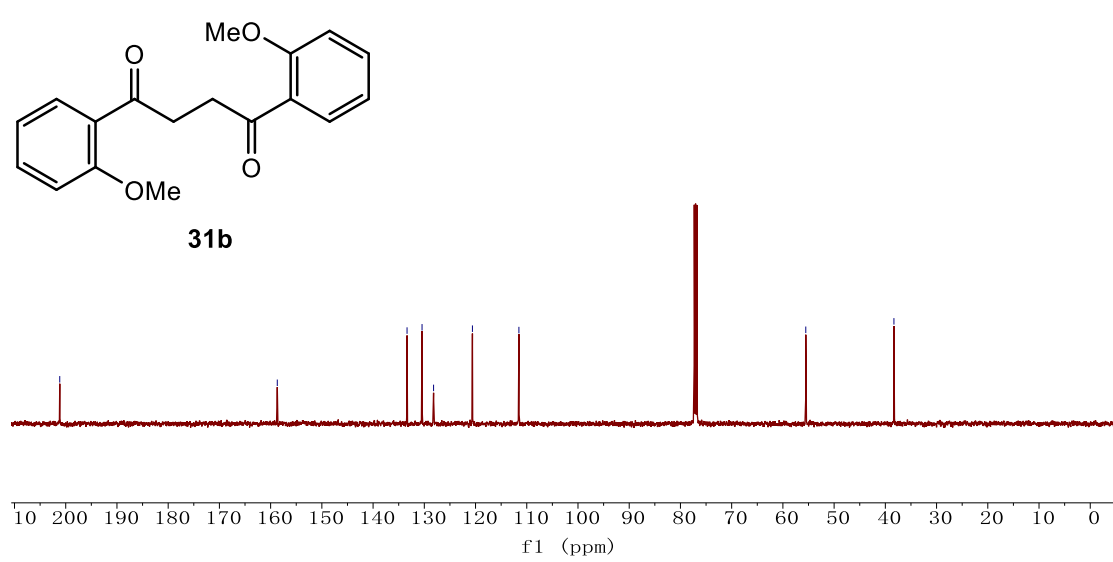
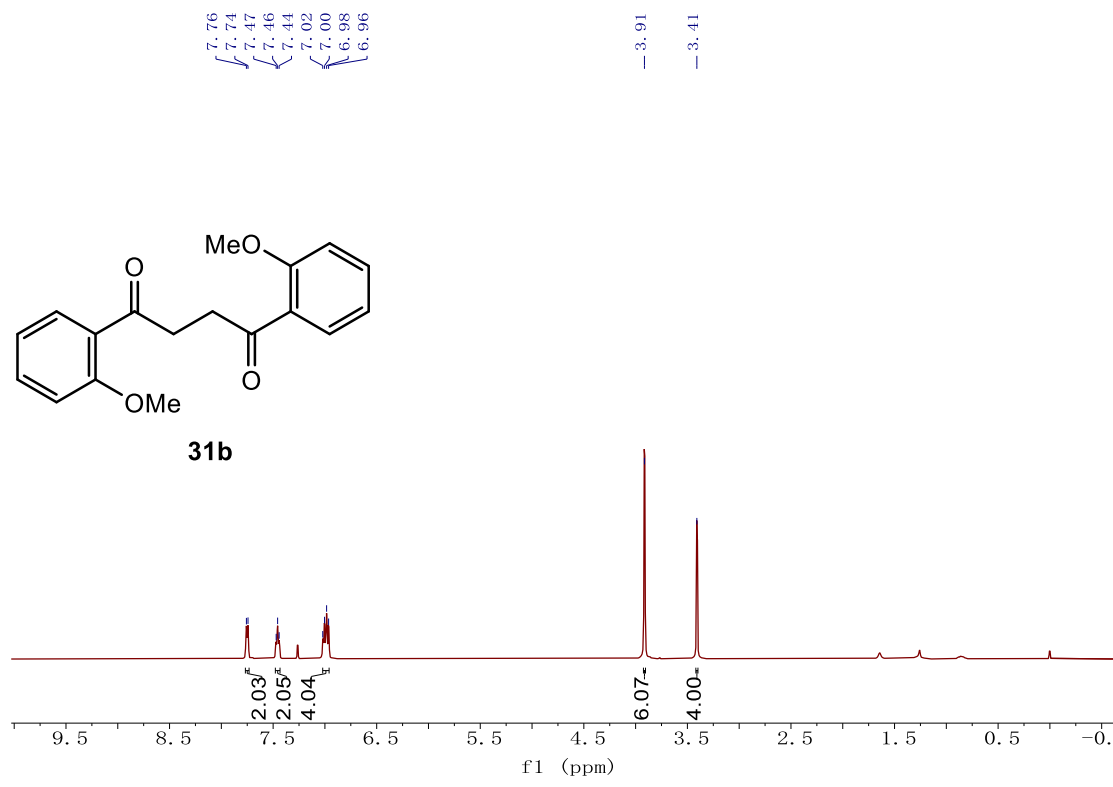






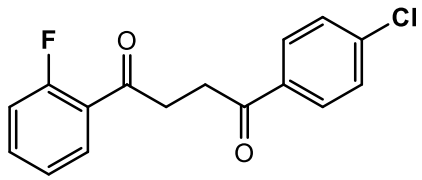




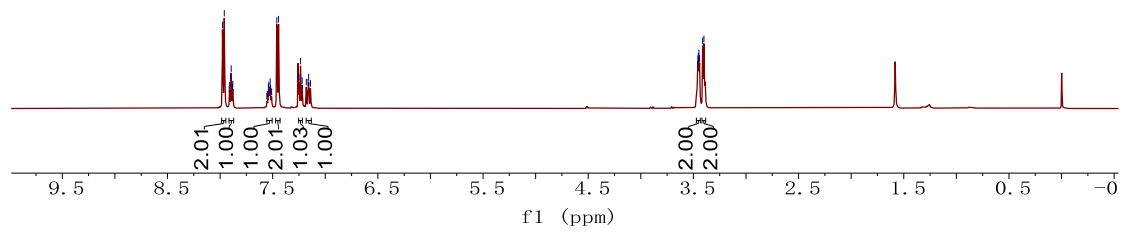


7.98
7.96
7.91
7.91
7.90
7.89
7.88
7.88
7.86
7.56
7.55
7.55
7.54
7.54
7.52
7.52
7.51
7.51
7.46
7.44
7.44
7.25
7.24
7.22
7.18
7.16
7.16
7.14

3.46
3.46
3.45
3.45
3.44
3.41
3.40



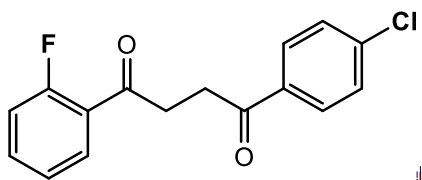
32b



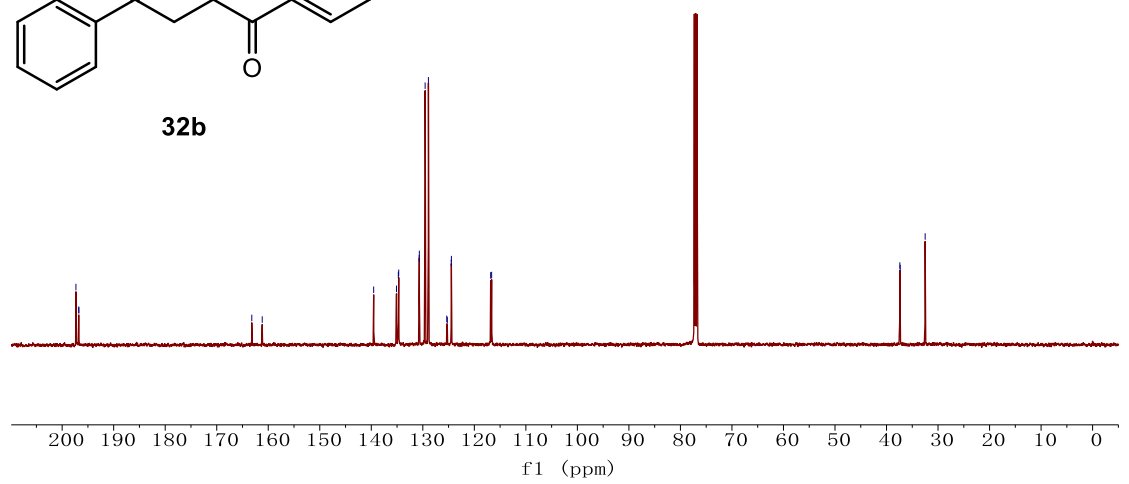
197.3
196.8
196.8

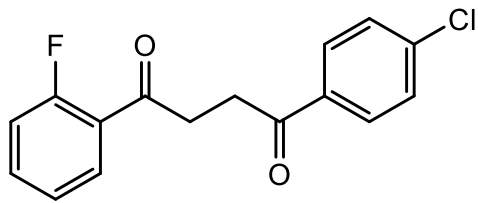
163.2
161.2
139.5
135.1
134.8
134.7
130.7
130.7
129.6
128.9
125.4
125.2
124.5
124.5
116.8
116.6

