

Supporting Information

Controllable Tertiary Amines-Promoted Photoactivation Metal-Free Carbonylation of Aryl Sulfonium Salts to Aryl Carboxylic Acid Derivatives

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1. General Information

Reagents, solvents, and analytical methods:

Unless otherwise noted, all reactions were carried out under carbon monoxide or nitrogen atmosphere. The reagents were ordered from Adamas-beta®, Energy Chemical, Sigma-Aldrich, Bidepharm and used without purification. All solvents were dried by standard techniques and distilled prior to use. Column chromatography was performed on silica gel (200-300 meshes) using petroleum ether (bp. 60~90 °C), dichloromethane and ethyl acetate as eluent. All NMR spectra were recorded at ambient temperature using Bruker Avance III 400 MHz NMR (^1H , 400 MHz; ^{13}C {1H}, 101 MHz, ^{19}F 376 MHz), Bruker AVANCE III HD 700 MHz NMR spectrometers (^1H , 700 MHz; ^{13}C {1H}, 176 MHz). ^1H NMR chemical shifts are reported relative to TMS and were referenced via residual proton resonances of the corresponding deuterated solvent (CDCl_3 : 7.26 ppm; d_6 -DMSO: 2.50 ppm) whereas ^{13}C {1H} NMR spectra are reported relative to TMS via the carbon signals of the deuterated solvent (CDCl_3 : 77.0 ppm; d_6 -DMSO: 39.5 ppm). Data for ^1H are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, br = broad), coupling constant (Hz), and integration. All ^{13}C NMR spectra were broad-band ^1H decoupled. All reactions were monitored by GC-FID or NMR analysis. HRMS data was obtained with Micromass HPLC-Q-TOF mass spectrometer (ESI-TOF) or Agilent 6540 Accurate-MS spectrometer (Q-TOF).

NOTE: Carbon monoxide should only be handled in a well-ventilated fume hood. The laboratory should be well-equipped with a CO detector and alarm system.

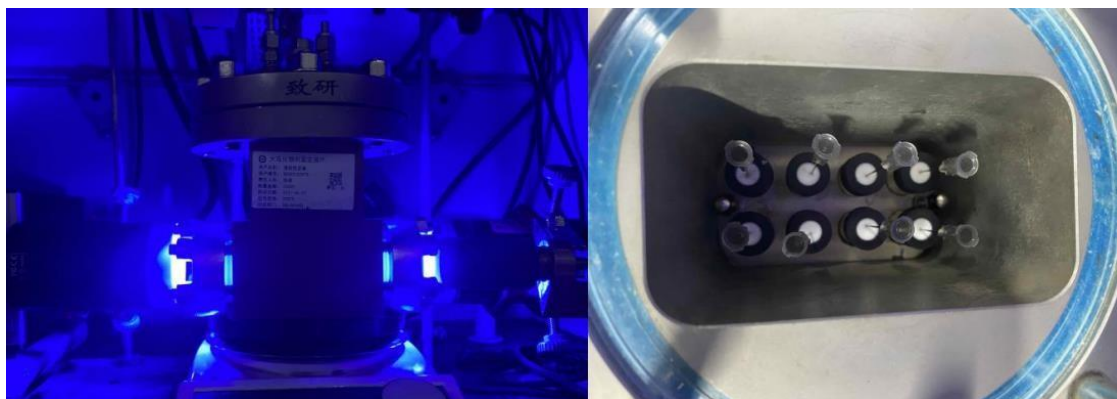
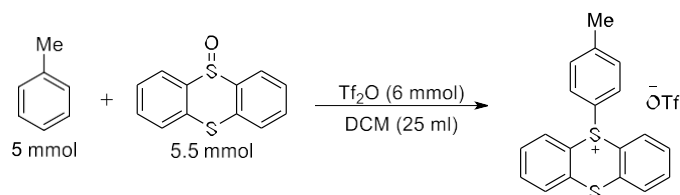


Figure S1. Photochemical Setup

2. Synthesis of the starting materials

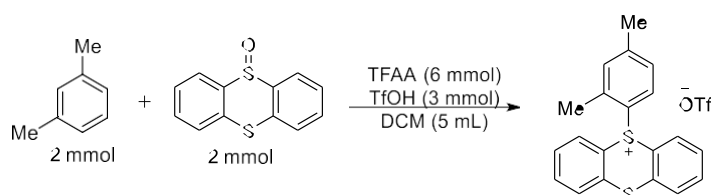
(1)



*General procedure:*¹ The known 5-(*p*-tolyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate was synthesized according to the literature. A 100 mL schlenk tube was charged with thianthrene S-oxide (1.276 g, 5.5 mmol, 1.1 equiv.), DCM (25 mL) and anisole (540 mg, 5 mmol, 1.0 equiv.) under a nitrogen atmosphere. The reaction mixture was then cooled to -40 °C and stirred at this temperature. Tf₂O (6 mmol, 1.2 equiv.) was added dropwise. The reaction mixture was stirred at -40 °C for 30 min, and then allowed to stir at room temperature for 12 h, neutralized by a saturated aqueous NaHCO₃ solution, and extracted with DCM. The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated to dryness under reduced pressure. The crude product was purified by crystallization from DCM / diethyl ether system to afford 5-(*p*-tolyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate as a white solid.

Aryl thianthrenium salts **1a**, **4a**, **5a**, **6a**, **8a**, **10a**, **11a** were synthesised by thianthrenation of the corresponding aromatics.

(2)



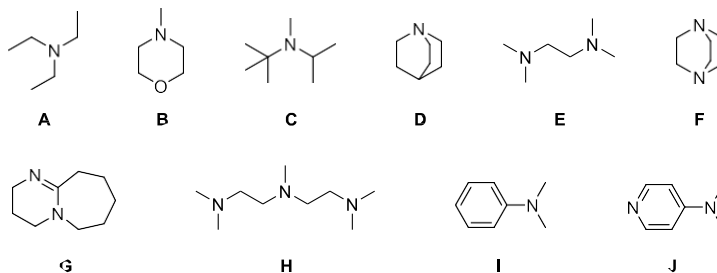
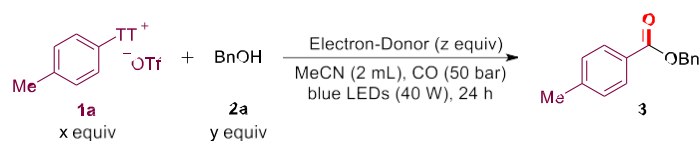
*General procedure:*¹ The known aryl sulfonium salts 5-(2,4-dimethylphenyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate was synthesized according to the literature^{1a}. A 25 mL schlenk tube was charged with thianthrene S-oxide (464 mg, 2 mmol, 1 equiv), DCM (5 mL) and *m*-xylene (212 mg, 2 mmol, 1.0 equiv) under a nitrogen atmosphere. The reaction mixture was then cooled to -40 °C and stirred at this temperature. trifluoroacetic anhydride (TFAA, 6 mmol, 3.0 equiv) and trifluoromethanesulfonic acid (TfOH, 3 mmol, 1.5 equiv) were added dropwise. The reaction mixture was stirred at -40 °C for 30 min, and then allowed to stir at room temperature for 12 h, neutralized by a saturated aqueous NaHCO₃ solution, and extracted with DCM. The combined organic layers were washed with aqueous NaOTf solution (3 × 20 mL, 5% (w/w)), dried over anhydrous Na₂SO₄, and concentrated to dryness under reduced pressure. The crude product was purified by crystallization from DCM / diethyl ether system to afford 5-(2,4-dimethylphenyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate as a white solid.

Aryl thianthrenium salts **7a**, **9a**, **12a**, **13a**, **14a**, **34a**, **35a**, **36a**, **37a**, **38a**, **39a**, **79a**, **80a** were synthesised by thianthrenation of the corresponding aromatics.

3. Optimization of the reaction conditions

3.1 Evaluation of Method A (DBU as a promoter).

Table S1. Optimization of the Reaction Conditions.

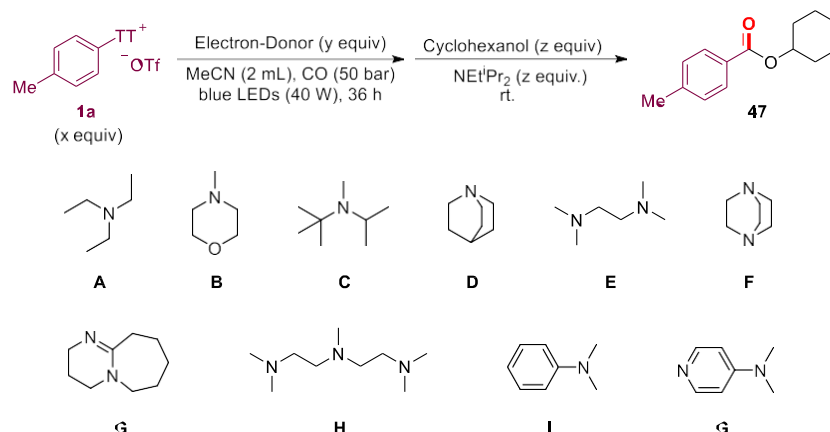


Entry	1a equiv.	2a equiv.	Electron-Donor	Solvent	Yield %
1	1	1.5	G (2.0 equiv)	Toluene	25%
2	1	1.5	G (2.0 equiv)	PhCF ₃	47%
3	1	1.5	G (2.0 equiv)	DCM	24%
4	1	1.5	G (2.0 equiv)	MeCN	48%
5	1	1.5	G (2.0 equiv)	THF	22%
6	1	1.5	G (2.0 equiv)	DMF	20%
7	1	1.5	G (2.0 equiv)	DCE	11%
8	1	2	G (2.0 equiv)	MeCN	46%
9	1	1.2	G (2.0 equiv)	MeCN	42%
10	1	1	G (2.0 equiv)	MeCN	41%
11	1.5	1	G (2.0 equiv)	MeCN	53%
12	2	1	G (2.0 equiv)	MeCN	82%
13	2	1	G (2.5 equiv)	MeCN	79%
14	2	1	G (3.0 equiv)	MeCN	93% (92%)
15	2	1	G (1.0 equiv)	MeCN	52%
14 ^b	2	1	G (3.0 equiv)	MeCN	<5%
15	2	1	A (3.0 equiv)	MeCN	trace
16	2	1	B (3.0 equiv)	MeCN	trace
17	2	1	C (3.0 equiv)	MeCN	trace
18	2	1	D (3.0 equiv)	MeCN	13%
19	2	1	E (3.0 equiv)	MeCN	trace
20	2	1	F (3.0 equiv)	MeCN	12%
21	2	1	H (3.0 equiv)	MeCN	trace
22	2	1	I (3.0 equiv)	MeCN	trace
23	2	1	J (3.0 equiv)	MeCN	28%

Reaction conditions: **1a** (x equiv), **2a** (y equiv), DBU (z equiv), CO (50 bar), MeCN (2 mL), 40W blue LEDs, 25-50 °C, 24 h. isolated yields. ^b10 bar CO. **A**, Triethylamine; **B**, 4-Methylmorpholine; **C**, *N*-Isopropyl-*N*-methyl-*tert*-butylamine; **D**, Quinuclidine; **E**, *N,N,N',N'*-Tetramethylethylenediamine; **F**, 1,4-Diazabicyclo[2.2.2]octane; **G**, 1,8-diazabicyclo[5.4.0]undec-7-ene; **H**, *N,N,N',N',N''*-Pentamethyldiethylenetriamine; **I**, *N,N*-Dimethylaniline; **J**, 4-Dimethylaminopyridine (DMAP).

3.2 Evaluation of Method B (DMAP as a promoter).

Table S2. Optimization of one-pot, two-step Reaction Conditions.