37.4
37.3
32.5



32b



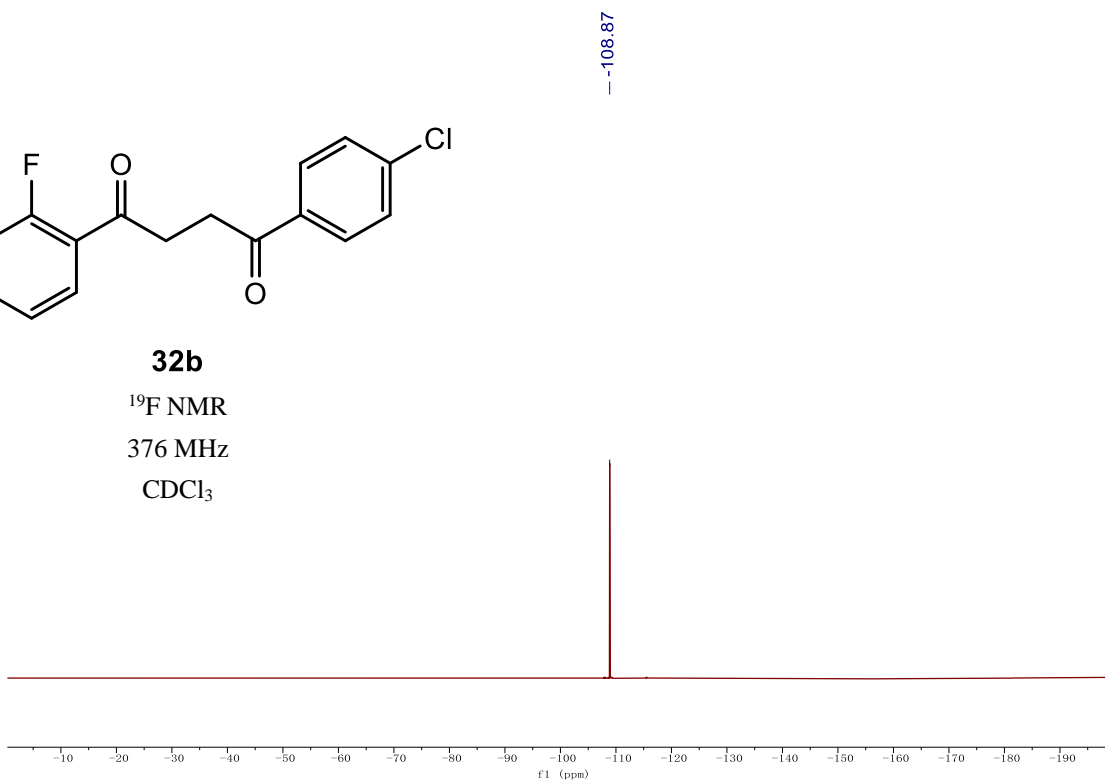


32b

¹⁹F NMR

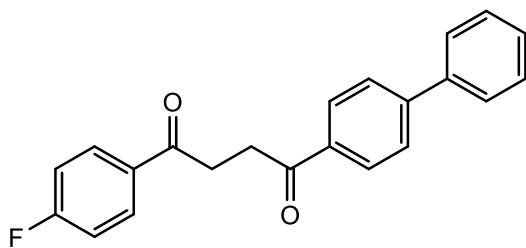
376 MHz

CDCl₃

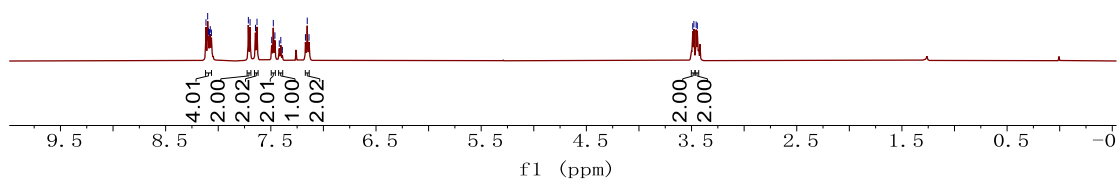


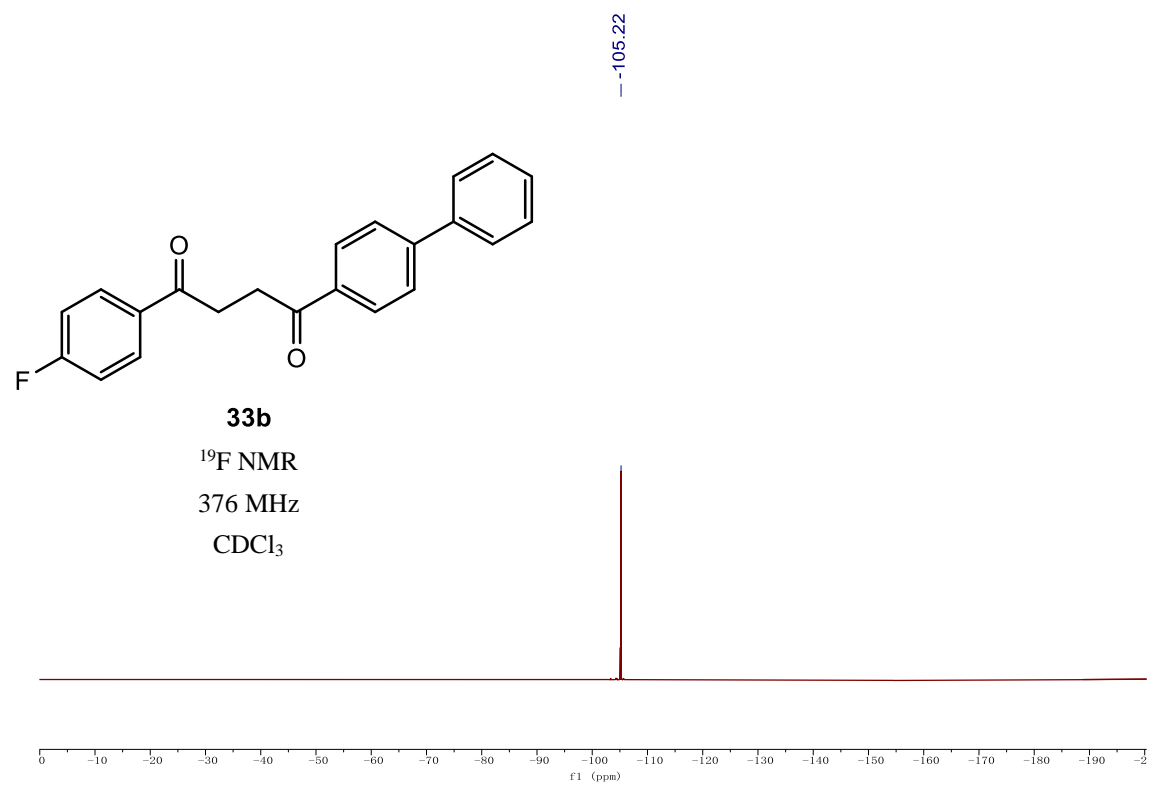
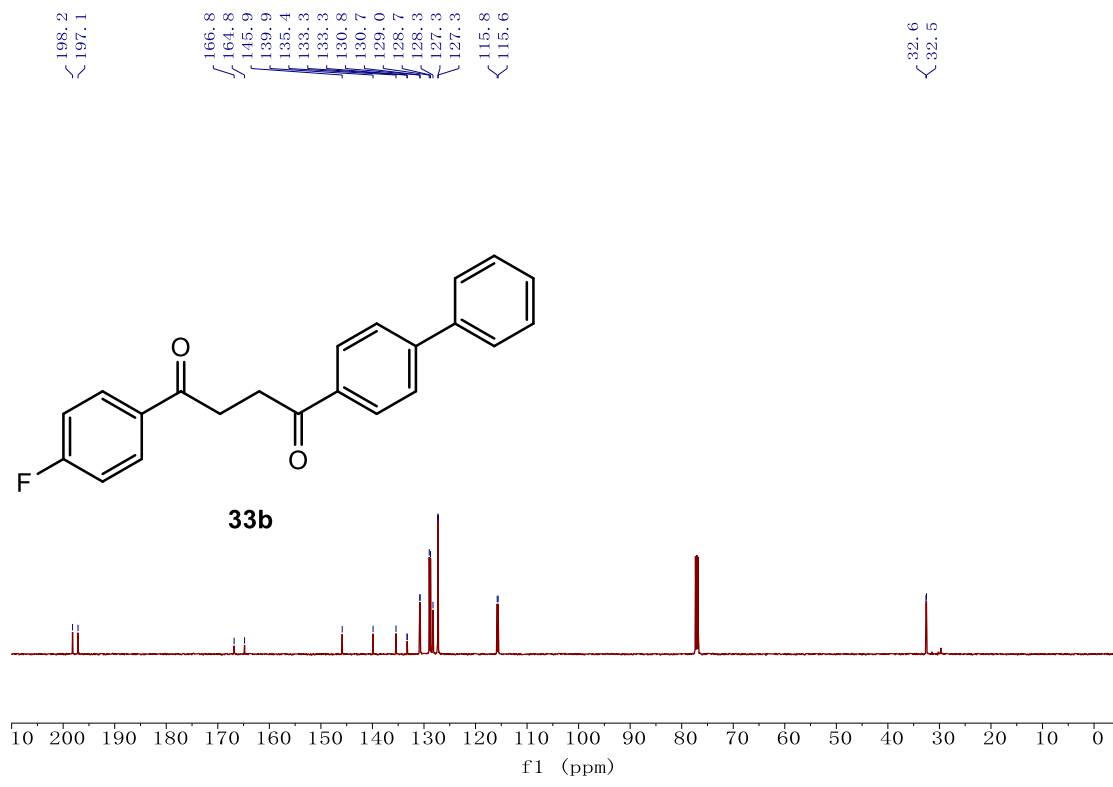
8.12
8.10
8.09
8.08
8.06
7.71
7.70
7.65
7.63
7.49
7.48
7.46
7.42
7.41
7.39
7.17
7.15
7.14

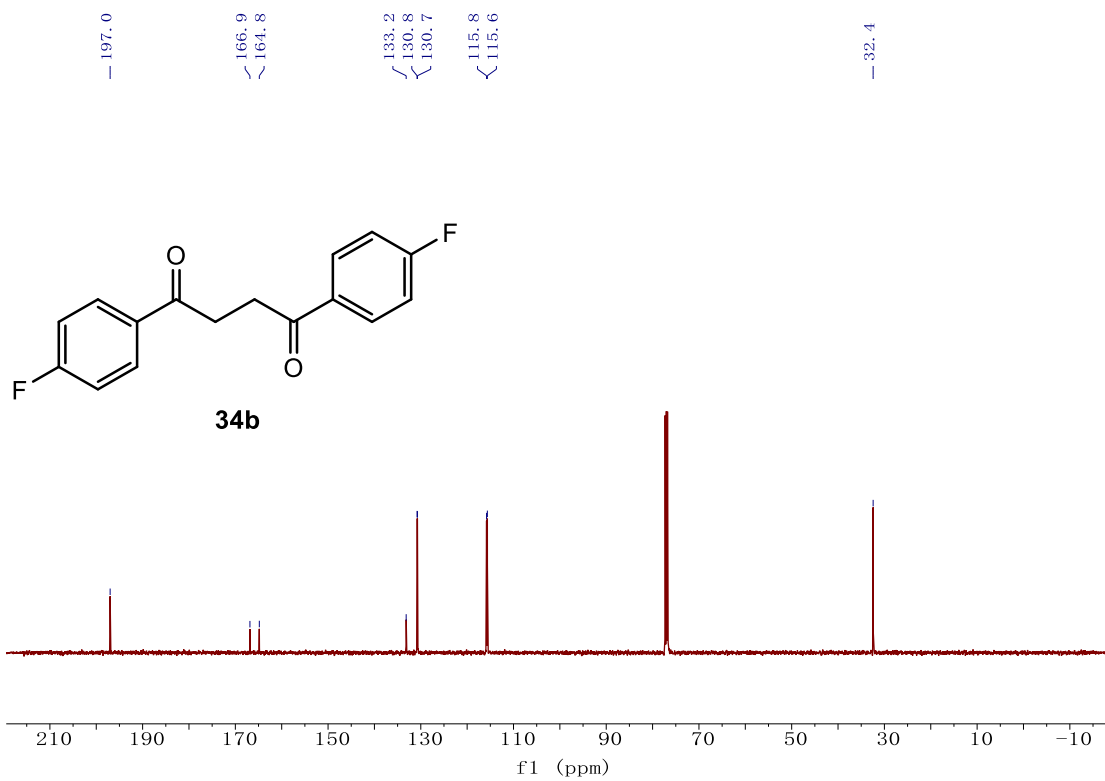
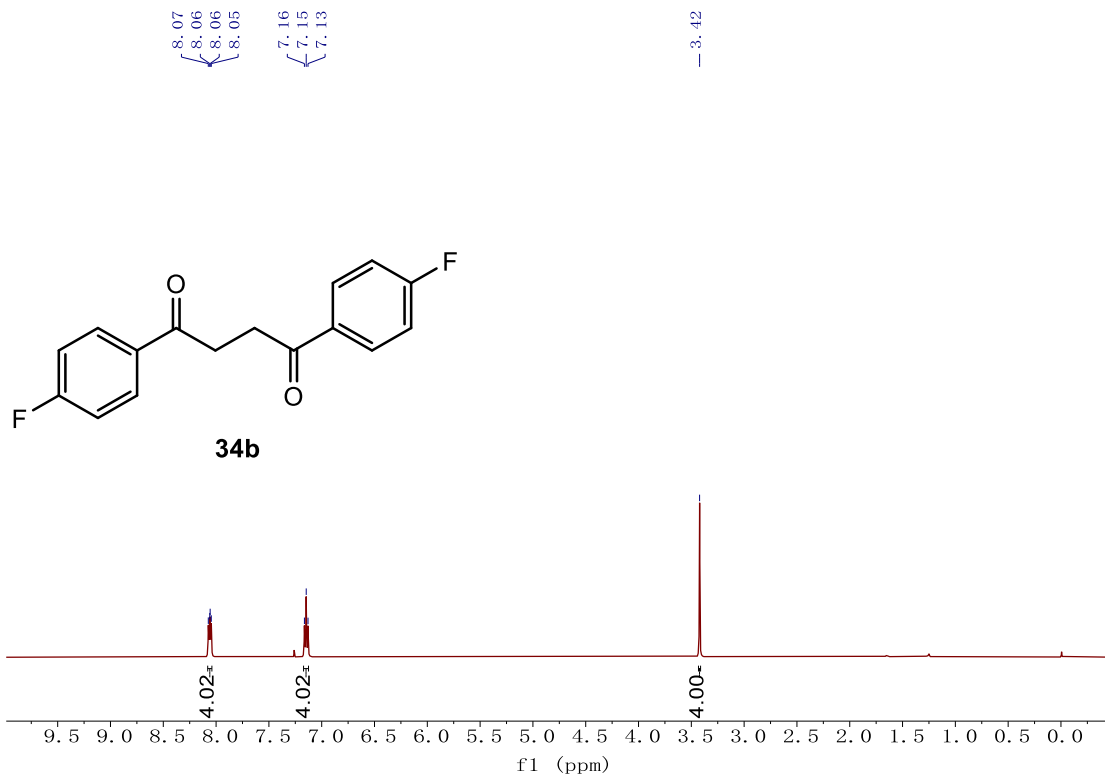
3.49
3.48
3.46
3.45

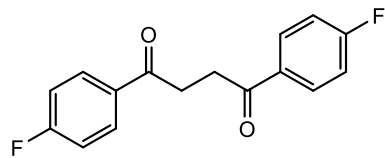


33b







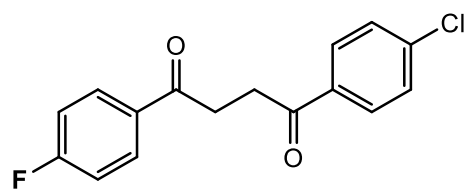
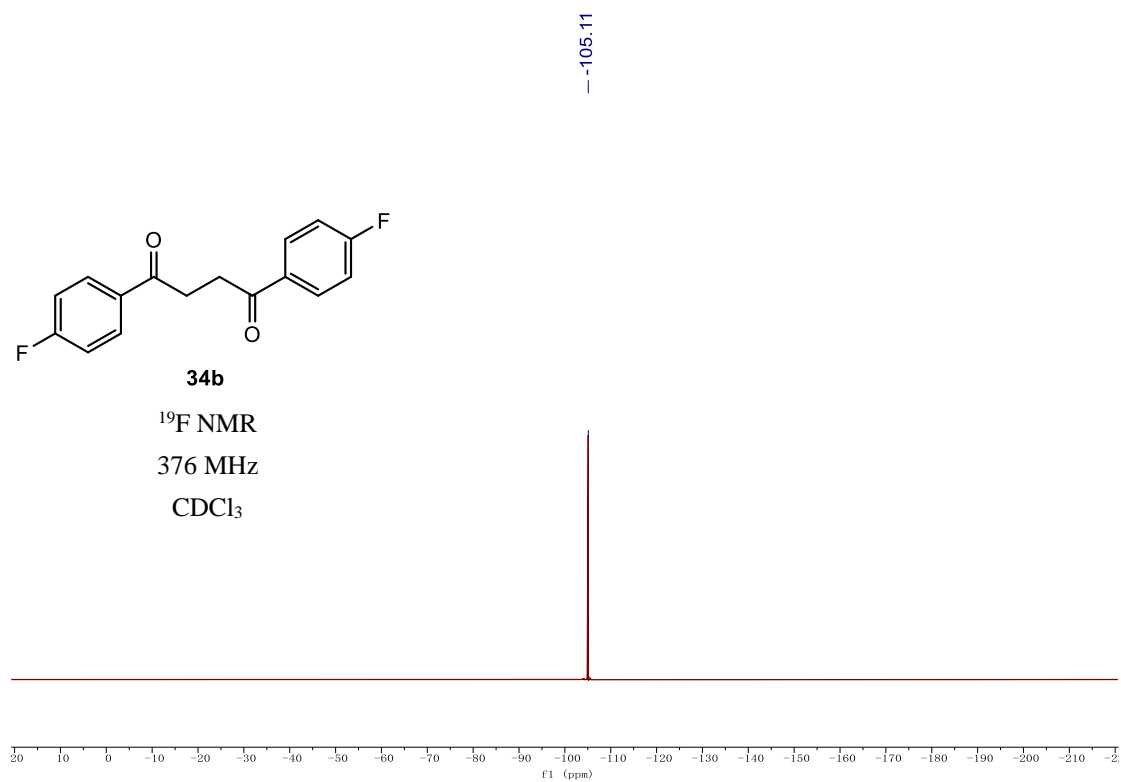