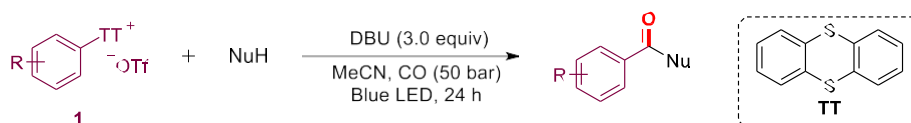


Entry	1a equiv	Cyclohexanol equiv	Electron-Donor	Solvent	Yield %
1	3	1	A (6.0 equiv)	MeCN	ND
2	3	1	B (6.0 equiv)	MeCN	ND
3	3	1	C (6.0 equiv)	MeCN	ND
4	3	1	D (6.0 equiv)	MeCN	ND
5	3	1	E (6.0 equiv)	MeCN	ND
6	3	1	F (6.0 equiv)	MeCN	ND
7	3	1	G (6.0 equiv)	MeCN	ND
8	3	1	H (6.0 equiv)	MeCN	ND
9	3	1	I (6.0 equiv)	MeCN	ND
10	3	1	G (6.0 equiv)	MeCN	94% (92%)
11	3	1	G (6.0 equiv)	PhCF ₃	84%
12	3	1	G (6.0 equiv)	DMF	trace
13	3	1	G (6.0 equiv)	Toluene	39%
14	3	1	G (6.0 equiv)	DCM	trace
15	3	1	G (6.0 equiv)	THF	22%
16	3	1	G (2.0 equiv)	1,4-dioxane	52%
18	3	1	G (3.0 equiv)	MeCN	71%
19	2.5	1	G (2.5 equiv)	MeCN	53%
20	2.5	1	G (3.0 equiv)	MeCN	63%
21	2.5	1	G (4.0 equiv)	MeCN	75%
22	2.5	1	G (5.0 equiv)	MeCN	78%
23	1	1	G (2.0 equiv)	MeCN	34%

Reaction conditions: **1a** (x mmol), Electron-Donor (y mmol), MeCN (2 mL), CO (50 bar), 40W blue LEDs, 25-50 °C, 36 h. Then Cyclohexanol (z mmol), NEt^tPr (z mmol) add to this reaction system at rt. for 16 h. isolated yields. **A**, Triethylamine; **B**, 4-Methylmorpholine; **C**, *N*-Isopropyl-*N*-methyl-tert-butylamine; **D**, Quinuclidine; **E**, *N,N,N',N'*-Tetramethylethylenediamine; **F**, 1,4-Diazabicyclo[2.2.2]octane; **G**, 1,8-diazabicyclo[5.4.0]undec-7-ene; **H**, *N,N,N',N'',N''*-Pentamethyldiethylenetriamine; **I**, *N,N*-Dimethylaniline; **J**, 4-Dimethylaminopyridine (DMAP).

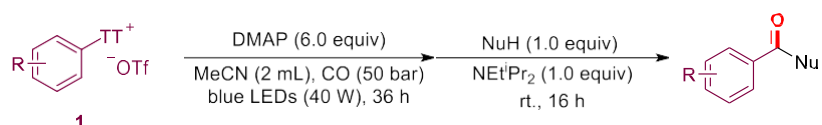
4. General Procedure

4.1 General procedure I (DBU as a promoter).



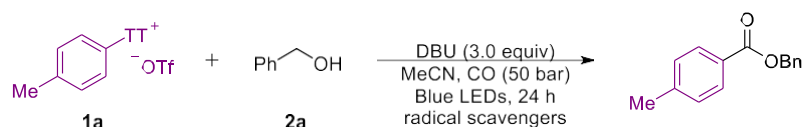
A 4 mL screw-cap vial was charged with aryl sulfonium salts **1** (0.4 mmol) and a stirring bar. The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. After MeCN (2 mL), DBU (0.6 mmol), and nucleophiles (0.2 mmol) were added with a syringe under a nitrogen atmosphere. The vials (usually 8) were placed on an alloy plate, which was transferred into an autoclave with two inserted quartz-glass windows. After the autoclave was flushed three times, it was pressurized with 50 bar of CO and irradiated with 40 W blue LEDs at 25-50 °C for 24 h. After completion, the reaction mixture was directly purified by column chromatography on silica gel using petroleum ether and ethyl acetate to afford the corresponding product.

4.2 General procedure II (DMAP as a promoter, one-pot, two-step Reaction).



A 4 mL screw-cap vial was charged with aryl sulfonium salts **1** (0.6 mmol), DMAP (1.2 mmol) and a stirring bar. The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. After MeCN (2 mL) was added with a syringe under a nitrogen atmosphere. The vials (usually 8) were placed on an alloy plate, which was transferred into an autoclave with two inserted quartz-glass windows. After the autoclave was flushed three times, it was pressurized with 50 bar of CO and irradiated with 40 W blue LEDs at 25-50 °C for 36 h. After the reaction is complete, CO is released from the alloy plate and backfilled with nitrogen three times. Then remove the screw cap vial from the alloy plate and add nucleophiles (0.2 mmol) and NEt^iPr_2 (0.2 mmol) to the reaction mixture. The reaction was stirred at room temperature for 16 h. After completion, the reaction mixture was directly purified by column chromatography on silica gel using petroleum ether and ethyl acetate to afford the corresponding product.

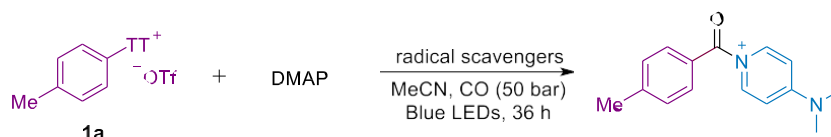
4.3 Typical Procedures for the Radical Trapping Experiments I.



A 4 mL screw-cap vial was charged with aryl sulfonium salts **1a** (0.4 mmol), radical scavengers (0.8 mmol) and a stirring bar. The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. After MeCN (2 mL), DBU (0.6 mmol), and nucleophiles (0.2 mmol) were added with a syringe under a nitrogen atmosphere. The vials were placed on an alloy plate, which was transferred into an autoclave with two inserted quartz-glass windows. After the autoclave was flushed three times, it was pressurized with 50 bar of CO and irradiated with 40 W blue LEDs at 25-50 °C for 24 h. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. After cooling to room temperature, the reaction mixture was

directly purified by column chromatography on silica gel using petroleum ether and ethyl acetate methanol to afford the corresponding product.

4.4 Typical Procedures for the Radical Trapping Experiments II.



A 4 mL screw-cap vial was charged with aryl sulfonium salts **1a** (0.2 mmol), DMAP (0.4 mmol), radical scavengers (0.4 mmol) and a stirring bar. The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. After MeCN (2 mL) was added with a syringe under a nitrogen atmosphere. The vials were placed on an alloy plate, which was transferred into an autoclave with two inserted quartz-glass windows. After the autoclave was flushed three times, it was pressurized with 50 bar of CO and irradiated with 40 W blue LEDs at 25-50 °C for 36 h. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. After cooling to room temperature, the reaction mixture was directly purified by column chromatography on silica gel using petroleum ether and ethyl acetate to afford the corresponding product.

4.5 UV-vis absorbance experiment I.

A UV-vis absorbance experiment has been carried out for confirming the formation of EDA complex as illustrated below in Figure S2. In Figure S2, the black UV absorbance line came from **1a** (0.4 mmol in 4 mL MeCN) solution, the red one came from DBU (0.6 mmol in 4 mL MeCN) solution, the blue one came from the mixed solution of **1a** (0.4 mmol in 4 mL MeCN) and DBU (0.6 mmol in 4 mL MeCN), the green line came from a mixed solution of **1a** (0.4 mmol in 4 mL MeCN) and DBU (0.6 mmol in 4 mL MeCN) exposed to a 456nm blue light for 20 minutes. A bathochromic shift was observed for a mixture of aryl thianthrenium salt **1a** (0.4 mmol), DBU (0.6 mmol), in MeCN (4 mL) after 20 min of irradiation at 456 nm, which was a visibly intense yellow in color (Figure S1). This indicates the formation of an electron donor-acceptor (EDA) complex.

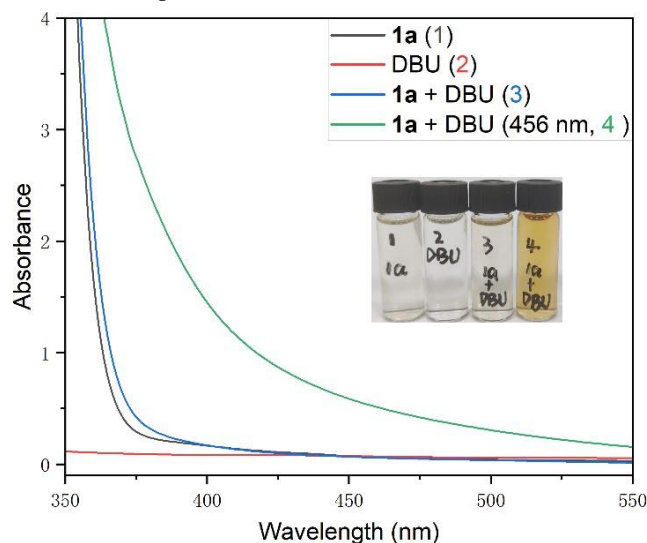


Figure S2. UV/vis absorption spectra of individual reaction components and a combination thereof. All spectra were measured in MeCN and with a concentration of 0.1 M thianthrenium salt **1a**, 0.15 M DBU.

4.6 UV-vis absorbance experiment II.

A UV-vis absorbance experiment has been carried out for confirming the formation of EDA complex as illustrated below in Figure S3. In Figure S3, the black UV absorbance line came from **1a** (0.6 mmol in 4 mL MeCN) solution, the red one came from DMAP (1.2 mmol in 4 mL MeCN) solution, the blue one came from the mixed solution of **1a** (0.6 mmol in 4 mL MeCN) and DMAP (1.2 mmol in 4 mL MeCN), the green line came from a mixed solution of **1a** (0.6 mmol in 4 mL MeCN) and DMAP (1.2 mmol in 4 mL MeCN) exposed to a 456nm blue light for 20 minutes, the purple line came from a mixed solution of **1a** (0.6 mmol in 4 mL MeCN) and DMAP (1.2 mmol in 4 mL MeCN) exposed to a 400nm blue light for 20 minutes. A bathochromic shift was observed for a mixture of aryl thianthrenium salt **1a** (0.6 mmol), DMAP (1.2 mmol), in MeCN (4 mL) after 20 min of irradiation at 400 nm, which was a visibly intense yellow in color (Figure S3). This indicates the formation of an electron donor-acceptor (EDA) complex.

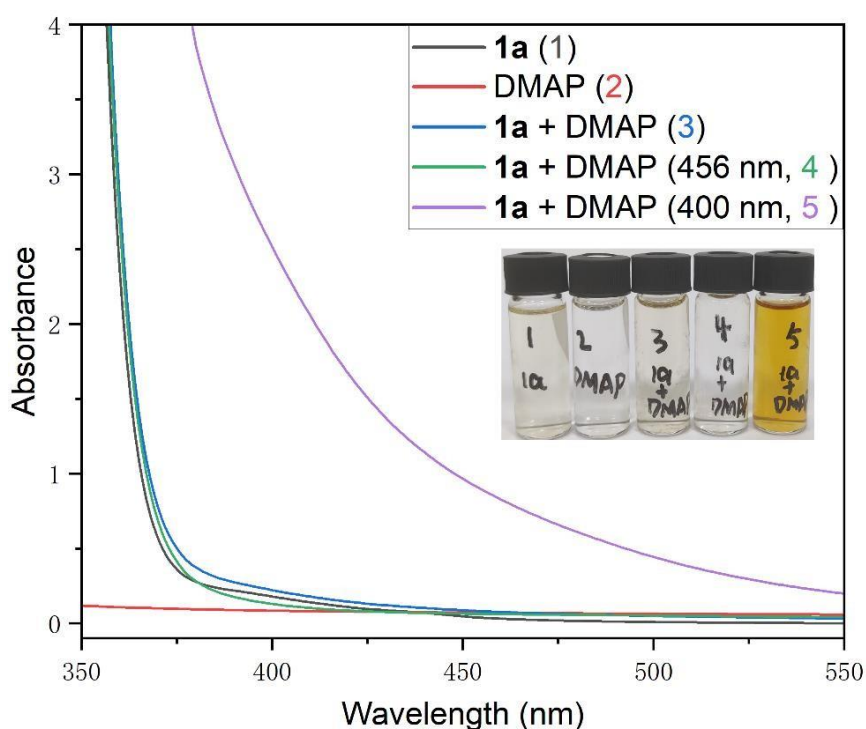
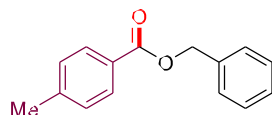


Figure S3. UV/vis absorption spectra of individual reaction components and a combination thereof. All spectra were measured in MeCN and with a concentration of 0.15 M thianthrenium salt **1a**, 0.3 M DMAP.

5. Characterization of Products

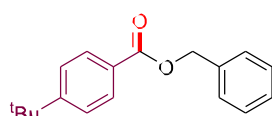


benzyl 4-methylbenzoate (**3**)²

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (41.6 mg, 92% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.46 – 7.42 (m, 2H), 7.41 – 7.36 (m, 2H), 7.35 – 7.31 (m, 1H), 7.22 (d, *J* = 8.0 Hz, 2H), 5.35 (s, 2H), 2.39 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.5, 143.8, 136.2, 129.8, 129.1, 128.6, 128.2, 128.2, 127.4, 66.5, 21.7.

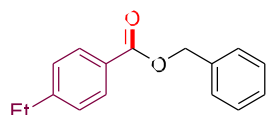


benzyl 4-(*tert*-butyl)benzoate (**4**)²

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (40.2 mg, 75% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 8.03 (d, *J* = 8.5 Hz, 2H), 7.49 – 7.43 (m, 4H), 7.42 – 7.30 (m, 3H), 5.37 (s, 2H), 1.35 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 166.5, 156.7, 136.3, 129.6, 128.6, 128.2, 128.1, 127.4, 125.4, 66.5, 35.1, 31.1.