34b

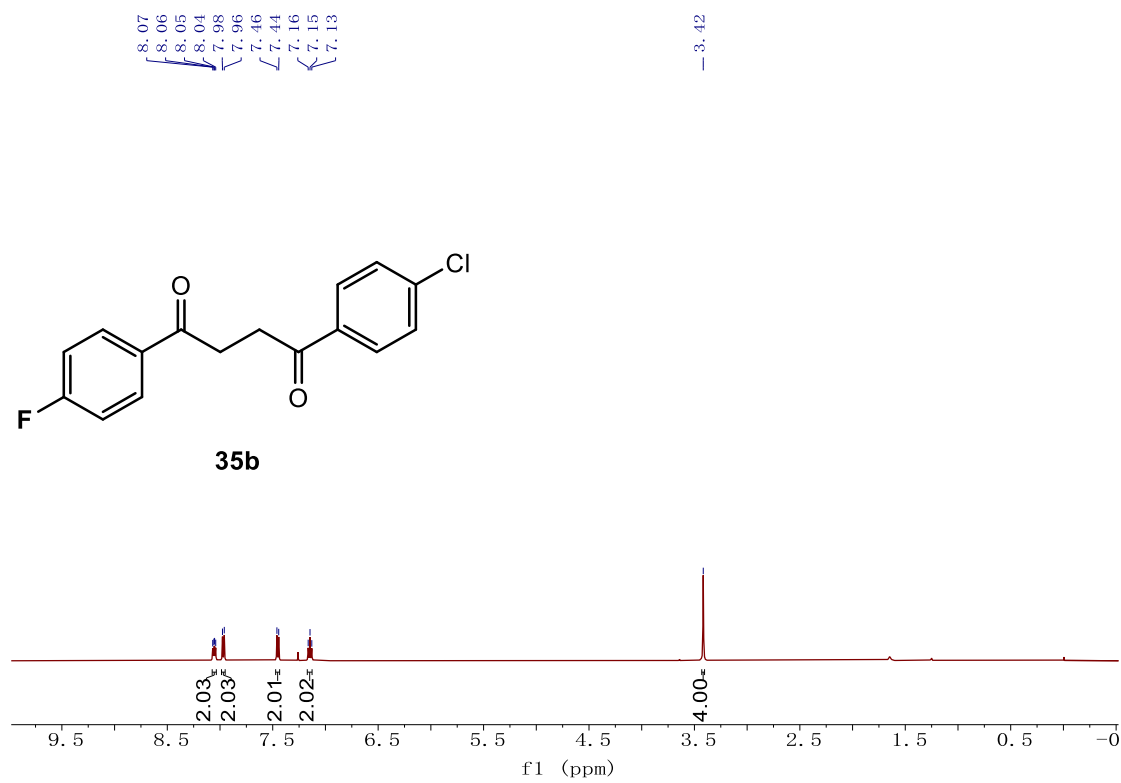
¹⁹F NMR

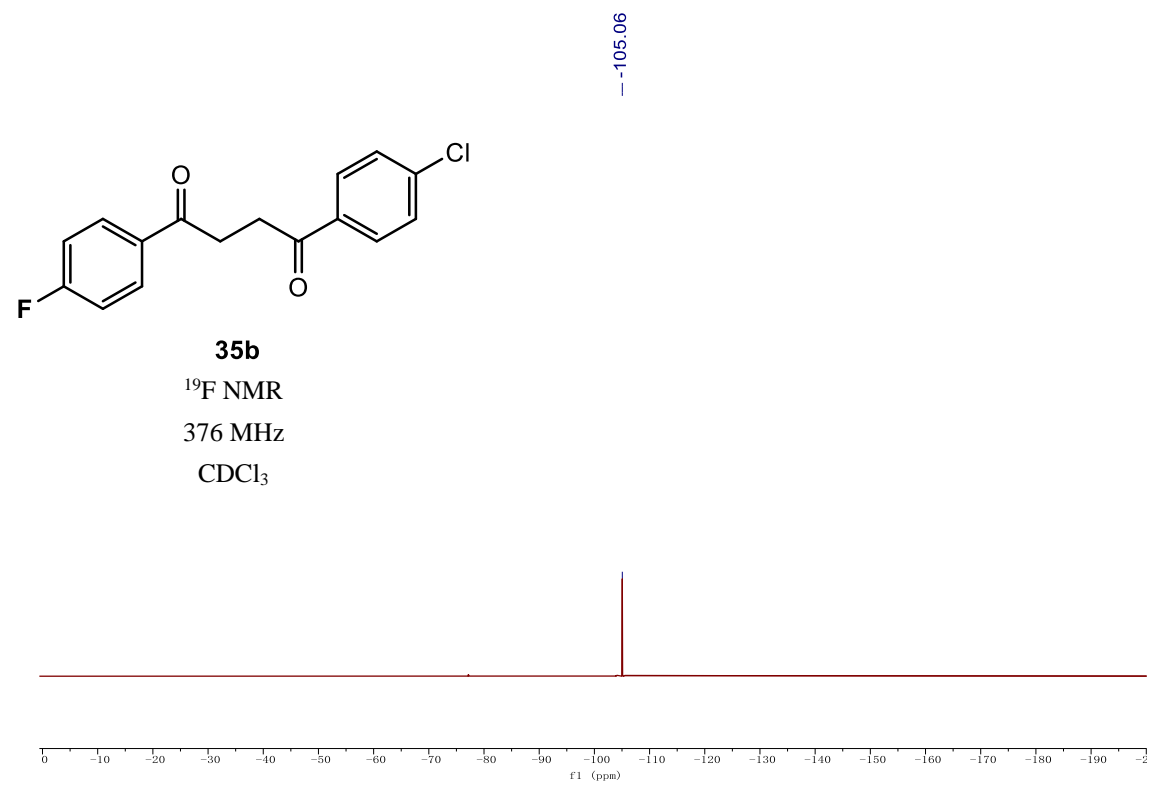
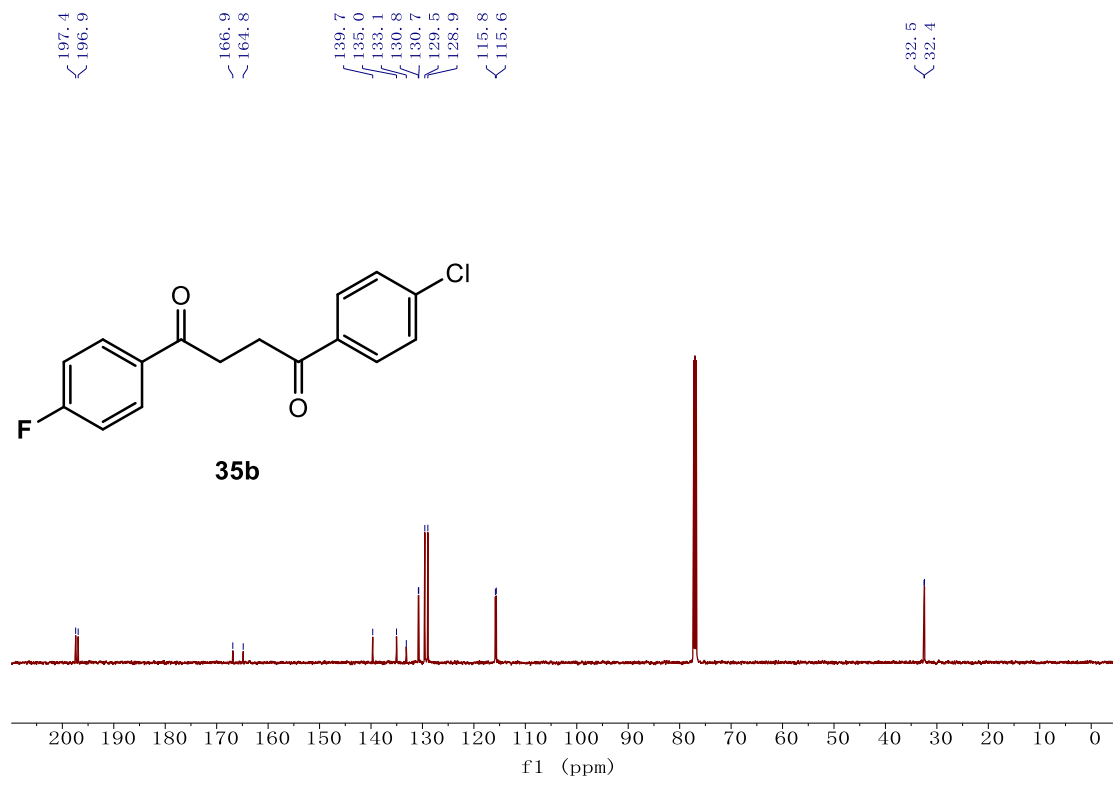
376 MHz

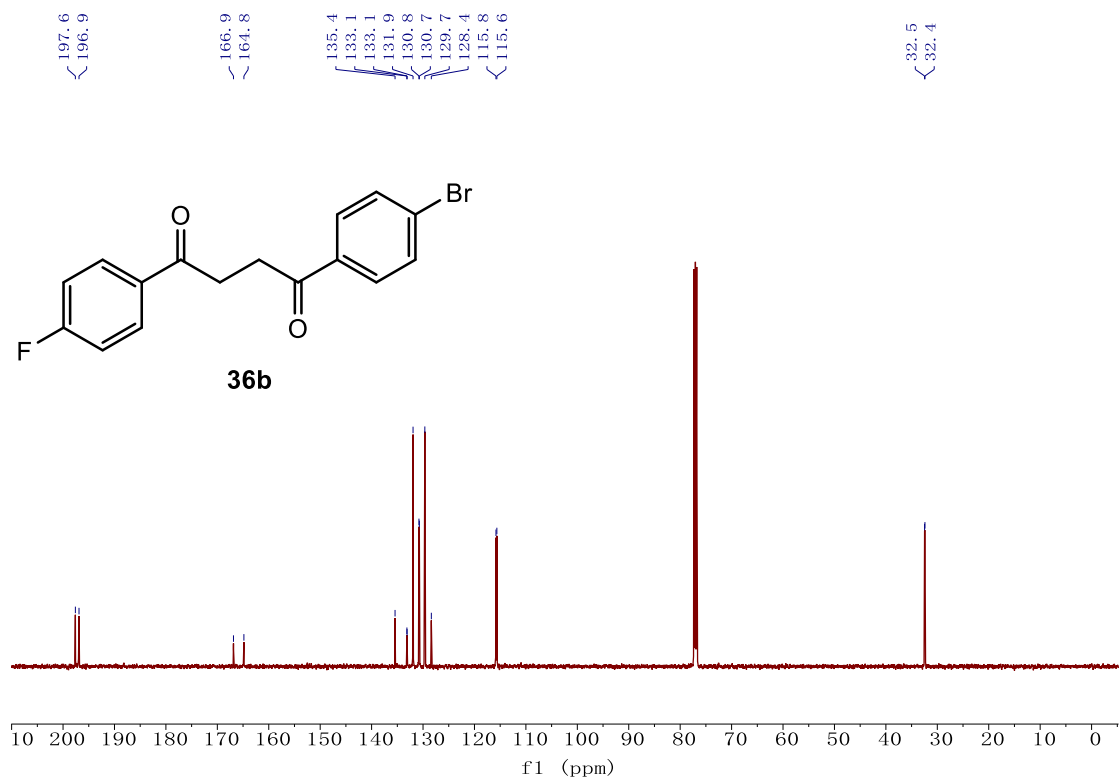
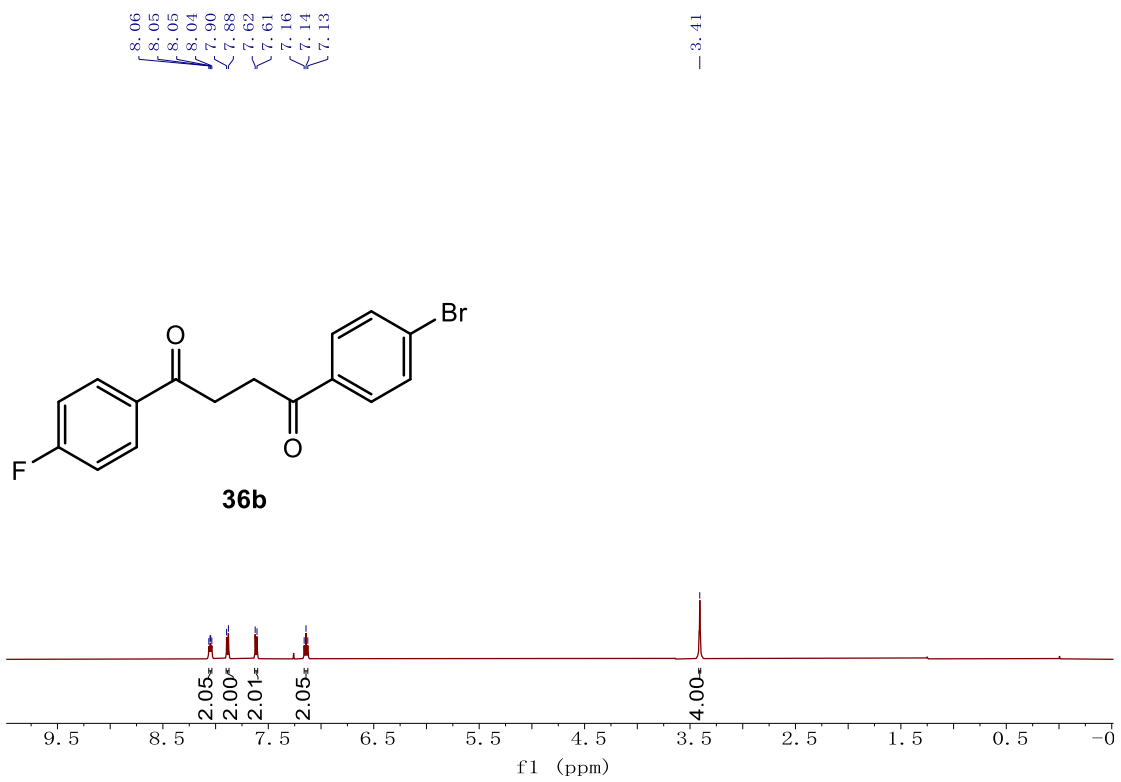
CDCl₃

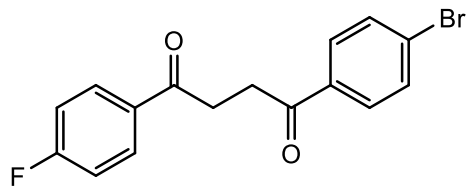


35b



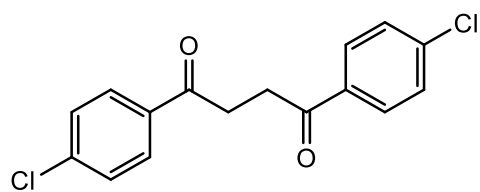
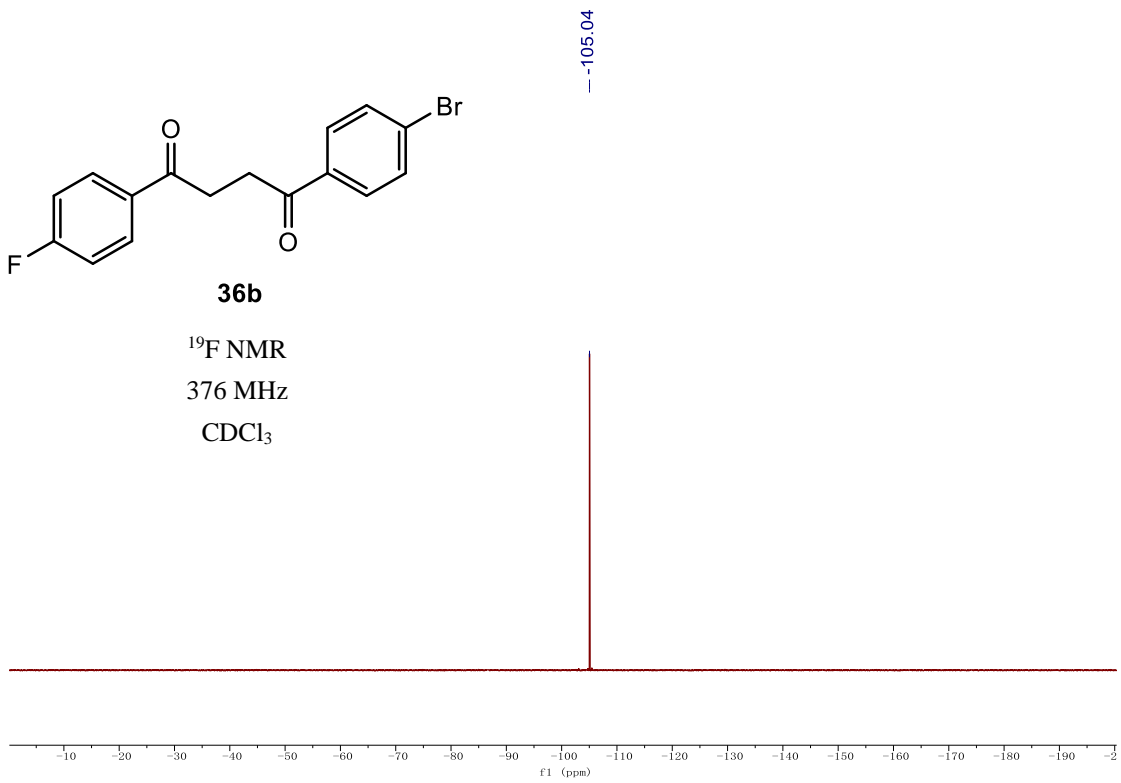




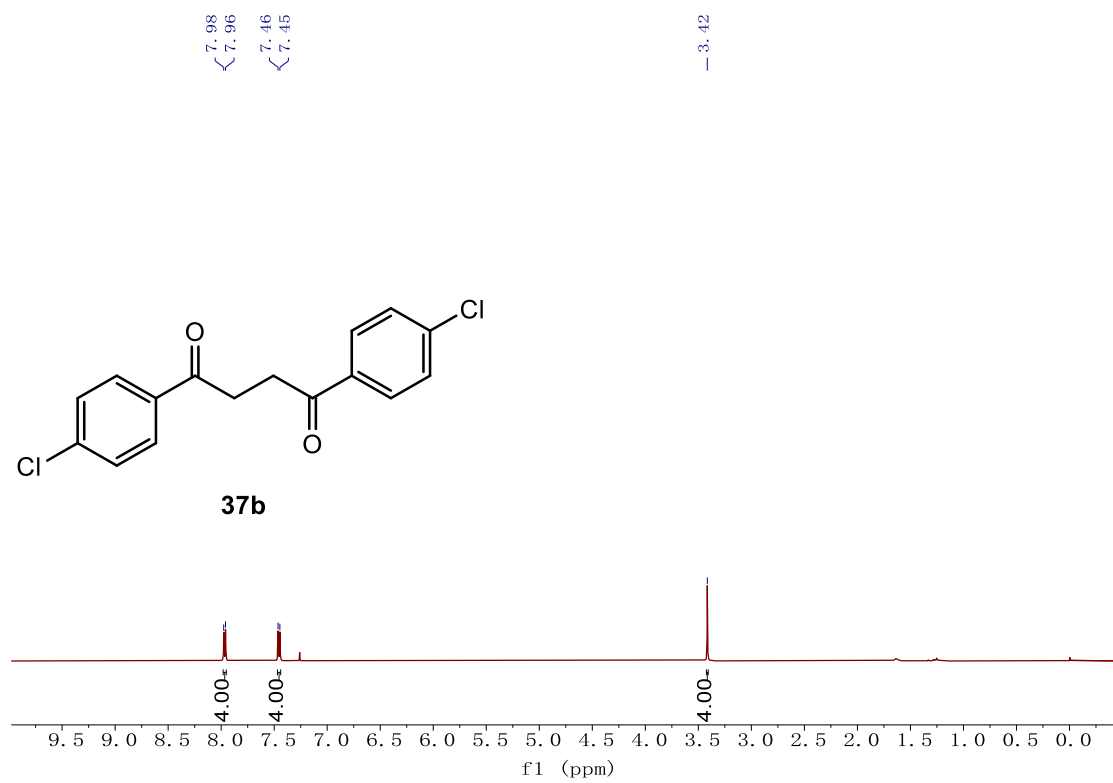


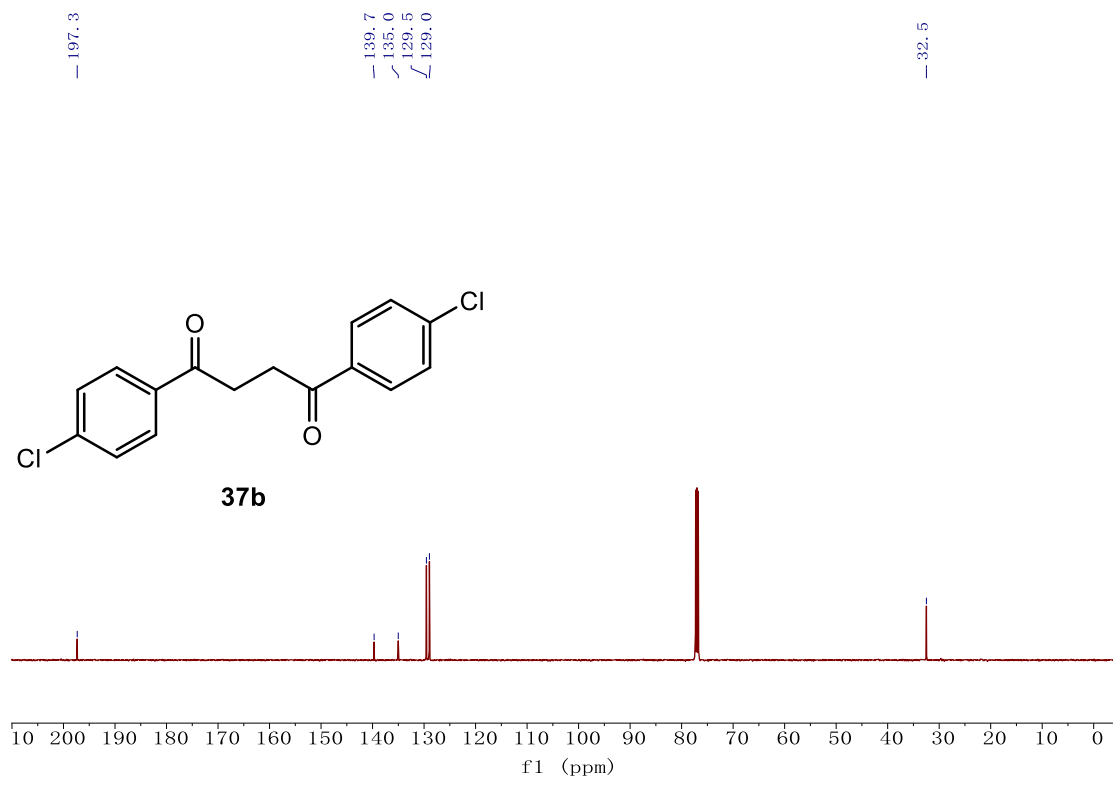
36b

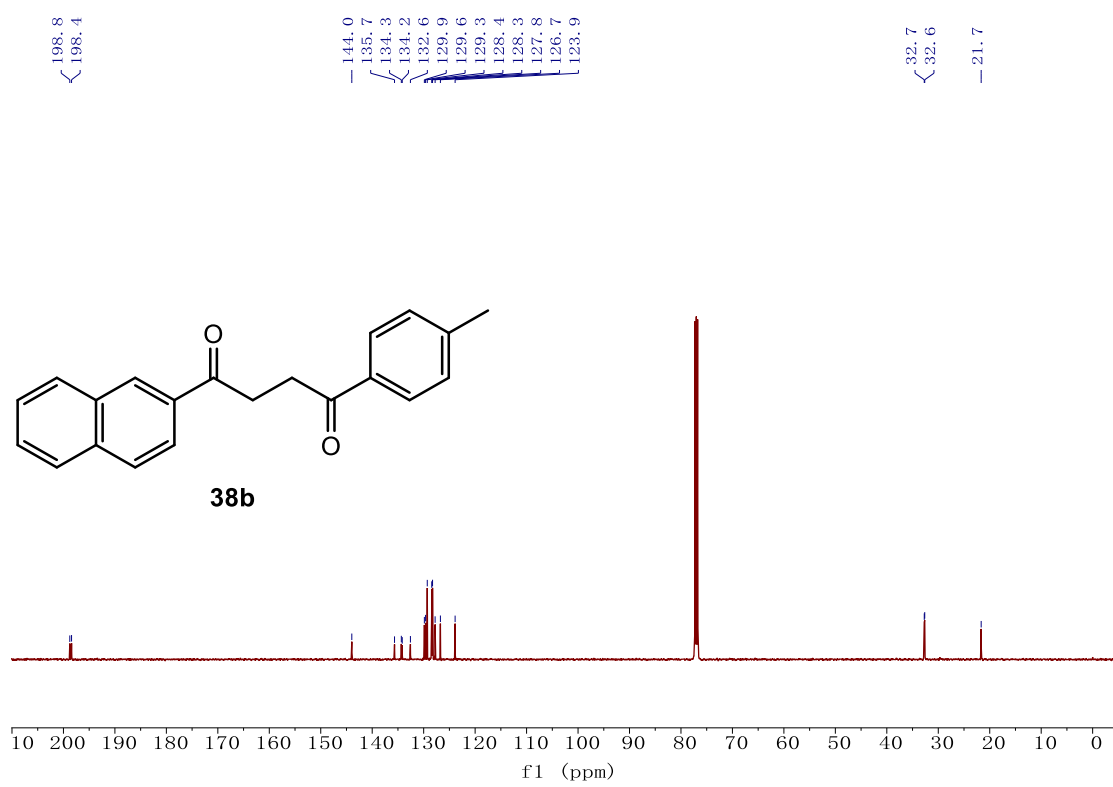
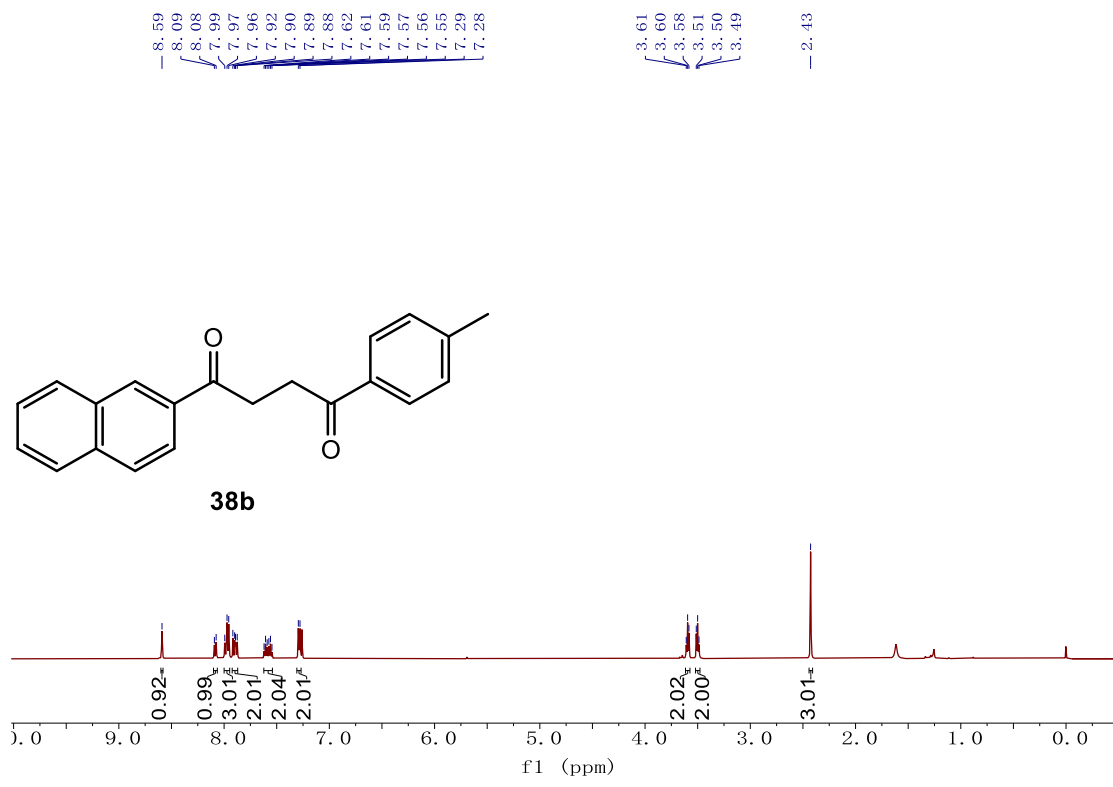
¹⁹F NMR
376 MHz
CDCl₃