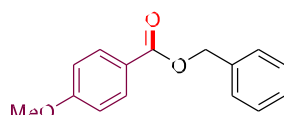


benzyl 4-ethylbenzoate (**5**)³

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (37.9 mg, 79% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.44 (d, *J* = 6.6 Hz, 2H), 7.41 – 7.30 (m, 3H), 7.25 (d, *J* = 8.2 Hz, 2H), 5.35 (s, 2H), 2.69 (q, *J* = 7.6 Hz, 2H), 1.24 (t, *J* = 7.6 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.5, 149.9, 136.3, 129.9, 128.6, 128.2, 128.1, 127.9, 127.6, 66.5, 29.0, 15.3.

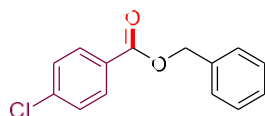


benzyl 4-methoxybenzoate (**6**)²

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (32.4 mg, 67% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 8.9 Hz, 2H), 7.48 – 7.42 (m, 2H), 7.42 – 7.36 (m, 2H), 7.36 – 7.30 (m, 1H), 6.92 (d, *J* = 8.9 Hz, 2H), 5.34 (s, 2H), 3.86 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.2, 163.5, 136.3, 131.8, 128.6, 128.2, 128.1, 122.6, 113.6, 66.4, 55.4.

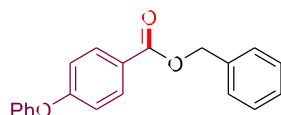


benzyl 4-chlorobenzoate (**7**)²

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (38.9 mg, 79% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 8.01 (d, *J* = 8.6 Hz, 2H), 7.50 – 7.32 (m, 7H), 5.36 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 165.6, 139.5, 135.8, 131.1, 128.8, 128.7, 128.6, 128.4, 128.3, 67.0.

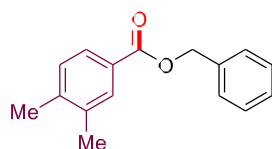


benzyl 4-phenoxybenzoate (**8**)⁴

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Yellow solid (46.2 mg, 76% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 8.05 (d, *J* = 8.8 Hz, 2H), 7.49 – 7.42 (m, 2H), 7.42 – 7.30 (m, 5H), 7.19 (t, *J* = 7.4 Hz, 1H), 7.06 (d, *J* = 7.5 Hz, 2H), 6.99 (d, *J* = 8.9 Hz, 2H), 5.36 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 166.0, 161.9, 155.6, 136.2, 131.8, 130.0, 128.6, 128.2, 128.1, 124.5, 124.4, 120.1, 117.3, 66.6.



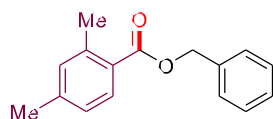
benzyl 3,4-dimethylbenzoate (**9**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (31.2 mg, 65% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 7.85 (s, 1H), 7.82 (d, *J* = 7.8 Hz, 1H), 7.45 (d, *J* = 7.0 Hz, 2H), 7.42 – 7.31 (m, 3H), 7.19 (d, *J* = 7.8 Hz, 1H), 5.36 (s, 2H), 2.32 (s, 3H), 2.30 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.7, 142.4, 136.8, 136.3, 130.7, 129.7, 128.6, 128.2, 128.1, 127.7, 127.3, 66.5, 20.0, 19.7.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₁₆H₁₇O₂ 241.1223; found: 241.1221.

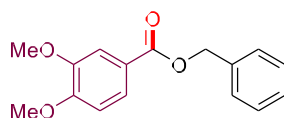


benzyl 2,4-dimethylbenzoate (**10**)⁵

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (22.1 mg, 46% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.88 (d, *J* = 7.8 Hz, 1H), 7.45 (d, *J* = 6.7 Hz, 2H), 7.42 – 7.30 (m, 3H), 7.04 (d, *J* = 10.6 Hz, 2H), 5.33 (s, 2H), 2.58 (s, 3H), 2.35 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 167.3, 142.6, 140.6, 136.3, 132.5, 130.9, 128.6, 128.2, 128.1, 126.5, 126.5, 66.3, 21.9, 21.4.

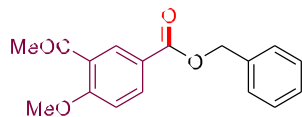


benzyl 3,4-dimethoxybenzoate (**11**)⁶

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a Colorless oil (29.9 mg, 55% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.77 – 7.69 (m, 1H), 7.58 (d, *J* = 2.0 Hz, 1H), 7.48 – 7.42 (m, 2H), 7.42 – 7.31 (m, 3H), 6.88 (d, *J* = 8.4 Hz, 1H), 5.35 (s, 2H), 3.93 (s, 3H), 3.92 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.3, 153.1, 148.7, 136.3, 128.6, 128.2, 128.1, 123.8, 122.6, 112.1, 110.3, 66.6, 56.0.



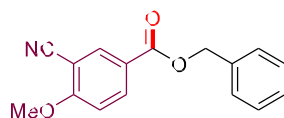
benzyl 3-acetyl-4-methoxybenzoate (**12**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a Yellow oil (31.3 mg, 55% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.41 (d, *J* = 2.3 Hz, 1H), 8.21 – 8.14 (m, 1H), 7.44 (d, *J* = 7.0 Hz, 2H), 7.41 – 7.29 (m, 3H), 7.00 (d, *J* = 8.8 Hz, 1H), 5.35 (s, 2H), 3.97 (s, 3H), 2.61 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 198.9, 165.6, 162.1, 136.0, 135.2, 132.3, 128.6, 128.3, 128.3, 122.7, 111.4, 66.7, 55.9, 31.6.

HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd. for C₁₇H₁₆O₄Na 307.0941; found: 307.0947.



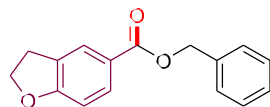
benzyl 3-cyano-4-methoxybenzoate (**13**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (31.0 mg, 58% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.34 – 8.20 (m, 2H), 7.50 – 7.32 (m, 5H), 7.01 (d, *J* = 8.9 Hz, 1H), 5.35 (s, 2H), 4.00 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 164.4, 164.3, 136.1, 135.7, 135.6, 128.7, 128.5, 128.3, 123.2, 115.4, 111.1, 102.2, 67.1, 56.5.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₁₆H₁₄NO₃ 268.0968; found: 268.0960.



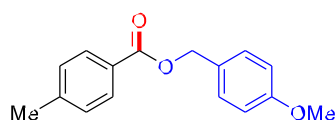
benzyl 2,3-dihydrobenzofuran-5-carboxylate (**14**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Yellow oil (39.1 mg, 77% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, *J* = 6.8 Hz, 2H), 7.44 (d, *J* = 6.6 Hz, 2H), 7.42 – 7.31 (m, 3H), 6.79 (d, *J* = 9.0 Hz, 1H), 5.33 (s, 2H), 4.64 (t, *J* = 8.8 Hz, 2H), 3.23 (t, *J* = 8.8 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 166.3, 164.3, 136.4, 131.3, 128.6, 128.1, 128.1, 127.4, 126.9, 122.6, 109.1, 72.1, 66.4, 29.1.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₁₆H₁₅O₃ 255.1016; found: 255.1015.

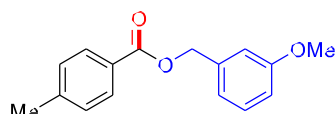


4-methoxybenzyl 4-methylbenzoate (**15**)²

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (37.4 mg, 73% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 8.2 Hz, 2H), 7.39 (d, *J* = 8.6 Hz, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 6.91 (d, *J* = 8.7 Hz, 2H), 5.29 (s, 2H), 3.82 (s, 3H), 2.40 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.6, 159.6, 143.6, 130.0, 129.7, 129.1, 128.3, 127.5, 114.0, 66.4, 55.3, 21.7.

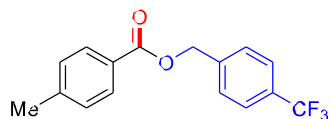


3-methoxybenzyl 4-methylbenzoate (**16**)⁷

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (40.0 mg, 78% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.30 (t, *J* = 7.9 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 7.6 Hz, 1H), 6.99 (s, 1H), 6.92 – 6.85 (m, 1H), 5.33 (s, 2H), 3.82 (s, 3H), 2.41 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.5, 159.8, 143.8, 137.8, 129.8, 129.7, 129.1, 127.4, 120.3, 113.6, 113.6, 66.4, 55.3, 21.7.



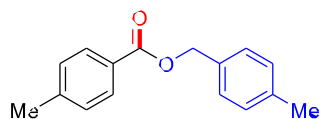
4-(trifluoromethyl)benzyl 4-methylbenzoate (**17**)²

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (57.6 mg, 98% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, and ¹⁹F NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 7.9 Hz, 2H), 7.64 (d, *J* = 7.9 Hz, 2H), 7.55 (d, *J* = 7.9 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 5.40 (s, 2H), 2.41 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.3, 144.1, 140.2, 130.4 (q, *J* = 32.5 Hz), 129.8, 129.2, 128.1, 127.0, 125.6 (q, *J* = 3.8 Hz), 124.1 (q, *J* = 272.2 Hz), 77.3, 77.0, 76.7, 65.5, 21.7.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.6.

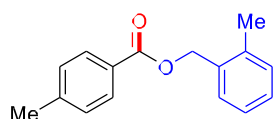


4-methylbenzyl 4-methylbenzoate (**18**)⁸

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (40.8 mg, 85% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 7.7 Hz, 2H), 7.25 – 7.17 (m, 4H), 5.32 (s, 2H), 2.41 (s, 3H), 2.38 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.6, 143.7, 138.0, 133.2, 129.8, 129.3, 129.1, 128.3, 127.5, 66.5, 21.7, 21.2.

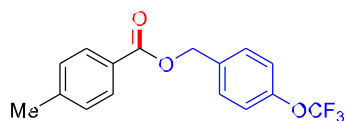


2-methylbenzyl 4-methylbenzoate (**19**)⁷

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (38.9 mg, 81% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.45 – 7.39 (m, 1H), 7.27 – 7.17 (m, 5H), 5.36 (s, 2H), 2.40 (s, 3H), 2.39 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.5, 143.7, 137.1, 134.2, 130.4, 129.7, 129.2, 129.1, 128.5, 127.4, 126.1, 65.1, 21.7, 19.0.



4-(trifluoromethoxy)benzyl 4-methylbenzoate (**20**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Yellow

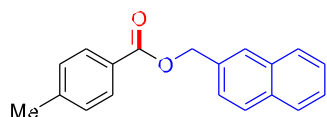
solid (52.1 mg, 84% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR, ^{19}F NMR, and HRMS.

^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 8.3$ Hz, 2H), 7.48 (d, $J = 8.6$ Hz, 2H), 7.26 – 7.21 (m, 4H), 5.35 (s, 2H), 2.41 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.4, 149.0, 144.0, 135.0, 129.7, 129.6, 129.2, 127.1, 121.1, 120.5 (q, $J = 257.4$ Hz), 65.5, 21.7.

^{19}F NMR (376 MHz, CDCl_3) δ -57.9.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{16}\text{H}_{14}\text{F}_3\text{O}_3$ 311.0890; found: 311.0891.



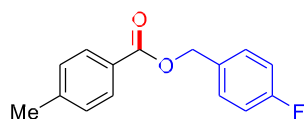
naphthalen-2-ylmethyl 4-methylbenzoate (**21**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (46.9 mg, 85% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR, and HRMS.

^1H NMR (400 MHz, CDCl_3) δ 8.00 (d, $J = 8.3$ Hz, 2H), 7.91 (s, 1H), 7.89 – 7.84 (m, 3H), 7.58 – 7.53 (m, 1H), 7.53 – 7.47 (m, 2H), 7.25 (d, $J = 8.2$ Hz, 2H), 5.52 (s, 2H), 2.41 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.6, 143.8, 133.6, 133.2, 133.1, 129.8, 129.1, 128.4, 128.0, 127.7, 127.4, 127.3, 126.3, 126.3, 125.9, 66.7, 21.7.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd. for $\text{C}_{19}\text{H}_{16}\text{O}_2\text{Na}$ 299.1043; found: 299.1046.



4-fluorobenzyl 4-methylbenzoate (**22**)

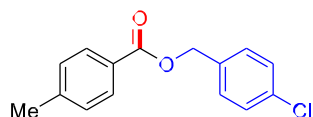
This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Yellow oil (43.9 mg, 90% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR, ^{19}F NMR and HRMS.

^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 8.3$ Hz, 2H), 7.47 – 7.38 (m, 2H), 7.24 (d, $J = 8.0$ Hz, 2H), 7.07 (t, $J = 8.7$ Hz, 2H), 5.31 (s, 2H), 2.41 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.5, 162.6 (d, $J = 246.8$ Hz), 143.8, 132.1 (d, $J = 3.3$ Hz), 130.2 (d, $J = 8.2$ Hz), 129.7, 129.1, 127.3, 115.5 (d, $J = 21.6$ Hz), 65.8, 21.7.

^{19}F NMR (376 MHz, CDCl_3) δ -113.8.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{15}\text{H}_{14}\text{FO}_2$ 245.0972; found: 245.0971.