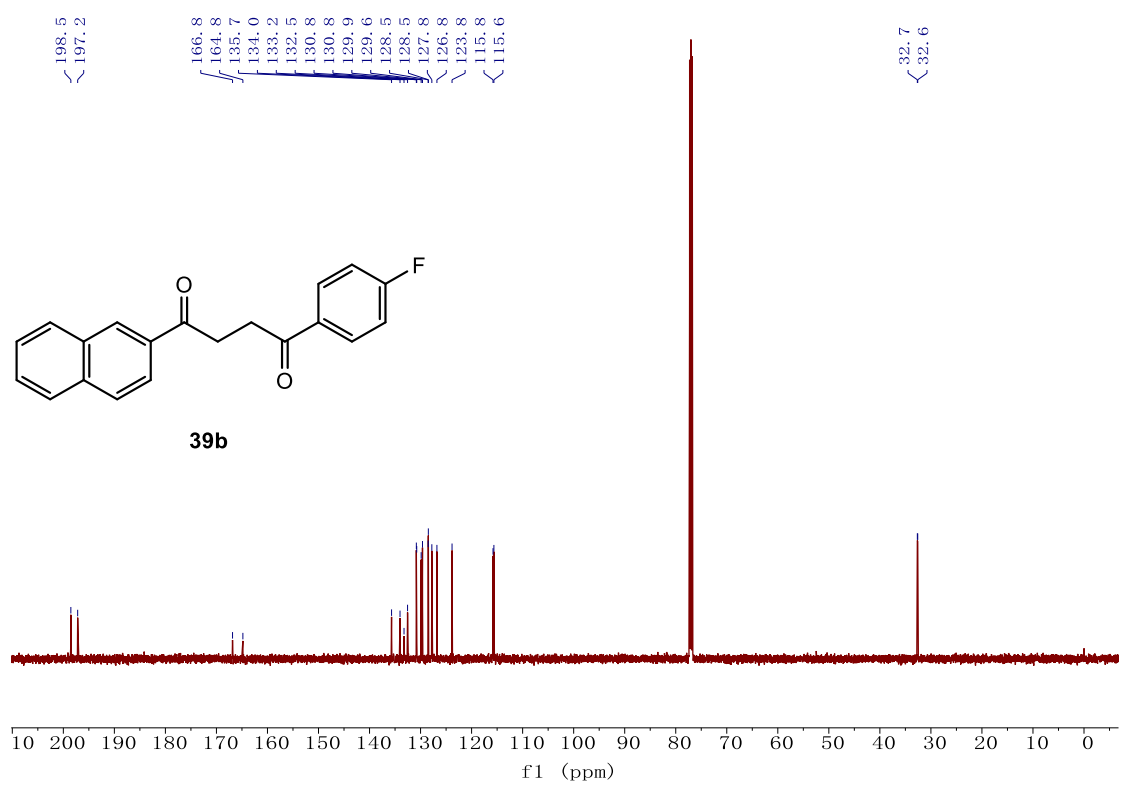
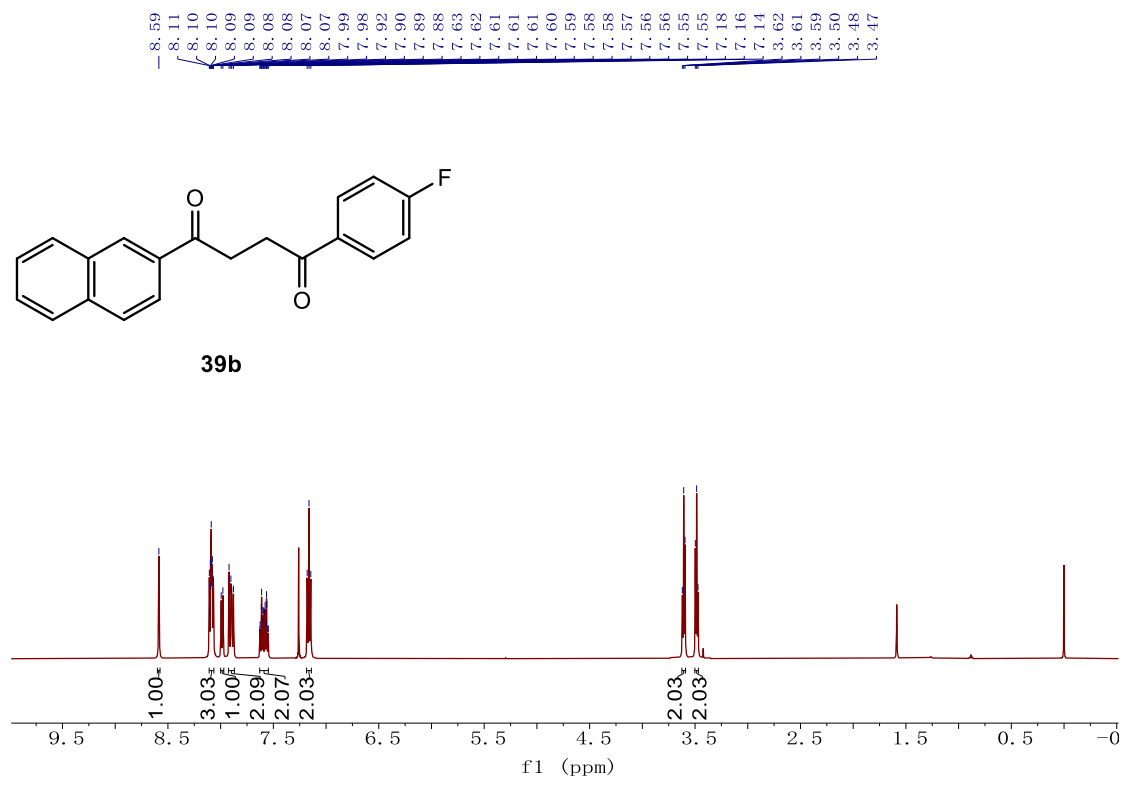


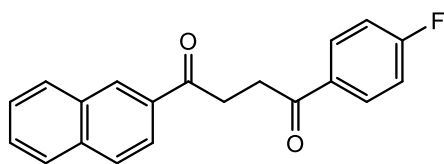
37b









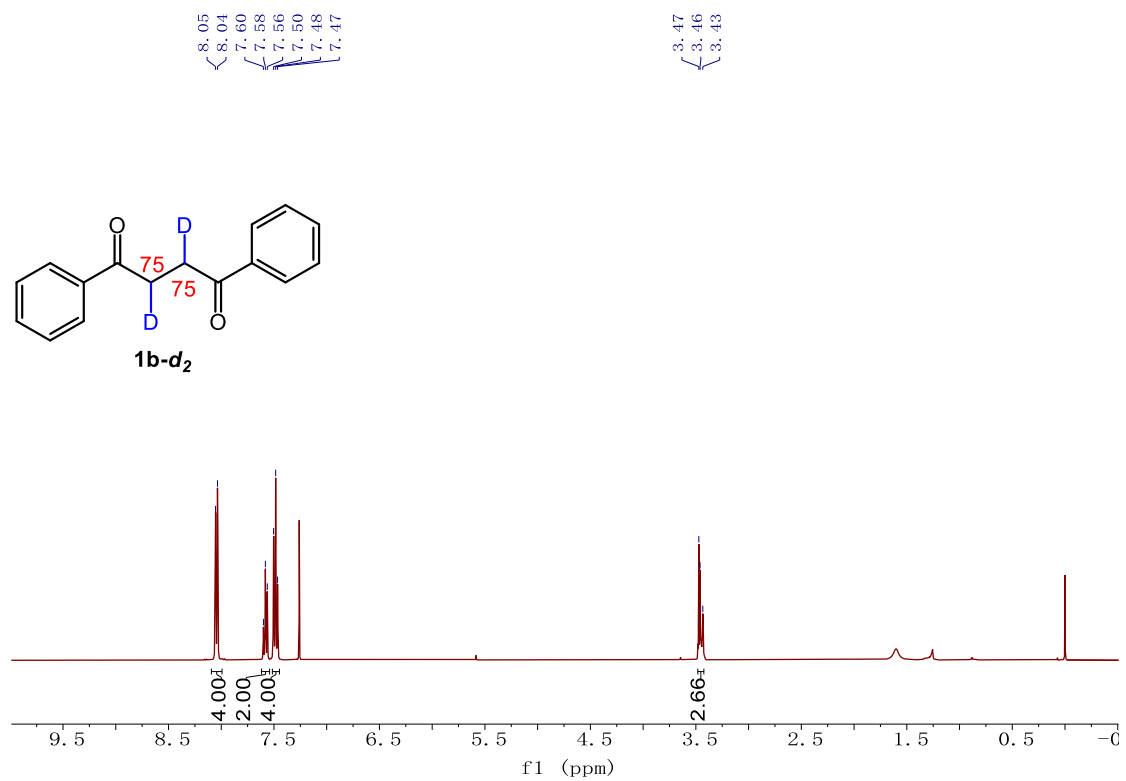
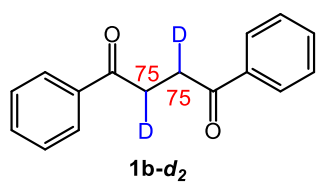
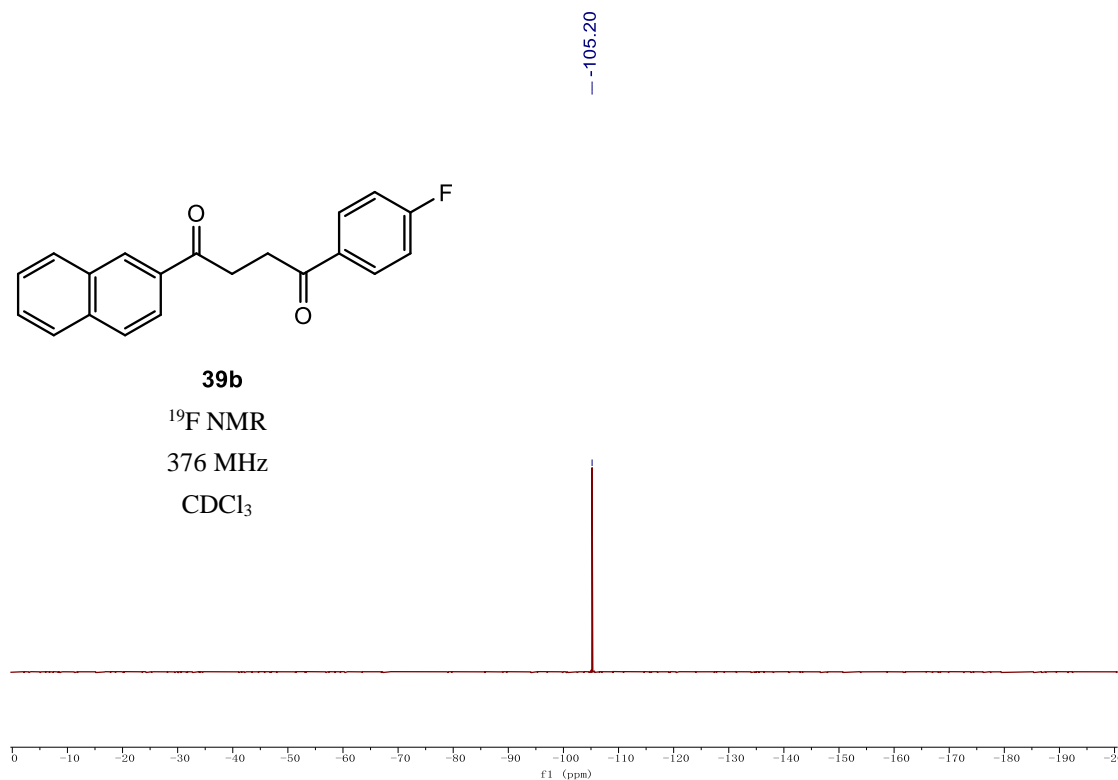