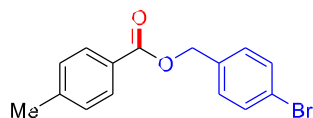


4-chlorobenzyl 4-methylbenzoate (**23**)⁷

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (45.3 mg, 87% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 8.1 Hz, 2H), 7.40 – 7.32 (m, 4H), 7.23 (d, *J* = 8.0 Hz, 2H), 5.30 (s, 2H), 2.40 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.4, 143.9, 134.7, 134.1, 129.7, 129.5, 129.2, 128.8, 127.2, 65.7, 21.7.

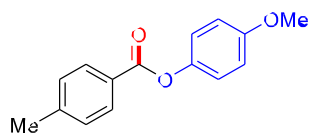


4-bromobenzyl 4-methylbenzoate (**24**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (52.3 mg, 86% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 5.29 (s, 2H), 2.41 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.4, 143.9, 135.3, 131.7, 129.8, 129.7, 129.2, 127.2, 122.2, 65.7, 21.7.

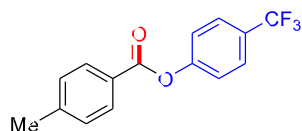


4-methoxyphenyl 4-methylbenzoate (**26**)²

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a White solid (45.0 mg, 93% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 8.09 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.15 – 7.10 (m, 2H), 6.97 – 6.92 (m, 2H), 3.83 (s, 3H), 2.45 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.7, 157.3, 144.5, 144.3, 130.2, 129.3, 126.9, 122.5, 114.5, 55.6, 21.8.



4-(trifluoromethyl)phenyl 4-methylbenzoate (**27**)

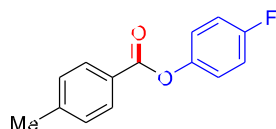
This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a White solid (33.0 mg, 59% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, ¹⁹F NMR and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.10 (d, *J* = 8.1 Hz, 2H), 7.70 (d, *J* = 8.5 Hz, 2H), 7.34 (t, *J* = 8.1 Hz, 4H), 2.47 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 164.8, 153.6, 145.0, 130.3, 129.4, 128.1 (q, *J* = 32.7 Hz), 126.8 (q, *J* = 3.8 Hz), 126.2, 123.9 (q, *J* = 271.9 Hz), 122.3, 21.8.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.2.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₁₅H₁₂F₃O₂ 281.0784; found: 281.0785.

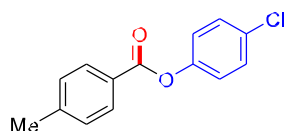


4-fluorophenyl 4-methylbenzoate (**28**)¹⁰

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 30/1) to afford the title compound as a White solid (32.2 mg, 70% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, and ¹⁹F NMR. **¹H NMR (400 MHz, CDCl₃)** δ 8.08 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.20 – 7.15 (m, 2H), 7.14 – 7.08 (m, 2H), 2.46 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.3, 160.3 (d, *J* = 244.0 Hz), 146.8 (d, *J* = 2.8 Hz), 144.6, 130.2, 129.4, 126.5, 123.2 (d, *J* = 8.6 Hz), 116.1 (d, *J* = 23.5 Hz), 21.8.

¹⁹F NMR (376 MHz, CDCl₃) δ -117.1.

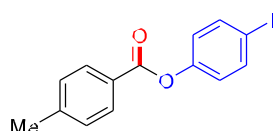


4-chlorophenyl 4-methylbenzoate (**29**)¹¹

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 30/1) to afford the title compound as a White solid (48.7 mg, 99% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, *J* = 8.2 Hz, 2H), 7.41 – 7.37 (m, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.19 – 7.14 (m, 2H), 2.46 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.0, 149.5, 144.7, 131.2, 130.3, 129.5, 129.4, 126.4, 123.2, 21.8.



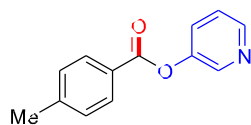
4-iodophenyl 4-methylbenzoate (**30**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 30/1) to afford the title compound as a White solid (48.7 mg, 72% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, *J* = 8.2 Hz, 2H), 7.73 (d, *J* = 8.7 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 6.99 (d, *J* = 8.8 Hz, 2H), 2.46 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 164.9, 150.9, 144.7, 138.5, 130.3, 129.4, 126.4, 124.0, 89.8, 21.8.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₁₄H₁₂IO₂ 338.9876; found: 338.9879.

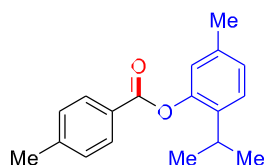


pyridin-3-yl 4-methylbenzoate (**31**)¹²

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a Yellow solid (26.8 mg, 63% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 8.55 (d, *J* = 2.7 Hz, 1H), 8.54 – 8.48 (m, 1H), 8.09 (d, *J* = 8.3 Hz, 2H), 7.65 – 7.57 (m, 1H), 7.40 – 7.36 (m, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 2.46 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 164.8, 147.7, 147.0, 145.0, 143.7, 130.4, 129.5, 126.0, 123.9, 21.8.



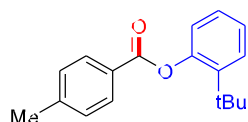
2-isopropyl-5-methylphenyl 4-methylbenzoate (**32**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (53.1 mg, 99% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.13 (d, *J* = 8.2 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 7.9 Hz, 1H), 7.08 (d, *J* = 7.9 Hz, 1H), 6.97 (s, 1H), 3.08 (h, *J* = 6.8 Hz, 1H), 2.48 (s, 3H), 2.36 (s, 3H), 1.23 (d, *J* = 6.9 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 165.5, 148.2, 144.4, 137.3, 136.6, 130.2, 129.4, 127.1, 126.9, 126.5, 123.0, 27.3, 23.1, 21.8, 20.9.

HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd. for C₁₈H₂₀O₂Na 291.1356; found: 291.1356.



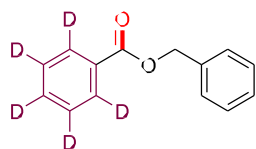
2-(*tert*-butyl)phenyl 4-methylbenzoate (**33**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure I**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (45.0 mg, 84% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.13 (d, *J* = 8.3 Hz, 2H), 7.47 – 7.43 (m, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.30 – 7.25 (m, 1H), 7.23 – 7.19 (m, 1H), 7.12 – 7.08 (m, 1H), 2.47 (s, 3H), 1.38 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 165.5, 149.5, 144.5, 141.4, 130.3, 129.4, 127.2, 127.2, 127.0, 125.7, 124.3, 34.6, 30.3, 21.8.

HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd. for C₁₈H₂₀O₂Na 291.1356; found: 291.1355.

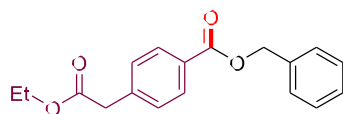


benzyl benzoate-*d*₅ (**34**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Yellow oil (38.6 mg, 89% yield). The identity of the product was confirmed by ¹H NMR and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.50 – 7.44 (m, 2H), 7.43 – 7.33 (m, 3H), 5.38 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 166.5, 136.1, 130.0, 129.6, 129.3, 129.1, 128.6, 128.3, 128.2, 127.9, 127.6, 66.7.



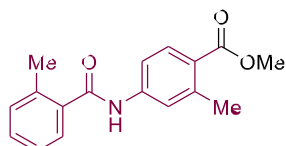
benzyl 4-(2-ethoxy-2-oxoethyl)benzoate (**35**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (54.3 mg, 91% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 8.3 Hz, 2H), 7.46 – 7.43 (m, 2H), 7.41 – 7.33 (m, 5H), 5.36 (s, 2H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.67 (s, 2H), 1.25 (t, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 170.8, 166.2, 139.4, 136.0, 129.9, 129.3, 128.9, 128.6, 128.2, 128.1, 66.6, 61.1, 41.4, 14.1.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₁₈H₁₉O₄ 299.1278; found: 299.1278.

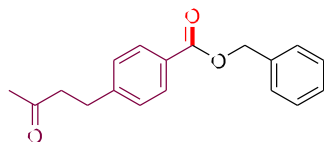


benzyl 4-(2-ethoxy-2-oxoethyl)benzoate (**36**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a Colorless oil (47.6 mg, 84% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.94 (d, *J* = 9.1 Hz, 1H), 7.75 (s, 1H), 7.52 (s, 2H), 7.45 (d, *J* = 7.6 Hz, 1H), 7.40 – 7.32 (m, 1H), 7.27 – 7.20 (m, 2H), 3.87 (s, 3H), 2.61 (s, 3H), 2.48 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 168.2, 167.4, 142.2, 141.2, 136.6, 136.0, 132.2, 131.4, 130.6, 126.6, 126.0, 125.0, 122.0, 116.5, 51.8, 22.1, 19.9.

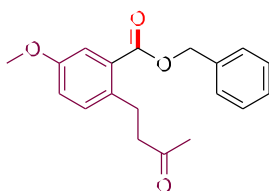


benzyl 4-(3-oxobutyl)benzoate (**37**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (51.9 mg, 92% yield). The identity of the product was confirmed by $^1\text{H NMR}$, $^{13}\text{C NMR}$, and HRMS. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.99 (d, $J = 8.3$ Hz, 2H), 7.44 (d, $J = 6.6$ Hz, 2H), 7.41 – 7.31 (m, 3H), 7.25 (d, $J = 8.1$ Hz, 2H), 5.35 (s, 2H), 2.94 (t, $J = 7.5$ Hz, 2H), 2.77 (t, $J = 7.5$ Hz, 2H), 2.14 (s, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 207.3, 166.4, 146.7, 136.1, 130.0, 128.6, 128.4, 128.2, 128.1, 128.1, 66.6, 44.6, 30.1, 29.6.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{18}\text{H}_{19}\text{O}_3$ 283.1329; found: 283.1327.

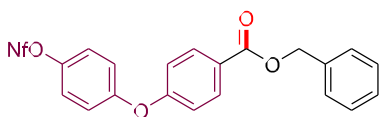


benzyl 5-methoxy-2-(3-oxobutyl)benzoate (**38**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (28.7 mg, 46% yield). The identity of the product was confirmed by $^1\text{H NMR}$, $^{13}\text{C NMR}$, and HRMS. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.63 (d, $J = 2.4$ Hz, 1H), 7.46 (d, $J = 6.8$ Hz, 2H), 7.38 (t, $J = 7.2$ Hz, 2H), 7.35 – 7.27 (m, 2H), 6.89 (d, $J = 8.5$ Hz, 1H), 5.34 (s, 2H), 3.88 (s, 3H), 2.87 – 2.81 (m, 2H), 2.75 – 2.70 (m, 2H), 2.12 (s, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 207.7, 166.0, 157.8, 136.2, 133.6, 132.6, 131.3, 128.5, 128.1, 128.0, 119.7, 112.2, 66.5, 56.1, 45.1, 30.1, 28.5.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{19}\text{H}_{21}\text{O}_4$ 313.1434; found: 313.1436.

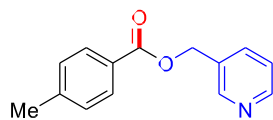


benzyl 4-(4-(perfluorobutoxy)phenoxy)benzoate (**39**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (50.3 mg, 83% yield). The identity of the product was confirmed by $^1\text{H NMR}$, and $^{13}\text{C NMR}$.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.09 (d, $J = 8.8$ Hz, 2H), 7.45 (d, $J = 6.8$ Hz, 2H), 7.43 – 7.33 (m, 3H), 7.29 (d, $J = 9.1$ Hz, 2H), 7.10 (d, $J = 9.2$ Hz, 2H), 7.03 (d, $J = 8.8$ Hz, 2H), 5.37 (s, 2H).

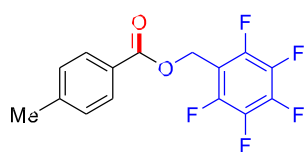
$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 165.7, 160.6, 155.7, 145.5, 136.0, 132.1, 128.6, 128.3, 128.2, 125.6, 123.1, 120.8, 118.1, 66.8.



pyridin-3-ylmethyl 4-methylbenzoate (**40**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (35.0 mg, 77% yield). The identity of the product was confirmed by $^1\text{H NMR}$, $^{13}\text{C NMR}$, and HRMS. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.72 (d, $J = 2.3$ Hz, 1H), 8.63 – 8.57 (m, 1H), 7.95 (d, $J = 8.3$ Hz, 2H), 7.77 (d, $J = 7.8$ Hz, 1H), 7.35 – 7.29 (m, 1H), 7.24 (d, $J = 8.0$ Hz, 2H), 5.36 (s, 2H), 2.40 (s, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 166.3, 149.7, 149.7, 144.0, 135.9, 131.8, 129.8, 129.2, 127.0, 123.5, 64.0, 21.7.

HRMS (ESI-TOF) m/z: $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{14}\text{H}_{14}\text{NO}_2$ 228.1019; found: 228.1017.