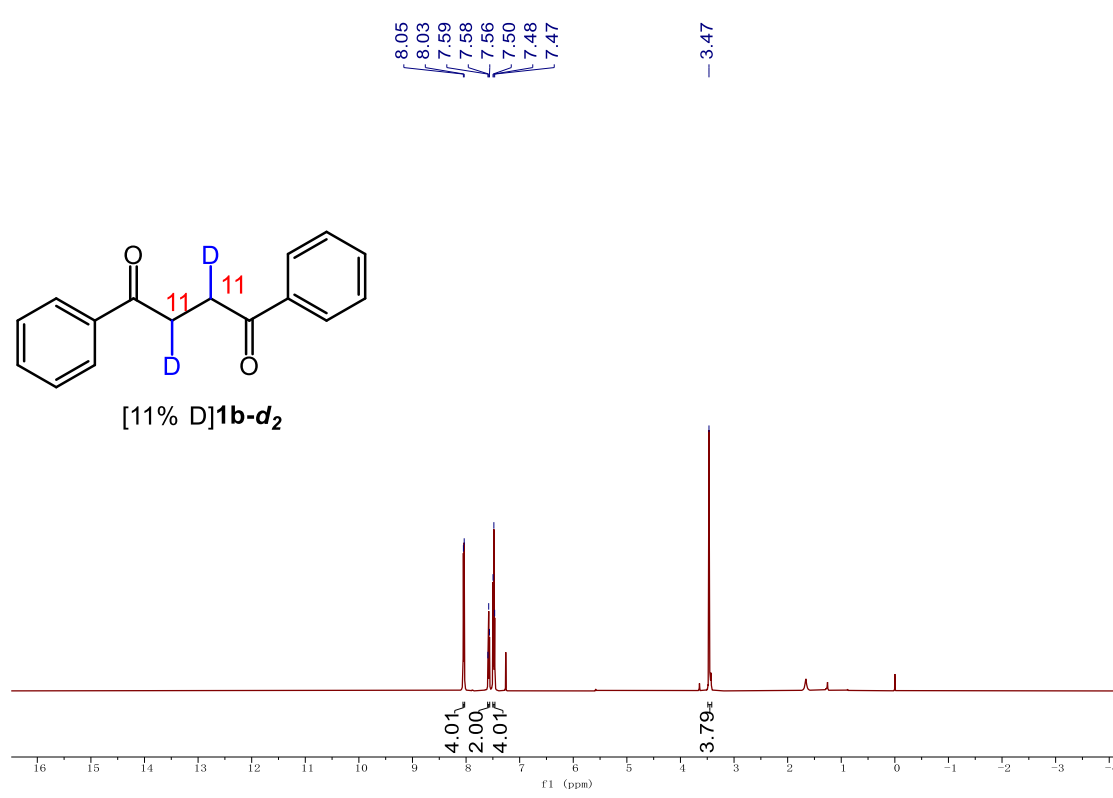
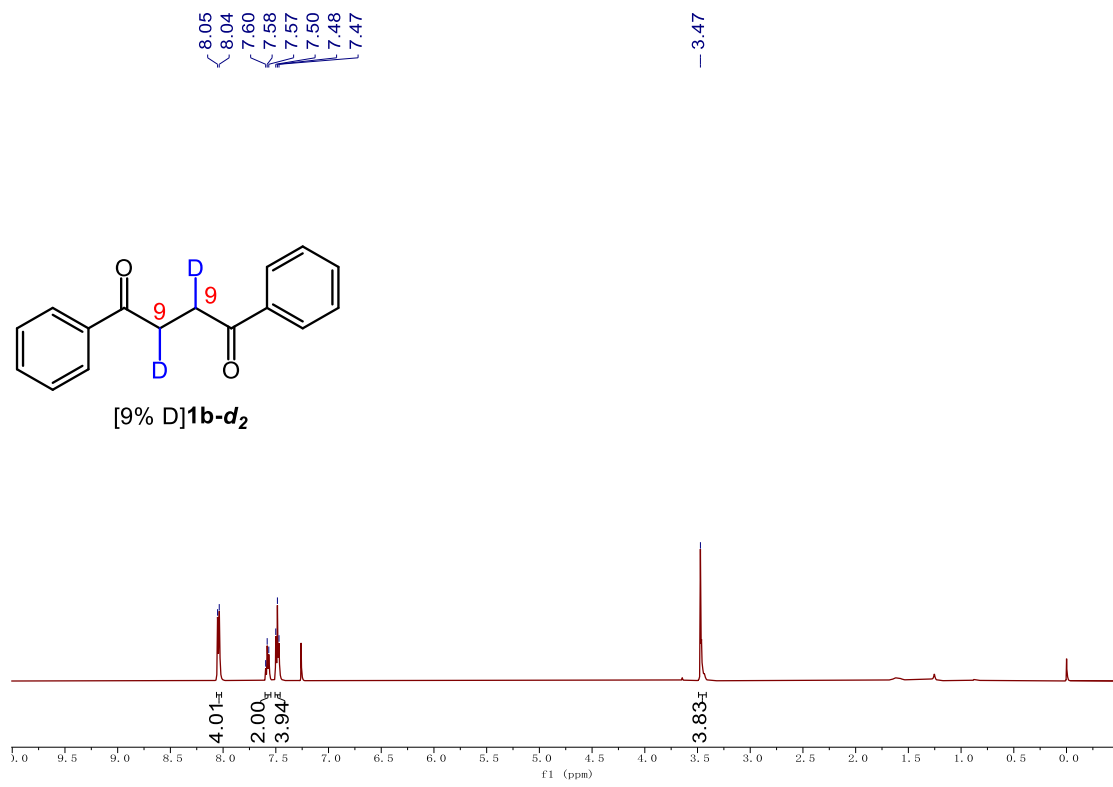
39b

^{19}F NMR

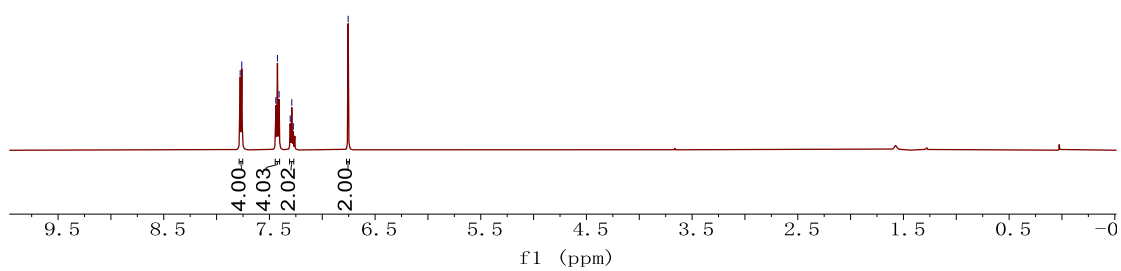
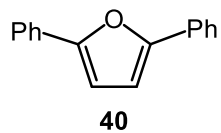
376 MHz

CDCl_3

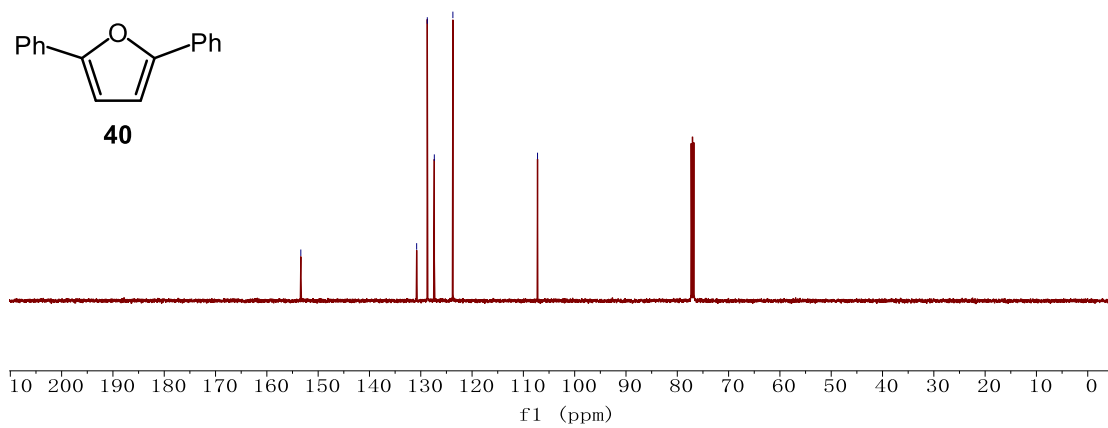
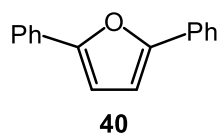


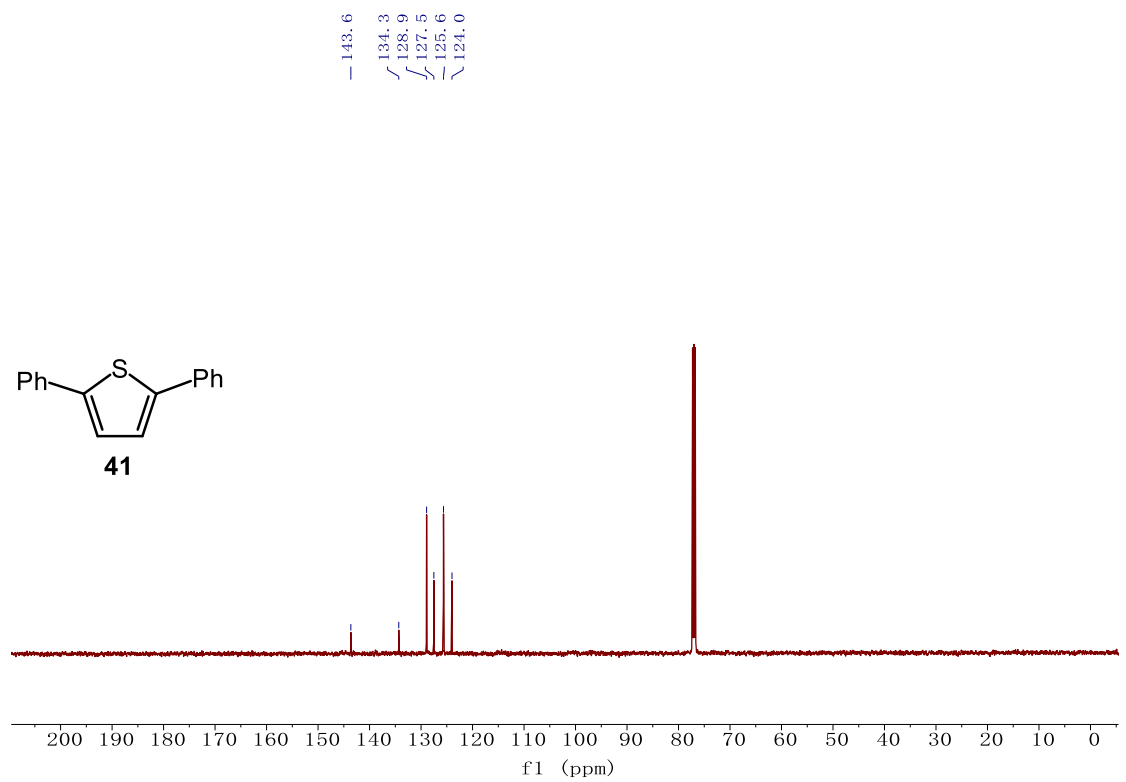
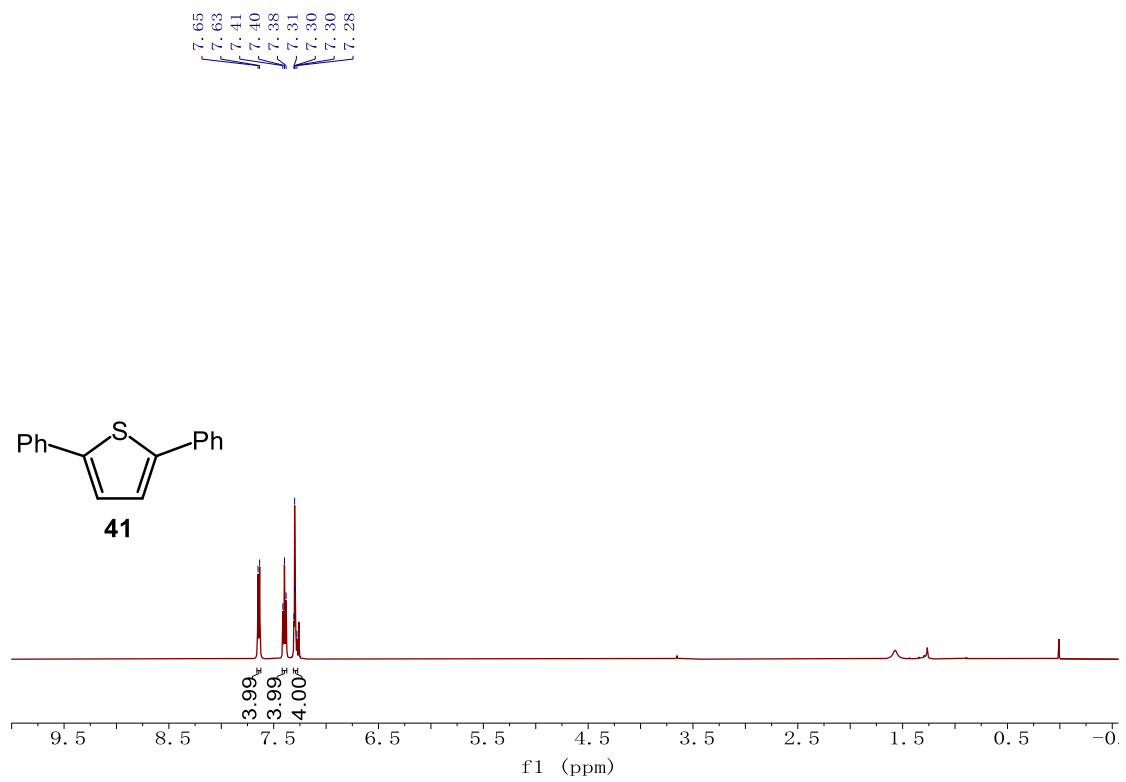