(perfluorophenyl)methyl 4-methylbenzoate (**41**)

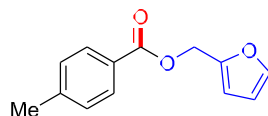
This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Yellow solid (58.8 mg, 93% yield). The identity of the product was confirmed by $^1\text{H NMR}$, $^{13}\text{C NMR}$, $^{19}\text{F NMR}$ and HRMS.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.90 (d, $J = 8.3$ Hz, 2H), 7.23 (d, $J = 8.0$ Hz, 2H), 5.43 (s, 2H), 2.41 (s, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 165.9, 146.5 (m), 144.5 (m), 144.3, 141.3 (m), 139.0 (m), 138.8 (m), 136.3 (m), 129.8, 129.2, 126.5, 53.7, 21.7.

$^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -141.5 – -141.8 (m, 2F), -152.5 – -152.8 (m, 1F), -161.4 – -161.8 (m, 2F).

HRMS (ESI-TOF) m/z: $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{15}\text{H}_{10}\text{F}_5\text{O}_2$ 317.0595; found: 317.0591.

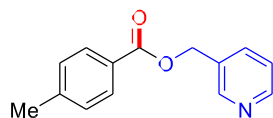


furan-2-ylmethyl 4-methylbenzoate (**42**)⁹

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Yellow oil (35.9 mg, 83% yield). The identity of the product was confirmed by $^1\text{H NMR}$, and $^{13}\text{C NMR}$.

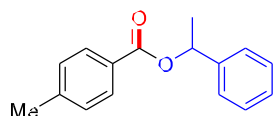
$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.3$ Hz, 2H), 7.47 – 7.42 (m, 1H), 7.22 (d, $J = 7.8$ Hz, 2H), 6.47 (d, $J = 3.2$ Hz, 1H), 6.42 – 6.36 (m, 1H), 5.29 (s, 2H), 2.39 (s, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 166.3, 149.7, 143.8, 143.2, 129.8, 129.1, 127.2, 110.7, 110.6, 58.4, 21.7.



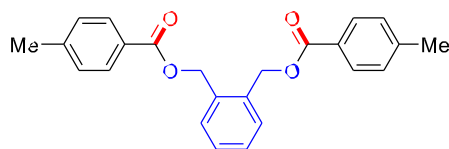
thiophen-2-ylmethyl 4-methylbenzoate (**43**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (45.5 mg, 98% yield). The identity of the product was confirmed by $^1\text{H NMR}$, $^{13}\text{C NMR}$ and HRMS. **$^1\text{H NMR}$ (400 MHz, CDCl_3)** δ 7.96 (d, $J = 8.3$ Hz, 2H), 7.36 – 7.30 (m, 1H), 7.23 (d, $J = 8.0$ Hz, 2H), 7.17 (d, $J = 3.0$ Hz, 1H), 7.03 – 6.99 (m, 1H), 5.50 (s, 2H), 2.40 (s, 3H). **$^{13}\text{C NMR}$ (101 MHz, CDCl_3)** δ 166.3, 143.8, 138.2, 129.8, 129.1, 128.1, 127.2, 126.8, 126.8, 60.9, 21.7. **HRMS (ESI-TOF) m/z:** $[\text{M}+\text{Na}]^+$ Calcd. for $\text{C}_{13}\text{H}_{12}\text{O}_2\text{SNa}$ 255.0450; found: 255.0447.



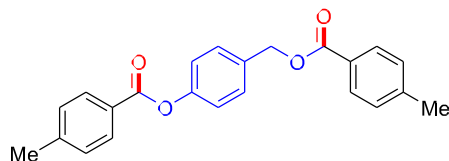
1-phenylethyl 4-methylbenzoate (**44**)⁷

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (27.9 mg, 58% yield). The identity of the product was confirmed by $^1\text{H NMR}$, and $^{13}\text{C NMR}$. **$^1\text{H NMR}$ (400 MHz, CDCl_3)** δ 7.97 (d, $J = 8.3$ Hz, 2H), 7.44 (d, $J = 6.9$ Hz, 2H), 7.36 (t, $J = 7.4$ Hz, 2H), 7.31 – 7.28 (m, 1H), 7.23 (d, $J = 7.9$ Hz, 2H), 6.12 (q, $J = 6.6$ Hz, 1H), 2.40 (s, 3H), 1.66 (d, $J = 6.6$ Hz, 3H). **$^{13}\text{C NMR}$ (101 MHz, CDCl_3)** δ 165.9, 143.6, 142.0, 129.7, 129.1, 128.5, 127.8, 126.0, 72.7, 22.5, 21.7.



1,2-phenylenebis(methylene) bis(4-methylbenzoate) (**45**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 30/1) to afford the title compound as a Colorless oil (38.2 mg, 51% yield). The identity of the product was confirmed by $^1\text{H NMR}$, $^{13}\text{C NMR}$ and HRMS. **$^1\text{H NMR}$ (400 MHz, CDCl_3)** δ 7.92 (d, $J = 8.2$ Hz, 4H), 7.56 – 7.51 (m, 2H), 7.42 – 7.37 (m, 2H), 7.18 (d, $J = 8.0$ Hz, 4H), 5.51 (s, 4H), 2.39 (s, 6H). **$^{13}\text{C NMR}$ (101 MHz, CDCl_3)** δ 166.4, 143.7, 134.9, 129.9, 129.7, 129.1, 128.8, 127.2, 64.2, 21.7. **HRMS (ESI-TOF) m/z:** $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{24}\text{H}_{23}\text{O}_4$ 375.1591; found: 375.1596.



4-((4-methylbenzoyl)oxy)benzyl 4-methylbenzoate (**46**)

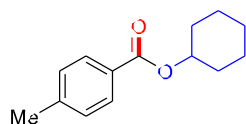
This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 30/1) to afford the title compound as a Yellow oil

(37.5 mg, 52% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR and HRMS.

^1H NMR (400 MHz, CDCl_3) δ 8.13 (d, $J = 8.1$ Hz, 2H), 8.01 (d, $J = 8.2$ Hz, 2H), 7.55 (d, $J = 8.6$ Hz, 2H), 7.35 (d, $J = 8.0$ Hz, 2H), 7.31 – 7.26 (m, 4H), 5.40 (s, 2H), 2.49 (s, 3H), 2.45 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.5, 165.2, 150.9, 144.5, 143.8, 133.8, 130.3, 129.8, 129.5, 129.3, 129.1, 127.3, 126.7, 122.0, 66.0, 21.8, 21.7.

HRMS (ESI-TOF) m/z: $[\text{M}+\text{Na}]^+$ Calcd. for $\text{C}_{23}\text{H}_{20}\text{O}_4\text{Na}$ 383.1254; found: 383.1251.



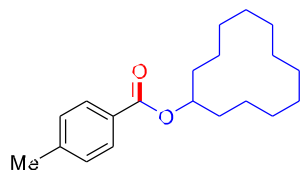
cyclohexyl 4-methylbenzoate (**47**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (40.1 mg, 92% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR and HRMS.

^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.2$ Hz, 2H), 7.23 (d, $J = 7.9$ Hz, 2H), 5.09 – 4.93 (m, 1H), 2.40 (s, 3H), 1.99 – 1.89 (m, 2H), 1.84 – 1.74 (m, 2H), 1.60 – 1.34 (m, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.1, 143.3, 129.6, 129.0, 128.3, 72.8, 31.7, 25.5, 23.7, 21.6.

HRMS (ESI-TOF) m/z: $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{14}\text{H}_{19}\text{O}_2$ 219.1380; found: 219.1375.



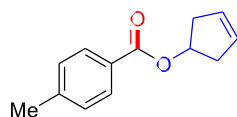
cyclododecyl 4-methylbenzoate (**48**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (58.0 mg, 96% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR and HRMS.

^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, $J = 8.2$ Hz, 2H), 7.22 (d, $J = 8.0$ Hz, 2H), 5.29 – 5.21 (m, 1H), 2.40 (s, 3H), 1.87 – 1.78 (m, 2H), 1.69 – 1.60 (m, 2H), 1.51 – 1.32 (m, 18H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.4, 143.3, 129.6, 129.0, 128.2, 72.7, 29.1, 24.2, 24.0, 23.4, 23.2, 21.6, 20.9.

HRMS (ESI-TOF) m/z: $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{20}\text{H}_{31}\text{O}_2$ 303.2319; found: 303.2317.



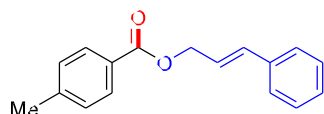
cyclopent-3-en-1-yl 4-methylbenzoate (**49**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (30.7 mg, 76% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR and HRMS.

^1H NMR (400 MHz, CDCl_3) δ 7.91 (d, $J = 8.2$ Hz, 2H), 7.22 (d, $J = 7.9$ Hz, 2H), 5.76 (s, 2H), 5.64 – 5.57 (m, 1H), 2.89 – 2.81 (m, 2H), 2.58 – 2.51 (m, 2H), 2.40 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.6, 143.4, 129.6, 129.0, 128.4, 127.9, 74.6, 39.8, 21.7.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{13}\text{H}_{15}\text{O}_2$ 203.1067; found: 203.1073.

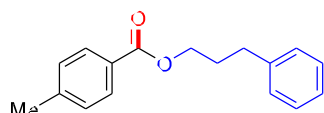


cinnamyl 4-methylbenzoate (**50**)¹⁰

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (35.3 mg, 70% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, CDCl_3) δ 7.97 (d, $J = 8.3$ Hz, 2H), 7.43 – 7.39 (m, 2H), 7.35 – 7.30 (m, 2H), 7.28 – 7.22 (m, 3H), 6.73 (d, $J = 15.9$ Hz, 1H), 6.44 – 6.36 (m, 1H), 4.96 (dd, $J = 6.4, 1.4$ Hz, 2H), 2.40 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.5, 143.7, 136.3, 134.1, 129.7, 129.1, 128.6, 128.1, 127.5, 126.7, 123.5, 65.4, 21.7.

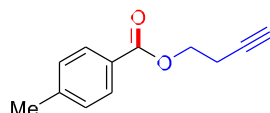


3-phenylpropyl 4-methylbenzoate (**51**)¹³

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (50.3 mg, 99% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, CDCl_3) δ 7.92 (d, $J = 8.3$ Hz, 2H), 7.32 – 7.27 (m, 2H), 7.25 – 7.17 (m, 5H), 4.32 (t, $J = 6.5$ Hz, 2H), 2.81 – 2.76 (m, 2H), 2.41 (s, 3H), 2.13 – 2.06 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.7, 143.5, 141.3, 129.6, 129.1, 128.5, 128.5, 127.7, 126.0, 64.1, 32.3, 30.4, 21.7.



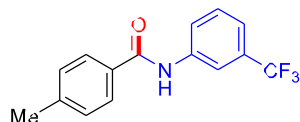
but-3-yn-1-yl 4-methylbenzoate (**52**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (33.1 mg, 88% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR and HRMS.

^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 8.2$ Hz, 2H), 7.24 (d, $J = 7.9$ Hz, 2H), 4.41 (t, $J = 6.8$ Hz, 2H), 2.69 – 2.64 (m, 2H), 2.41 (s, 3H), 2.02 (t, $J = 2.7$ Hz, 1H).

^{13}C NMR (176 MHz, CDCl_3) δ 166.4, 143.8, 129.7, 129.1, 127.2, 80.1, 69.9, 62.4, 21.7, 19.1.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{12}\text{H}_{13}\text{O}_2$ 189.0910; found: 189.0909.



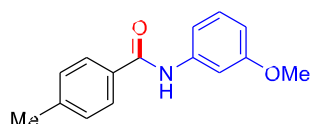
4-methyl-*N*-(3-(trifluoromethyl)phenyl)benzamide (**53**)¹⁴

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (46.9 mg, 84% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and ¹⁹F NMR.

¹H NMR (400 MHz, CDCl₃) δ 8.09 (s, 1H), 7.93 (s, 1H), 7.85 (d, *J* = 8.3 Hz, 1H), 7.76 (d, *J* = 8.3 Hz, 2H), 7.45 (t, *J* = 7.9 Hz, 1H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.26 (d, *J* = 7.9 Hz, 2H), 2.41 (s, 3H).

¹³C NMR (176 MHz, CDCl₃) δ 165.9, 142.9, 138.6, 131.5, 131.4 (q, *J* = 32.5 Hz), 129.6, 129.5, 127.1, 123.9 (q, *J* = 272.5 Hz), 123.3, 121.0 (q, *J* = 3.8 Hz), 116.9 (q, *J* = 4.1 Hz), 21.5.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.7.

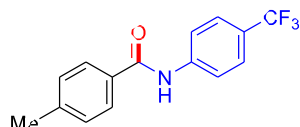


N-(3-methoxyphenyl)-4-methylbenzamide (**54**)¹⁴

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (38.1 mg, 79% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.93 (s, 1H), 7.79 (d, *J* = 8.2 Hz, 2H), 7.49 (s, 1H), 7.33 – 7.27 (m, 3H), 7.16 – 7.10 (m, 1H), 6.77 – 6.70 (m, 1H), 3.85 (s, 3H), 2.45 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.7, 160.2, 142.4, 139.3, 132.1, 129.7, 129.5, 127.0, 112.2, 110.5, 105.7, 55.3, 21.5.



4-methyl-*N*-(4-(trifluoromethyl)phenyl)benzamide (**55**)

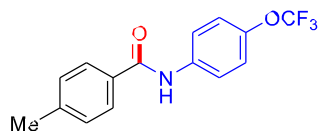
This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (48.6 mg, 87% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, ¹⁹F NMR and HRMS.

¹H NMR (400 MHz, DMSO-*d*₆) δ 10.51 (s, 1H), 8.03 (d, *J* = 8.5 Hz, 2H), 7.90 (d, *J* = 8.2 Hz, 2H), 7.72 (d, *J* = 8.6 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 2.40 (s, 3H).

¹³C NMR (101 MHz, DMSO) δ 166.3, 143.4, 142.5, 132.0, 129.5, 128.3, 126.4 (q, *J* = 3.7 Hz), 124.9 (d, *J* = 271.5 Hz), 123.9 (d, *J* = 31.9 Hz), 120.5, 21.5.

¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -60.3.

HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd. for C₁₅H₁₃F₃NO 280.0944; found: 280.0950.

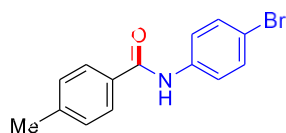


4-methyl-*N*-(4-(trifluoromethoxy)phenyl)benzamide (**56**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (53.7 mg, 91% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR, and ^{19}F NMR. ^1H NMR (400 MHz, DMSO- d_6) δ 10.36 (s, 1H), 7.90 (t, J = 8.1 Hz, 4H), 7.40 – 7.32 (m, 4H), 2.39 (s, 3H).

^{13}C NMR (101 MHz, DMSO) δ 166.0, 144.2, 142.3, 139.0, 132.2, 129.4, 128.2, 122.1, 121.9, 119.4, 21.5.

^{19}F NMR (376 MHz, DMSO- d_6) δ -57.0.

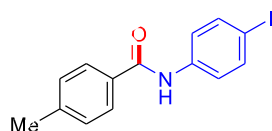


N-(4-bromophenyl)-4-methylbenzamide (**57**)¹⁴

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (50.3 mg, 87% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, DMSO- d_6) δ 10.29 (s, 1H), 7.87 (d, J = 8.2 Hz, 2H), 7.77 (d, J = 8.9 Hz, 2H), 7.53 (d, J = 8.9 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 2.39 (s, 3H).

^{13}C NMR (101 MHz, DMSO) δ 165.9, 142.3, 139.1, 132.3, 131.9, 129.4, 128.2, 122.6, 115.7, 21.5.

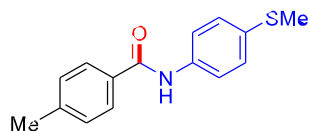


N-(4-iodophenyl)-4-methylbenzamide (**58**)¹⁵

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (43.8 mg, 65% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, DMSO- d_6) δ 10.26 (s, 1H), 7.87 (d, J = 8.2 Hz, 2H), 7.69 (d, J = 8.9 Hz, 2H), 7.64 (d, J = 8.9 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 2.39 (s, 3H).

^{13}C NMR (101 MHz, DMSO) δ 165.9, 142.2, 139.6, 137.7, 132.3, 129.4, 128.2, 122.9, 87.7, 21.5.

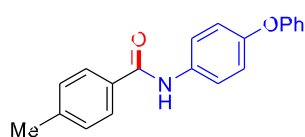


4-methyl-*N*-(4-(methylthio)phenyl)benzamide (**59**)¹⁶

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (44.2 mg, 86% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, DMSO-*d*₆) δ 10.17 (s, 1H), 7.87 (d, *J* = 8.1 Hz, 2H), 7.75 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.27 (d, *J* = 8.7 Hz, 2H), 2.47 (s, 3H), 2.39 (s, 3H).

¹³C NMR (101 MHz, DMSO) δ 165.7, 142.0, 137.2, 132.6, 132.4, 129.4, 128.1, 127.3, 121.4, 21.5, 15.9.

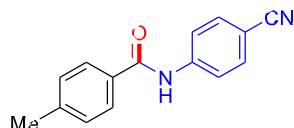


4-methyl-*N*-(4-phenoxyphenyl)benzamide (**60**)¹⁷

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (38.8 mg, 64% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, DMSO-*d*₆) δ 10.23 (s, 1H), 7.90 (d, *J* = 8.2 Hz, 2H), 7.82 (d, *J* = 9.0 Hz, 2H), 7.42 – 7.29 (m, 4H), 7.11 (t, *J* = 7.4 Hz, 1H), 7.04 (d, *J* = 9.0 Hz, 2H), 7.00 (d, *J* = 7.5 Hz, 2H), 2.39 (s, 3H).

¹³C NMR (101 MHz, DMSO) δ 165.7, 157.8, 152.5, 142.0, 135.6, 132.5, 130.4, 129.4, 128.1, 123.5, 122.5, 119.7, 118.4, 21.5.

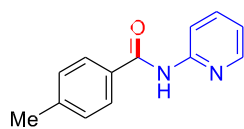


N-(4-cyanophenyl)-4-methylbenzamide (**61**)¹⁴

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (24.1 mg, 51% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, DMSO-*d*₆) δ 10.57 (s, 1H), 8.01 (d, *J* = 8.8 Hz, 2H), 7.89 (d, *J* = 8.2 Hz, 2H), 7.82 (d, *J* = 8.8 Hz, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 2.40 (s, 3H).

¹³C NMR (101 MHz, DMSO) δ 166.4, 144.1, 142.7, 133.6, 131.9, 129.5, 128.4, 120.6, 119.6, 105.7, 21.5.



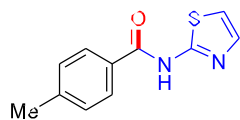
4-methyl-*N*-(pyridin-2-yl)benzamide (**62**)¹⁴

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was

purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a White solid (21.2 mg, 50% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, DMSO- d_6) δ 10.70 (s, 1H), 8.43 – 8.34 (m, 1H), 8.20 (d, J = 8.3 Hz, 1H), 7.96 (d, J = 8.2 Hz, 2H), 7.89 – 7.80 (m, 1H), 7.32 (d, J = 8.0 Hz, 2H), 7.20 – 7.12 (m, 1H), 2.38 (s, 3H).

^{13}C NMR (101 MHz, DMSO) δ 166.3, 152.7, 148.4, 142.5, 138.5, 131.7, 129.4, 128.5, 120.2, 115.2, 21.5.

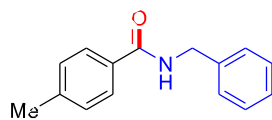


4-methyl-*N*-(thiazol-2-yl)benzamide (**63**)¹⁴

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a White solid (43.2 mg, 99% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, DMSO- d_6) δ 12.57 (s, 1H), 8.01 (d, J = 8.2 Hz, 2H), 7.56 (d, J = 3.5 Hz, 1H), 7.34 (d, J = 8.0 Hz, 2H), 7.27 (d, J = 3.6 Hz, 1H), 2.38 (s, 3H).

^{13}C NMR (101 MHz, DMSO) δ 165.4, 159.2, 143.2, 138.1, 129.8, 129.6, 128.6, 114.2, 21.5.

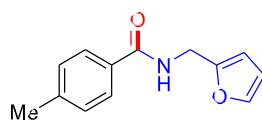


N-benzyl-4-methylbenzamide (**64**)¹⁷

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (44.6 mg, 99% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, DMSO- d_6) δ 9.00 (t, J = 6.0 Hz, 1H), 7.83 (d, J = 8.2 Hz, 2H), 7.32 (d, J = 4.4 Hz, 4H), 7.29 – 7.20 (m, 3H), 4.49 (d, J = 6.0 Hz, 2H), 2.35 (s, 3H).

^{13}C NMR (101 MHz, DMSO) δ 166.6, 141.6, 140.3, 132.0, 129.3, 128.7, 127.8, 127.7, 127.2, 43.0, 21.4.

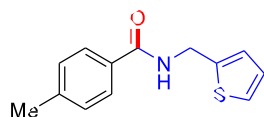


N-(furan-2-ylmethyl)-4-methylbenzamide (**65**)¹⁸

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (40.9 mg, 95% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, DMSO- d_6) δ 8.91 (t, J = 5.8 Hz, 1H), 7.80 (d, J = 8.3 Hz, 2H), 7.57 (s, 1H), 7.26 (d, J = 7.9 Hz, 2H), 6.43 – 6.35 (m, 1H), 6.27 (d, J = 3.2 Hz, 1H), 4.47 (d, J = 5.7 Hz, 2H), 2.34 (s, 3H).

^{13}C NMR (101 MHz, DMSO) δ 166.4, 153.0, 142.4, 141.6, 131.8, 129.3, 127.8, 110.9, 107.3, 36.5, 21.4.



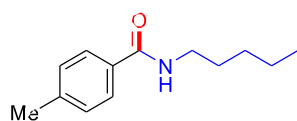
4-methyl-*N*-(thiophen-2-ylmethyl)benzamide (**66**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (37.0 mg, 80% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR and HRMS.

^1H NMR (400 MHz, DMSO- d_6) δ 9.08 (t, J = 6.0 Hz, 1H), 7.80 (d, J = 8.2 Hz, 2H), 7.41 – 7.34 (m, 1H), 7.27 (d, J = 8.0 Hz, 2H), 7.04 – 7.00 (m, 1H), 6.98 – 6.94 (m, 1H), 4.63 (d, J = 5.9 Hz, 2H), 2.34 (s, 3H).

^{13}C NMR (101 MHz, DMSO) δ 166.4, 143.3, 141.7, 131.8, 129.3, 127.8, 127.1, 125.8, 125.4, 38.2, 21.4.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd. for $\text{C}_{13}\text{H}_{13}\text{NOSNa}$ 254.0610; found: 254.0614.

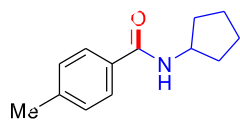


4-methyl-*N*-pentylbenzamide (**67**)¹⁹

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a White solid (37.7 mg, 92% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, DMSO- d_6) δ 8.37 (t, J = 5.7 Hz, 1H), 7.75 (d, J = 8.2 Hz, 2H), 7.25 (d, J = 8.0 Hz, 2H), 3.30 – 3.18 (m, 2H), 2.34 (s, 3H), 1.52 (p, J = 7.2 Hz, 2H), 1.37 – 1.22 (m, 4H), 0.93 – 0.81 (m, 3H).

^{13}C NMR (101 MHz, DMSO) δ 166.4, 141.2, 132.4, 129.2, 127.6, 39.5, 29.3, 29.2, 22.4, 21.4, 14.4.

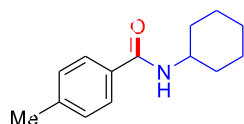


N-cyclopentyl-4-methylbenzamide (**68**)²⁰

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (30.1 mg, 74% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

^1H NMR (400 MHz, CDCl_3) δ 7.64 (d, J = 8.2 Hz, 2H), 7.20 (d, J = 8.0 Hz, 2H), 6.08 (s, 1H), 4.39 (h, J = 7.0 Hz, 1H), 2.38 (s, 3H), 2.12 – 2.03 (m, 2H), 1.76 – 1.60 (m, 4H), 1.53 – 1.43 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 167.1, 141.6, 132.1, 129.1, 126.8, 51.6, 33.3, 23.8, 21.4.

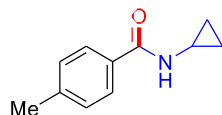


N-cyclohexyl-4-methylbenzamide (**69**)¹⁷

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (39.1 mg, 90% yield). The identity of the product was confirmed by ^1H NMR, and ^{13}C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 7.9 Hz, 2H), 6.01 (d, *J* = 8.0 Hz, 1H), 4.01 – 3.88 (m, 1H), 2.38 (s, 3H), 2.06 – 1.98 (m, 2H), 1.78 – 1.70 (m, 2H), 1.68 – 1.61 (m, 1H), 1.47 – 1.35 (m, 2H), 1.27 – 1.16 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.6, 141.6, 132.2, 129.1, 126.8, 48.6, 33.3, 25.6, 25.0, 21.4.

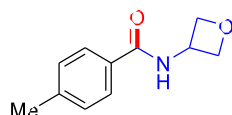


N-cyclopropyl-4-methylbenzamide (**70**)²²

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (24.9 mg, 71% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.63 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 6.39 (s, 1H), 2.92 – 2.82 (m, 1H), 2.37 (s, 3H), 0.87 – 0.79 (m, 2H), 0.64 – 0.57 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 168.9, 141.8, 131.6, 129.2, 126.9, 23.1, 21.4, 6.8.

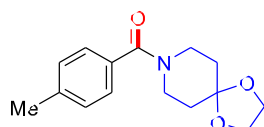


4-methyl-*N*-(oxetan-3-yl)benzamide (**71**)²¹

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (29.0 mg, 76% yield). The identity of the product was confirmed by ¹H NMR, and ¹³C NMR.

¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 6.93 (s, 1H), 5.27 – 5.17 (m, 1H), 4.98 (t, *J* = 7.1 Hz, 2H), 4.60 (t, *J* = 6.6 Hz, 2H), 2.39 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 167.1, 142.4, 130.9, 129.3, 127.0, 78.6, 45.2, 21.5.



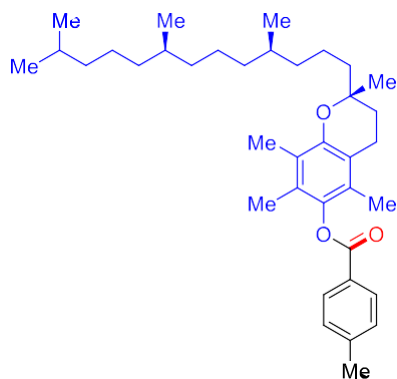
(1,4-dioxo-8-azaspiro[4.5]decan-8-yl)(*p*-tolyl)methanone (**72**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a Colorless oil (44.9 mg, 86% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 7.29 (d, *J* = 8.1 Hz, 2H), 7.19 (d, *J* = 7.8 Hz, 2H), 3.97 (s, 4H), 3.66 (d, *J* = 128.6 Hz, 4H), 2.36 (s, 3H), 1.86 – 1.60 (m, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 170.6, 139.8, 133.1, 129.1, 127.0, 107.0, 64.5, 45.8, 40.3, 35.7, 34.8, 21.4.

HRMS (ESI-TOF) *m/z*: [M+Na]⁺ Calcd. for C₁₅H₁₉NO₃Na 284.1257; found: 284.1256.



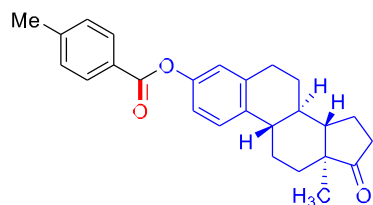
(*R*)-2,5,7,8-tetramethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl 4-methylbenzoate (**73**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 3/1) to afford the title compound as a Colorless oil (80.1 mg, 73% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR and HRMS.

^1H NMR (700 MHz, CDCl_3) δ 8.16 (d, $J = 8.0$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H), 2.64 (t, $J = 6.9$ Hz, 2H), 2.48 (s, 3H), 2.14 (s, 3H), 2.08 (s, 3H), 2.04 (s, 3H), 1.87 – 1.76 (m, 2H), 1.62 – 1.53 (m, 3H), 1.51 – 1.46 (m, 1H), 1.45 – 1.39 (m, 3H), 1.36 – 1.25 (m, 11H), 1.18 – 1.07 (m, 6H), 0.91 – 0.87 (m, 12H).

^{13}C NMR (176 MHz, CDCl_3) δ 165.3, 149.4, 144.2, 140.7, 130.2, 129.3, 127.0, 127.0, 125.2, 123.1, 117.5, 75.1, 40.4, 39.7, 39.4, 37.5, 37.3, 32.8, 31.3, 31.1, 28.0, 24.9, 24.5, 24.2, 23.8, 22.8, 22.7, 21.8, 21.1, 20.7, 19.8, 19.7, 13.1, 12.2, 11.9.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{NH}_4]^+$ Calcd. for $\text{C}_{37}\text{H}_{60}\text{NO}_3$ 566.4568; found: 566.4574.



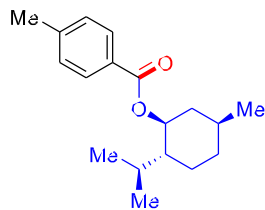
(8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-3-yl 4-methylbenzoate (**74**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a White solid (63.7 mg, 82% yield). The identity of the product was confirmed by ^1H NMR, ^{13}C NMR and HRMS.

^1H NMR (700 MHz, CDCl_3) δ 8.08 (d, $J = 8.2$ Hz, 2H), 7.33 (d, $J = 8.5$ Hz, 1H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.00 – 6.97 (m, 1H), 6.94 (d, $J = 2.6$ Hz, 1H), 2.99 – 2.91 (m, 2H), 2.54 – 2.49 (m, 1H), 2.45 (s, 3H), 2.44 – 2.41 (m, 1H), 2.33 – 2.29 (m, 1H), 2.19 – 2.12 (m, 1H), 2.08 – 2.01 (m, 2H), 2.00 – 1.96 (m, 1H), 1.66 – 1.56 (m, 3H), 1.55 – 1.45 (m, 3H), 0.93 (s, 3H).

^{13}C NMR (176 MHz, CDCl_3) δ 220.8, 165.5, 148.9, 144.3, 138.1, 137.3, 130.2, 129.3, 126.9, 126.5, 121.8, 118.9, 50.5, 48.0, 44.2, 38.1, 35.9, 31.6, 29.5, 26.4, 25.8, 21.8, 21.6, 13.9.

HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{26}\text{H}_{29}\text{O}_3$ 389.2111; found: 389.2120.

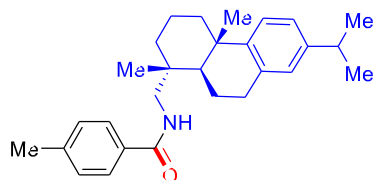


(1*S*,2*R*,5*S*)-2-isopropyl-5-methylcyclohexyl 4-methylbenzoate (**75**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 10/1) to afford the title compound as a Colorless oil (51.0 mg, 93% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and HRMS. **¹H NMR (400 MHz, CDCl₃)** δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 7.9 Hz, 2H), 4.96 – 4.88 (m, 1H), 2.41 (s, 3H), 2.17 – 2.09 (m, 1H), 2.01 – 1.92 (m, 1H), 1.77 – 1.68 (m, 2H), 1.60 – 1.50 (m, 2H), 1.19 – 1.07 (m, 2H), 0.96 – 0.89 (m, 7H), 0.79 (d, *J* = 7.0 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.2, 143.3, 129.6, 129.0, 128.2, 74.6, 47.3, 41.0, 34.4, 31.5, 26.5, 23.7, 22.1, 21.6, 20.8, 16.6.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₁₈H₂₇O₂ 275.2006; found: 275.2007.



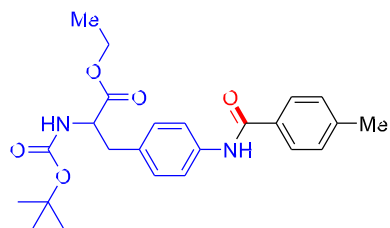
N-(((1*R*,4*aS*,10*aR*)-7-isopropyl-1,4*a*-dimethyl-1,2,3,4,4*a*,9,10,10*a*-octahydrophenanthren-1-yl)methyl)-4-methylbenzamide (**76**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a White solid (42.7 mg, 53% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 7.63 (d, *J* = 8.2 Hz, 2H), 7.21 (d, *J* = 7.9 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 1H), 7.02 – 6.96 (m, 1H), 6.88 (d, *J* = 2.0 Hz, 1H), 6.09 (t, *J* = 6.4 Hz, 1H), 3.48 – 3.29 (m, 2H), 2.97 – 2.90 (m, 1H), 2.87 – 2.78 (m, 2H), 2.38 (s, 3H), 2.30 (d, *J* = 13.0 Hz, 1H), 2.01 – 1.93 (m, 1H), 1.84 – 1.73 (m, 2H), 1.72 – 1.66 (m, 1H), 1.56 – 1.46 (m, 2H), 1.43 – 1.34 (m, 2H), 1.23 (d, *J* = 3.7 Hz, 6H), 1.21 (s, 3H), 1.01 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 167.5, 147.1, 145.6, 141.8, 134.8, 132.0, 129.3, 127.0, 126.8, 124.3, 123.9, 50.3, 45.8, 38.4, 37.7, 37.6, 36.4, 33.4, 30.5, 25.5, 24.0, 24.0, 21.4, 19.1, 18.8, 18.7.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₂₈H₃₈NO 404.2948; found: 404.2942.



ethyl 2-((*tert*-butoxycarbonyl)amino)-3-(4-(4-methylbenzamido)phenyl)propanoate (**77**)

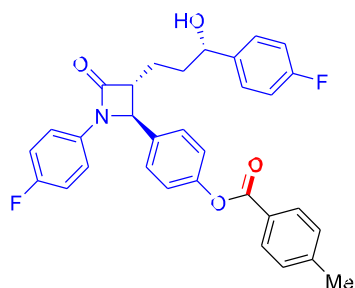
This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was

purified by silica gel chromatography (PE/EA = 1/0 to 3/1) to afford the title compound as a White solid (80.1 mg, 94% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.18 (s, 1H), 7.75 (d, *J* = 8.0 Hz, 2H), 7.57 (s, 2H), 7.22 (d, *J* = 7.9 Hz, 2H), 7.09 (d, *J* = 8.3 Hz, 2H), 5.03 (d, *J* = 8.2 Hz, 1H), 4.51 (d, *J* = 7.0 Hz, 1H), 4.20 – 4.10 (m, 2H), 3.19 – 2.90 (m, 2H), 2.39 (s, 3H), 1.42 (s, 9H), 1.23 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 171.9, 165.8, 155.2, 142.3, 137.2, 132.1, 132.0, 129.9, 129.3, 127.1, 120.3, 79.9, 61.4, 54.5, 37.7, 28.3, 21.5, 14.2.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₂₄H₃₁N₂O₅ 427.2227; found: 427.2236.



4-((2*S*,3*R*)-1-(4-fluorophenyl)-3-((*S*)-3-(4-fluorophenyl)-3-hydroxypropyl)-4-oxoazetidin-2-yl)phenyl 4-methylbenzoate (**78**)

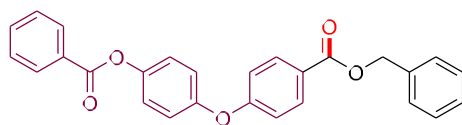
This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a White solid (85.4 mg, 81% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR, ¹⁹F NMR and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, *J* = 8.1 Hz, 2H), 7.51 – 7.45 (m, 2H), 7.42 – 7.36 (m, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.20 – 7.11 (m, 4H), 7.04 (t, *J* = 8.7 Hz, 2H), 6.93 (t, *J* = 8.6 Hz, 2H), 6.76 (s, 1H), 4.80 (d, *J* = 9.1 Hz, 1H), 4.68 – 4.60 (m, 1H), 2.45 (s, 3H), 2.42 – 2.29 (m, 2H), 2.23 – 2.16 (m, 1H), 2.09 – 2.03 (m, 1H), 1.76 – 1.66 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 170.8, 165.3, 162.2 (d, *J* = 245.1 Hz), 159.6 (d, *J* = 243.8 Hz), 150.8, 144.6, 138.5, 138.2 (d, *J* = 3.2 Hz), 132.9 (d, *J* = 2.7 Hz), 130.2, 129.4, 127.6, 127.4 (d, *J* = 8.1 Hz), 126.6, 122.8 (d, *J* = 8.0 Hz), 122.1, 115.5 (d, *J* = 22.5 Hz), 115.2 (d, *J* = 21.4 Hz), 81.5, 79.2, 53.2, 32.6, 27.6, 21.8.

¹⁹F NMR (376 MHz, CDCl₃) δ -115.0, -117.6.

HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd. for C₃₂H₂₇F₂NO₄Na 550.1800; found: 550.1808.



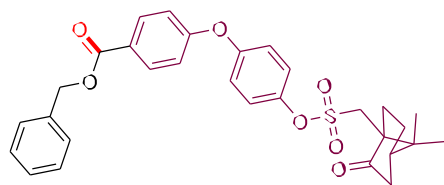
benzyl 4-(4-(benzoyloxy)phenoxy)benzoate (**79**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a White solid (70.4 mg, 83% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and HRMS.

¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, *J* = 6.9 Hz, 2H), 8.06 (d, *J* = 8.8 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.7 Hz, 2H), 7.44 (d, *J* = 6.9 Hz, 2H), 7.41 – 7.30 (m, 3H), 7.24 (d, *J* = 8.9 Hz, 2H), 7.11 (d, *J* = 8.9 Hz, 2H), 7.02 (d, *J* = 8.8 Hz, 2H), 5.35 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 165.9, 165.2, 161.8, 153.2, 147.3, 136.2, 133.8, 131.9, 130.2, 129.4, 128.7, 128.6, 128.2, 128.2, 124.7, 123.2, 120.9, 117.4, 66.6.

HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd. for C₂₇H₂₁O₅ 425.1384; found: 425.1387.