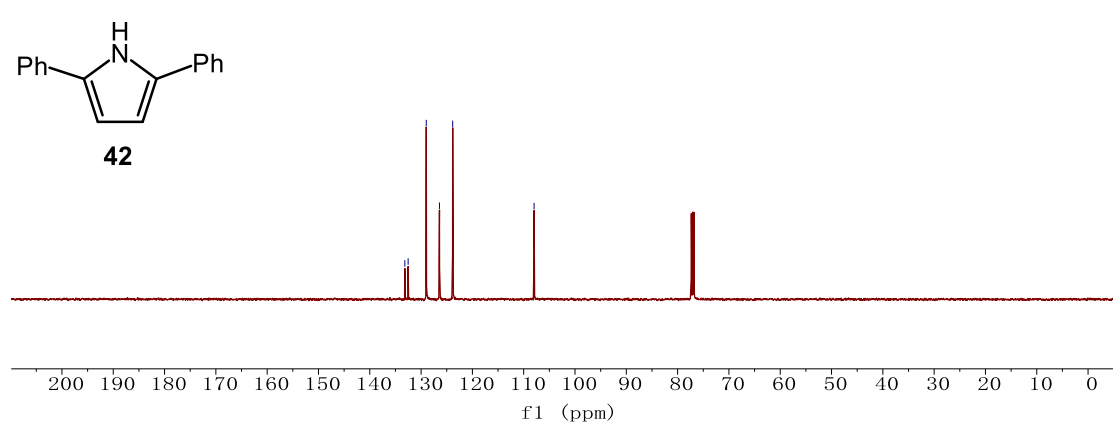
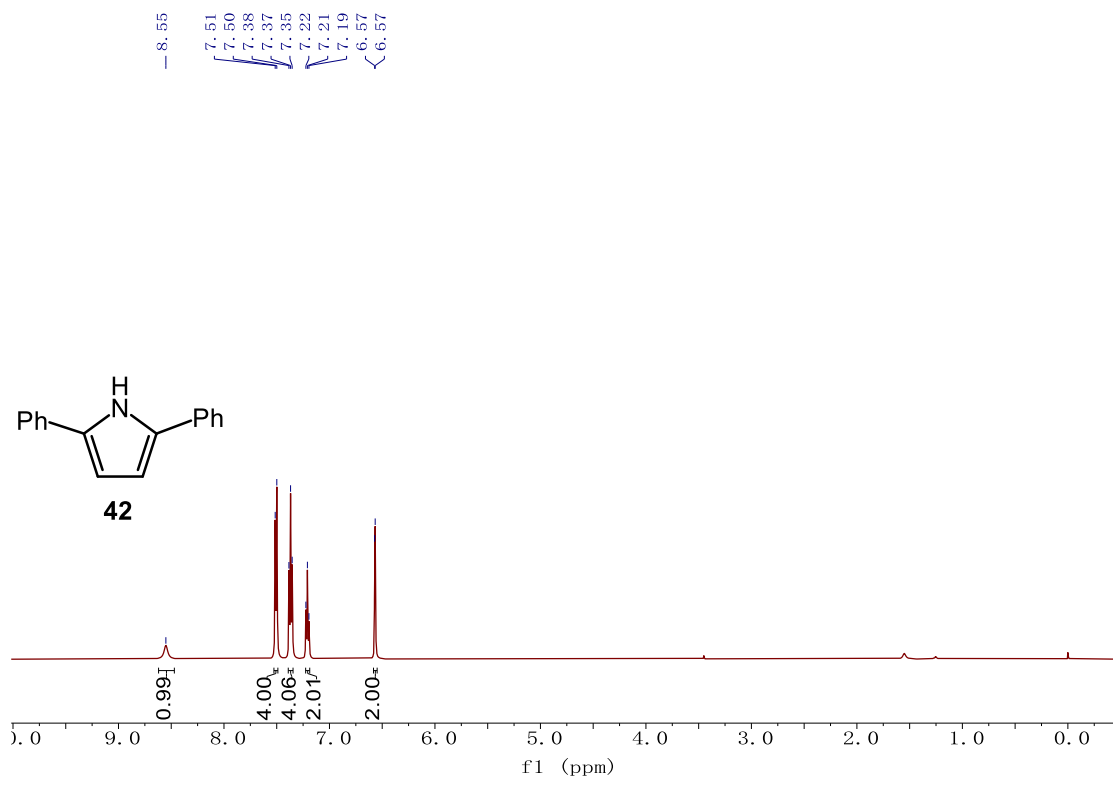
7.78
7.76
7.44
7.42
7.41
7.30
7.29
7.27
6.75

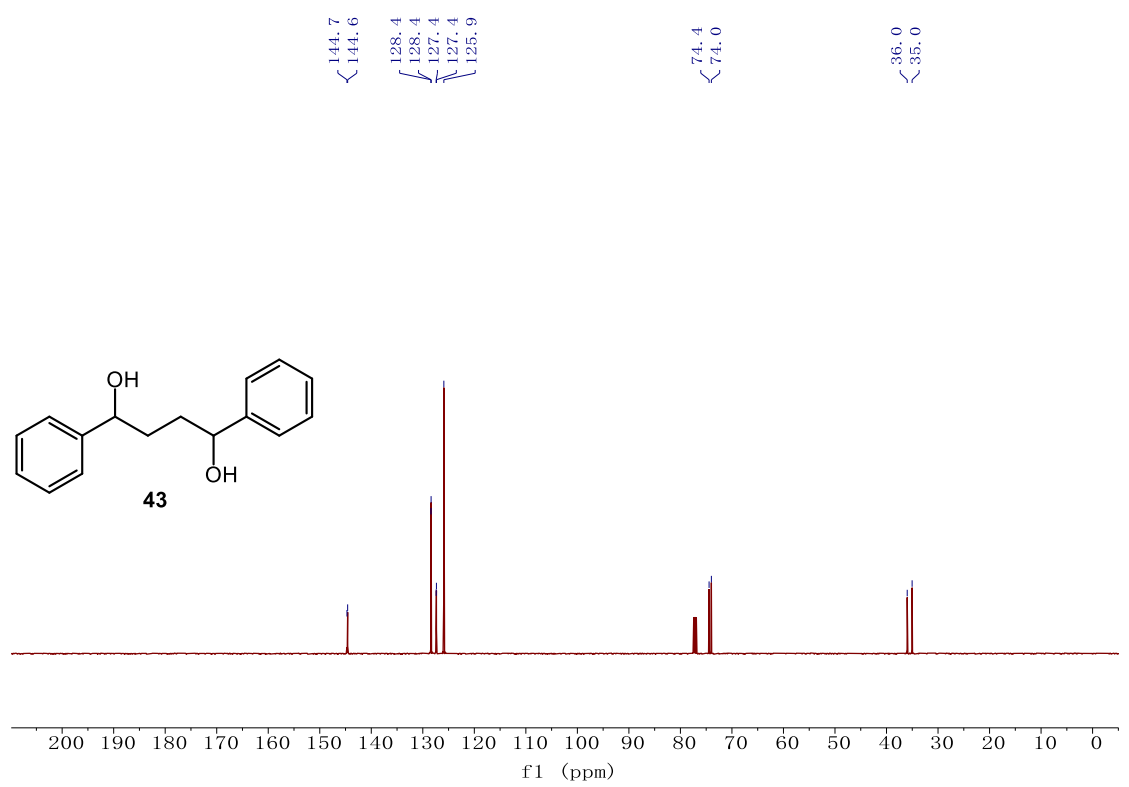
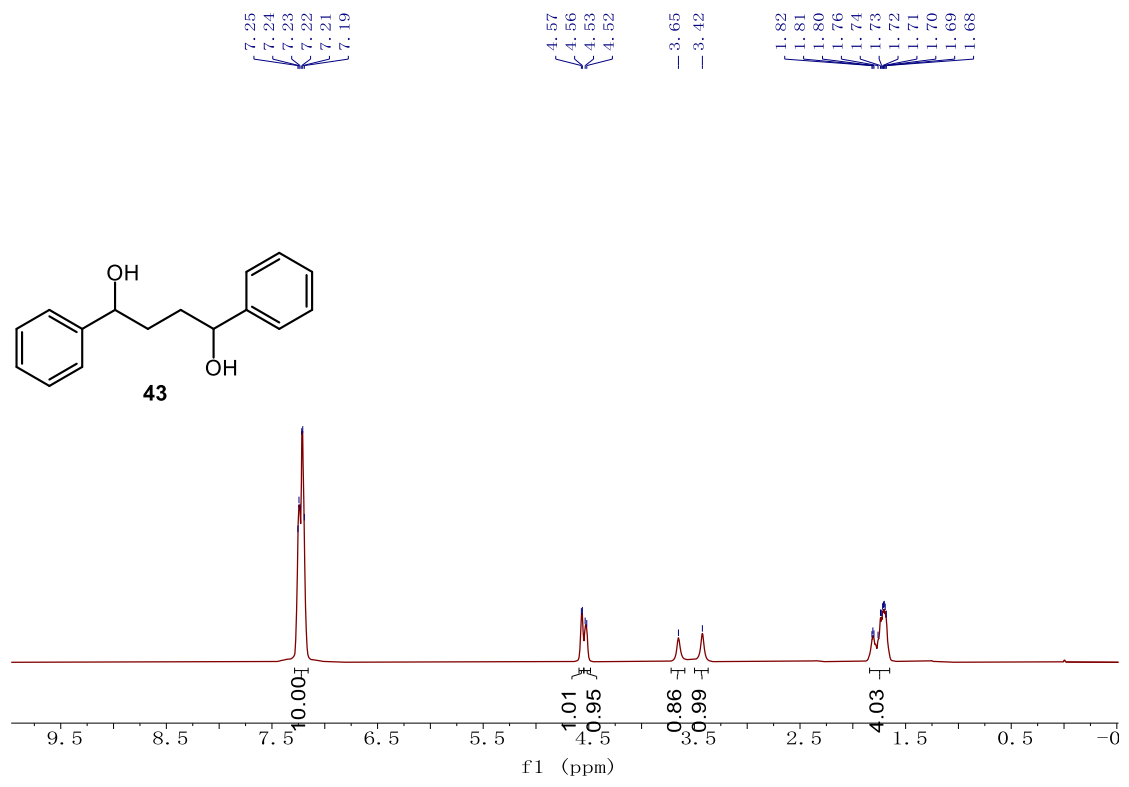


153.4
130.8
128.7
127.4
123.7
107.2

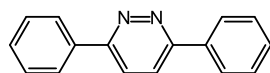




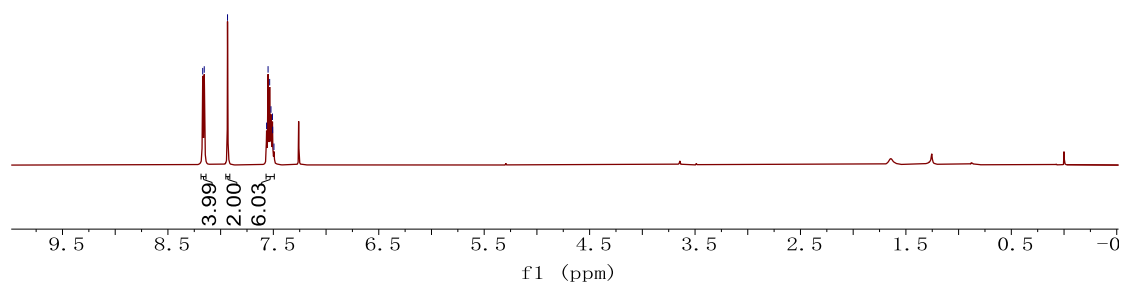




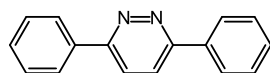
8.17
8.16
7.93
7.56
7.55
7.53
7.52
7.51
7.49



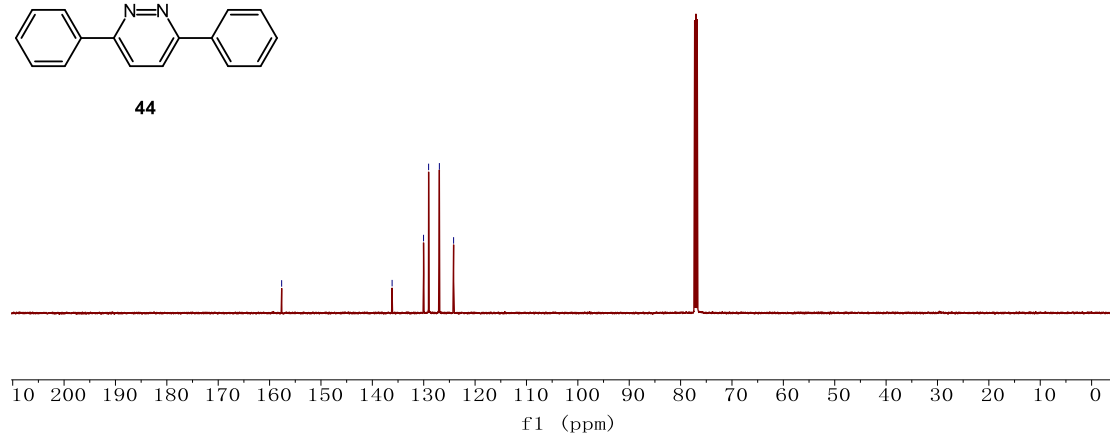
44

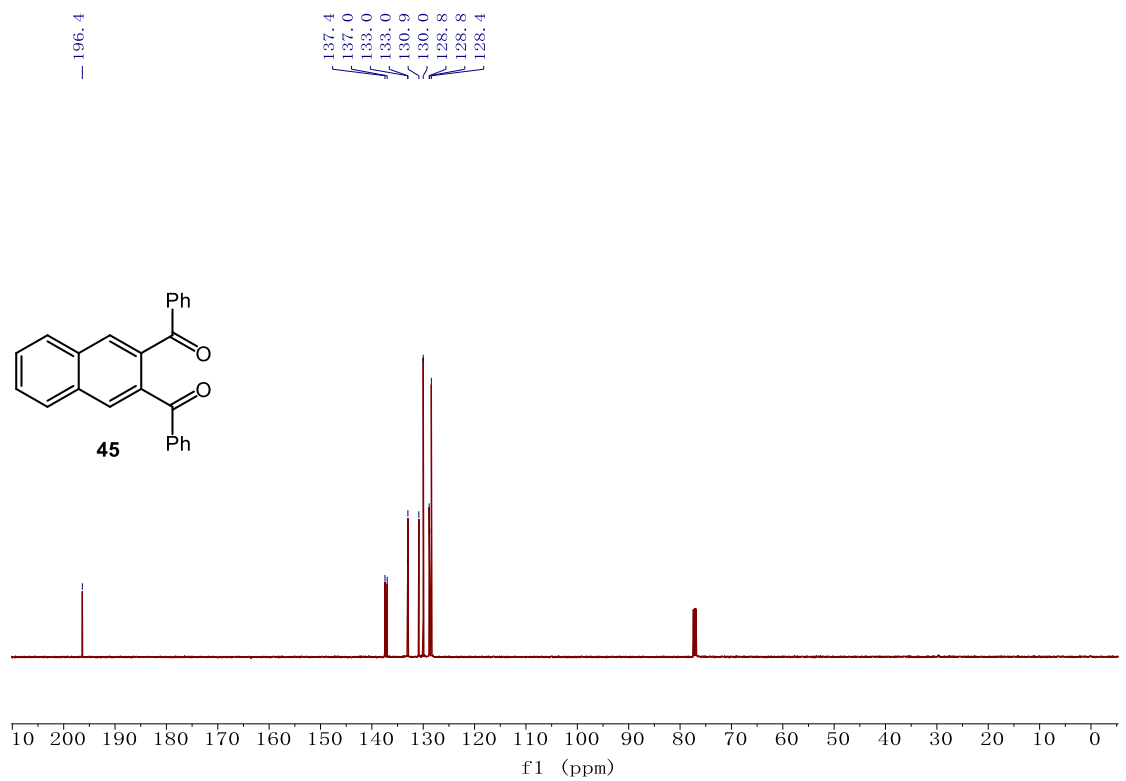
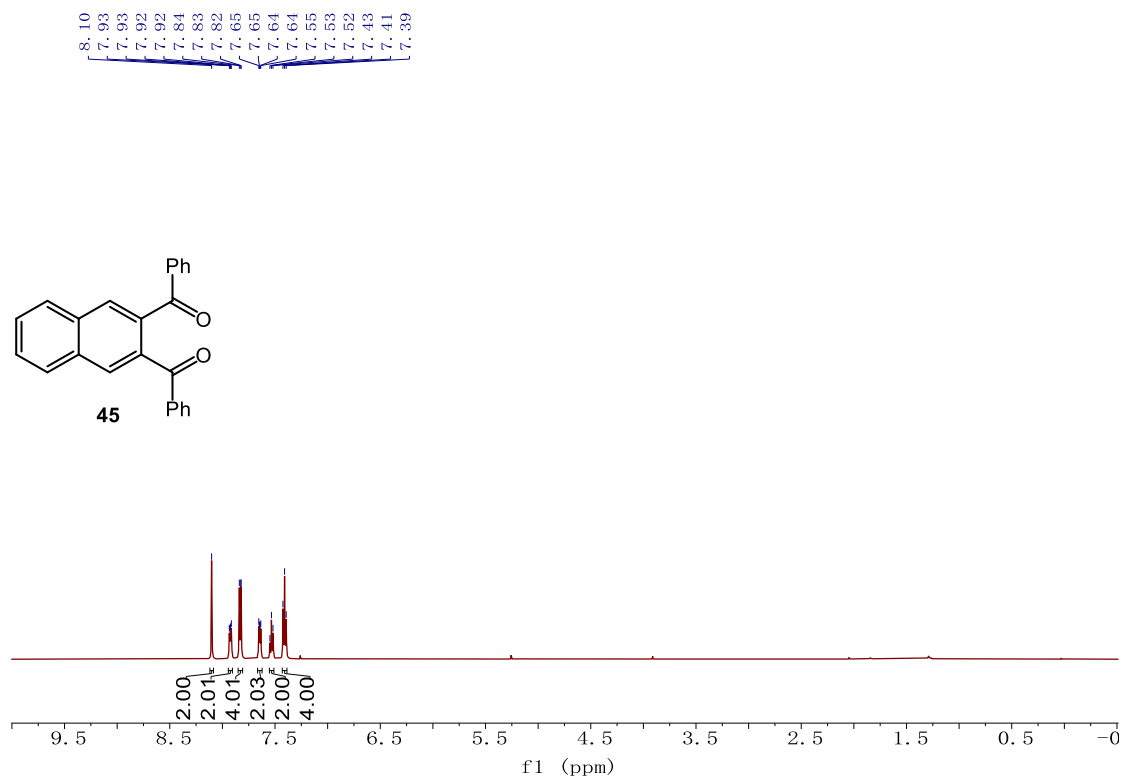


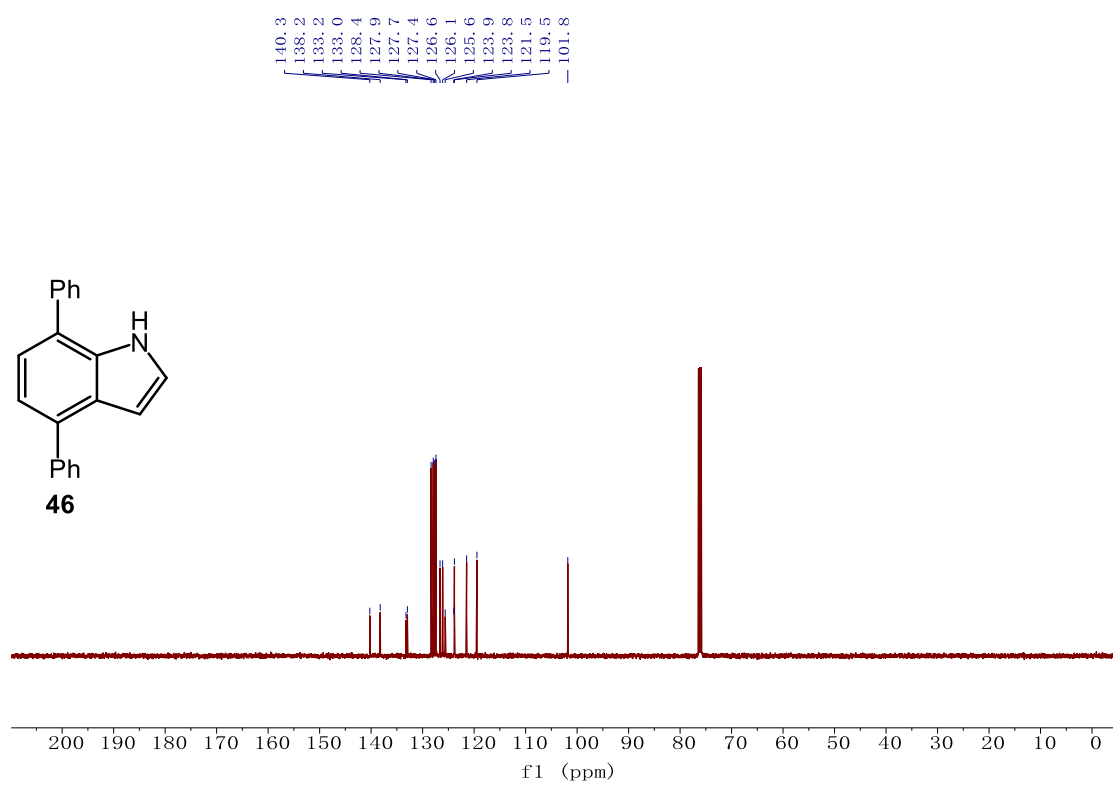
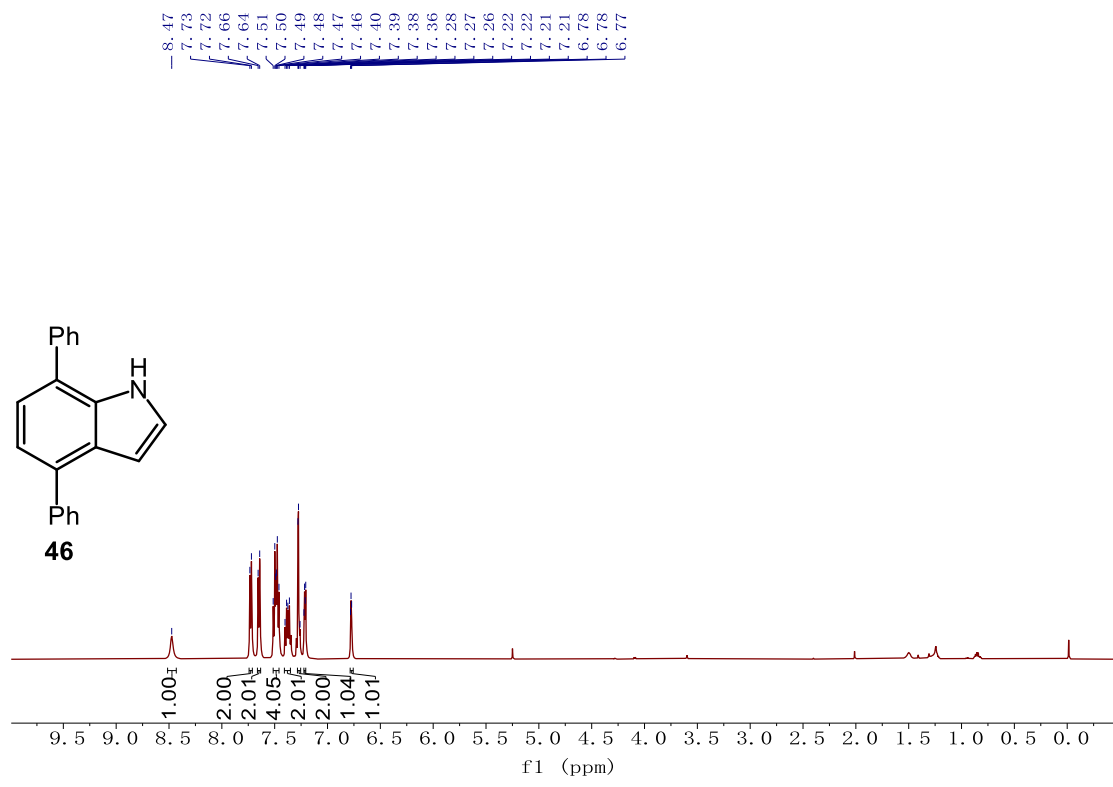
157.7
136.2
130.0
129.0
127.0
124.2



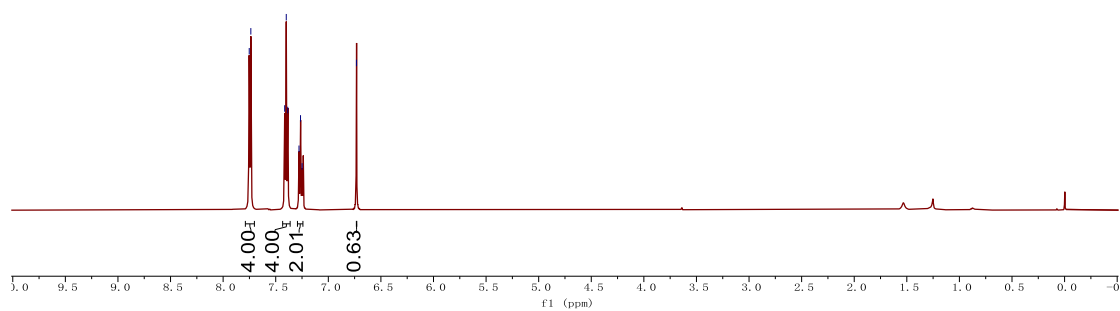
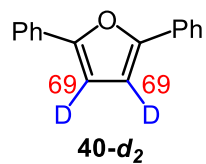
44







7.75
7.74
7.41
7.40
7.39
7.28
7.25
- 6.73



7.66
7.65
7.43
7.41
7.39
7.32
7.31
7.29