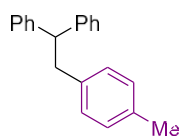


benzyl 4-(4-(((7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methyl)sulfonyl)oxy)phenoxy)benzoate (**80**)

This reaction was conducted on a 0.2 mmol scale with the **General procedure II**. The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 5/1) to afford the title compound as a Colorless oil (98.3 mg, 92% yield). The identity of the product was confirmed by ¹H NMR, ¹³C NMR and HRMS. **¹H NMR (400 MHz, CDCl₃)** δ 8.07 (d, *J* = 8.8 Hz, 2H), 7.47 – 7.42 (m, 2H), 7.41 – 7.31 (m, 5H), 7.08 (d, *J* = 9.0 Hz, 2H), 7.01 (d, *J* = 8.8 Hz, 2H), 5.35 (s, 2H), 3.84 (d, *J* = 15.0 Hz, 1H), 3.21 (d, *J* = 15.0 Hz, 1H), 2.62 – 2.50 (m, 1H), 2.49 – 2.39 (m, 1H), 2.17 – 2.06 (m, 2H), 1.99 (d, *J* = 18.6 Hz, 1H), 1.78 – 1.70 (m, 1H), 1.50 – 1.42 (m, 1H), 1.17 (s, 3H), 0.92 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 214.2, 165.8, 161.2, 154.5, 145.2, 136.1, 132.0, 128.6, 128.3, 128.2, 125.1, 123.8, 121.0, 117.7, 66.7, 58.2, 48.0, 47.6, 42.8, 42.5, 26.9, 25.1, 20.0, 19.8.

HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd. for C₃₀H₃₀O₇Na 557.1604; found: 557.1607.

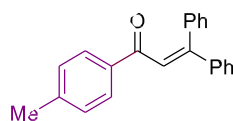


(2-(*p*-tolyl)ethane-1,1-diyl)dibenzene (**82**)

The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 50/1) to afford the title compound as a Colorless oil (DBU as a promoter, 18.5 mg, 34% yield), (DMAP as a promoter, 27.8 mg, 51% yield)

¹H NMR (400 MHz, CDCl₃) δ 7.26 – 7.19 (m, 8H), 7.17 – 7.11 (m, 2H), 6.97 (d, *J* = 7.8 Hz, 2H), 6.89 (d, *J* = 8.0 Hz, 2H), 4.21 (t, *J* = 7.8 Hz, 1H), 3.32 (d, *J* = 7.8 Hz, 2H), 2.25 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 144.6, 137.2, 135.3, 128.9, 128.8, 128.3, 128.1, 126.2, 53.1, 41.7, 21.0.



3,3-diphenyl-1-(*p*-tolyl)prop-2-en-1-one (**83**)

The crude product was purified by silica gel chromatography (PE/EA = 1/0 to 20/1) to afford the title compound as a Colorless oil (DBU as a promoter, 13.7 mg, 23% yield), (DMAP as a promoter, 10.7 mg, 18% yield)

¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, *J* = 8.2 Hz, 2H), 7.38 (s, 4H), 7.30 – 7.25 (m, 4H), 7.20 – 7.16 (m, 4H), 7.11 (s, 1H), 2.37 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 192.3, 154.1, 143.5, 141.5, 139.1, 135.7, 129.7, 129.2, 129.1, 128.9, 128.6, 128.4, 128.3, 128.0, 124.3, 21.7.

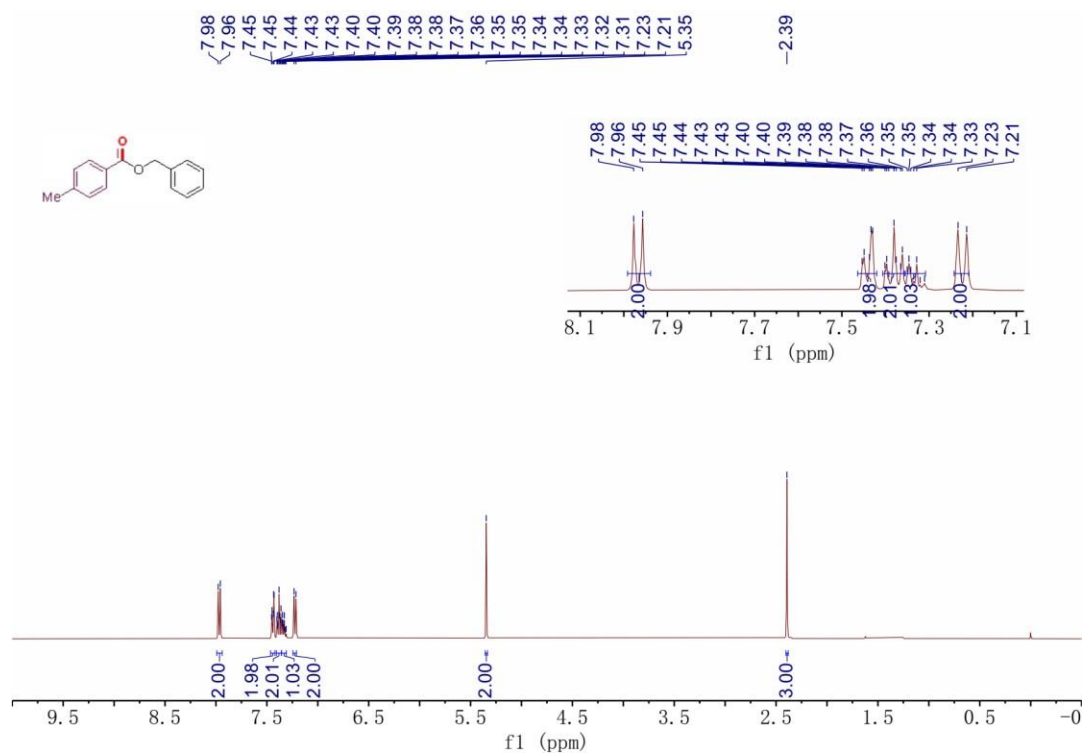
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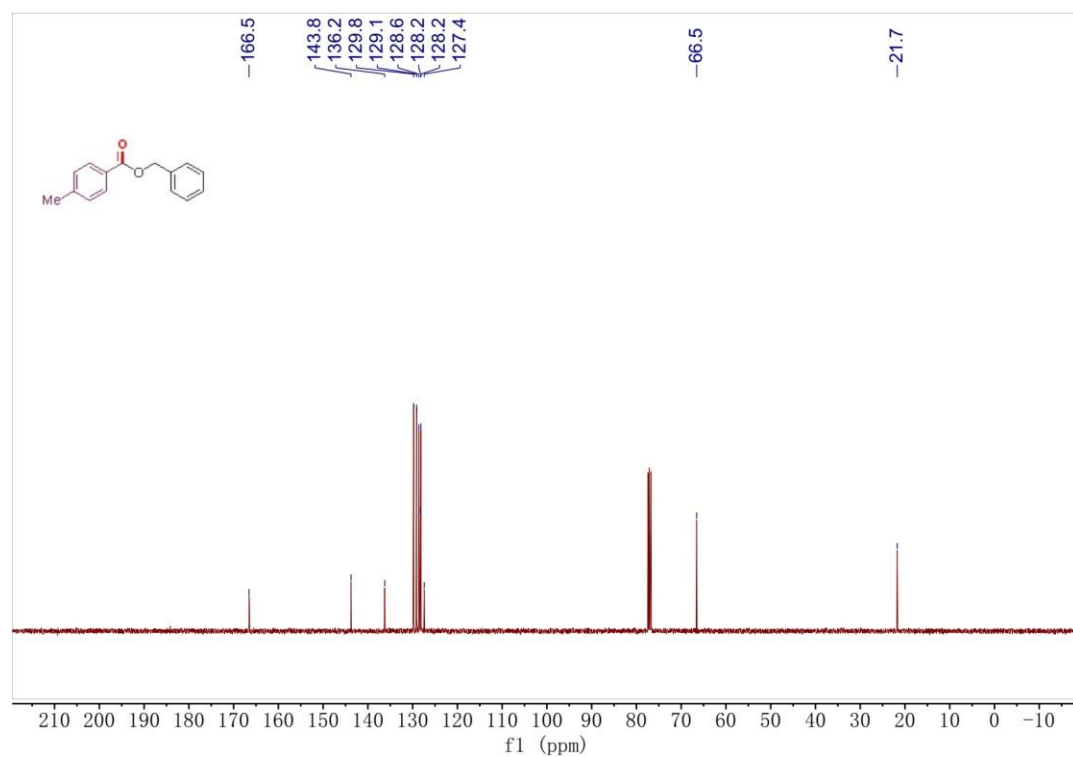
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7. Copy of ^1H and ^{13}C NMR Spectra of Products

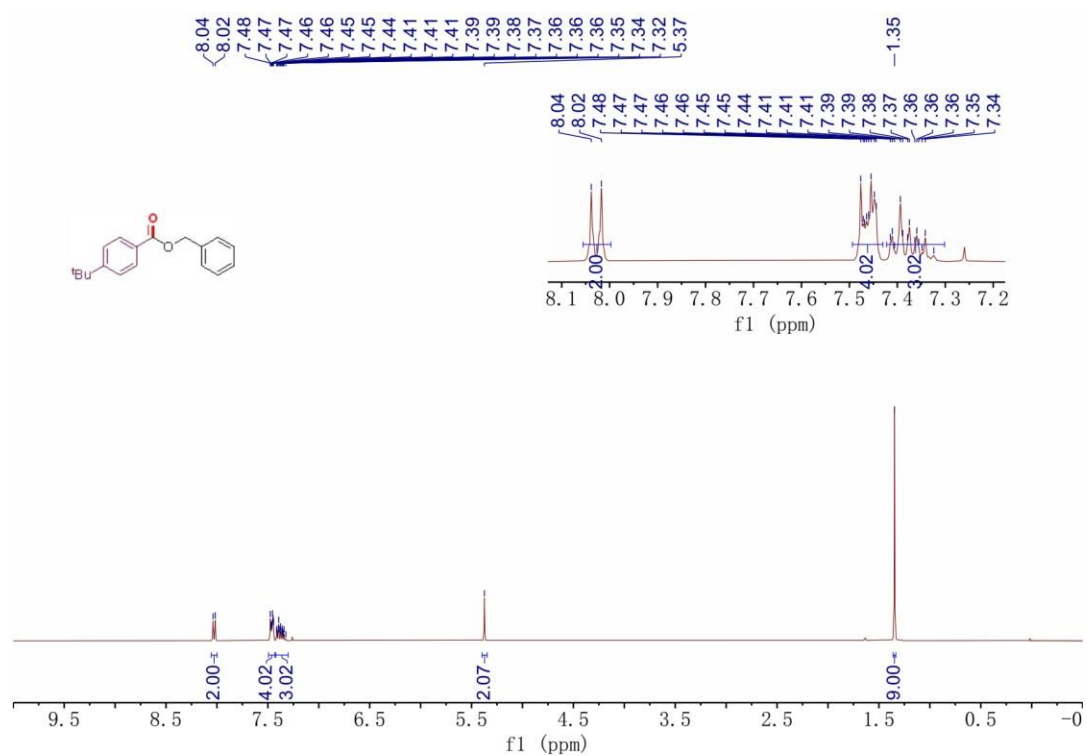
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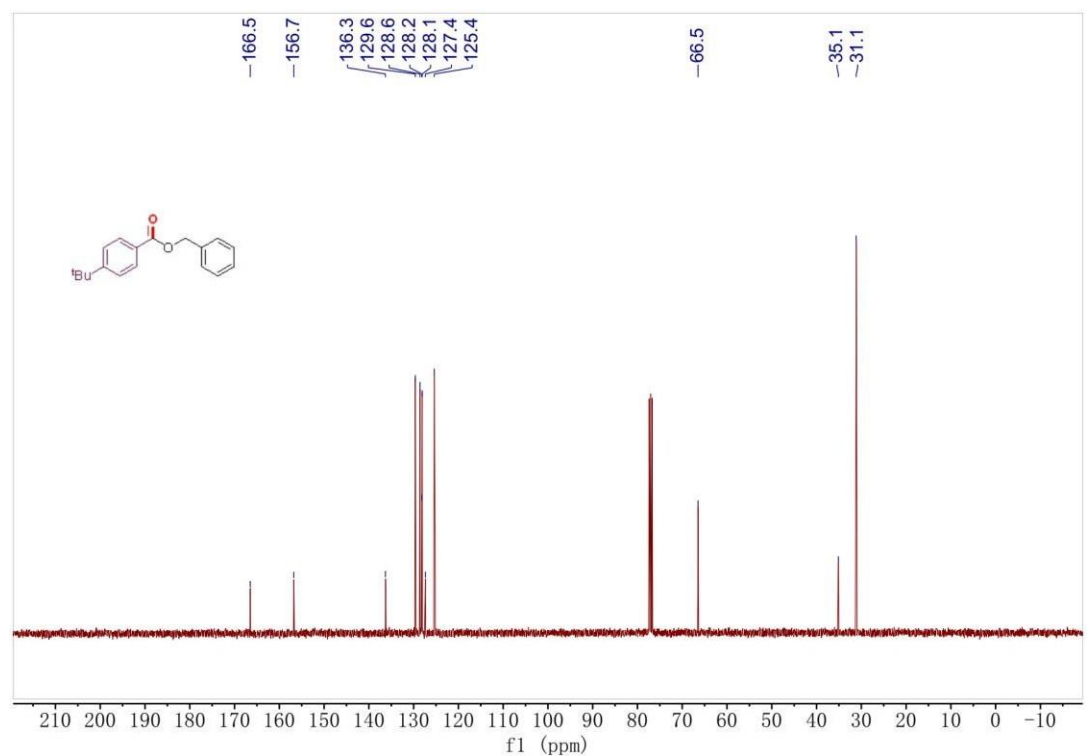
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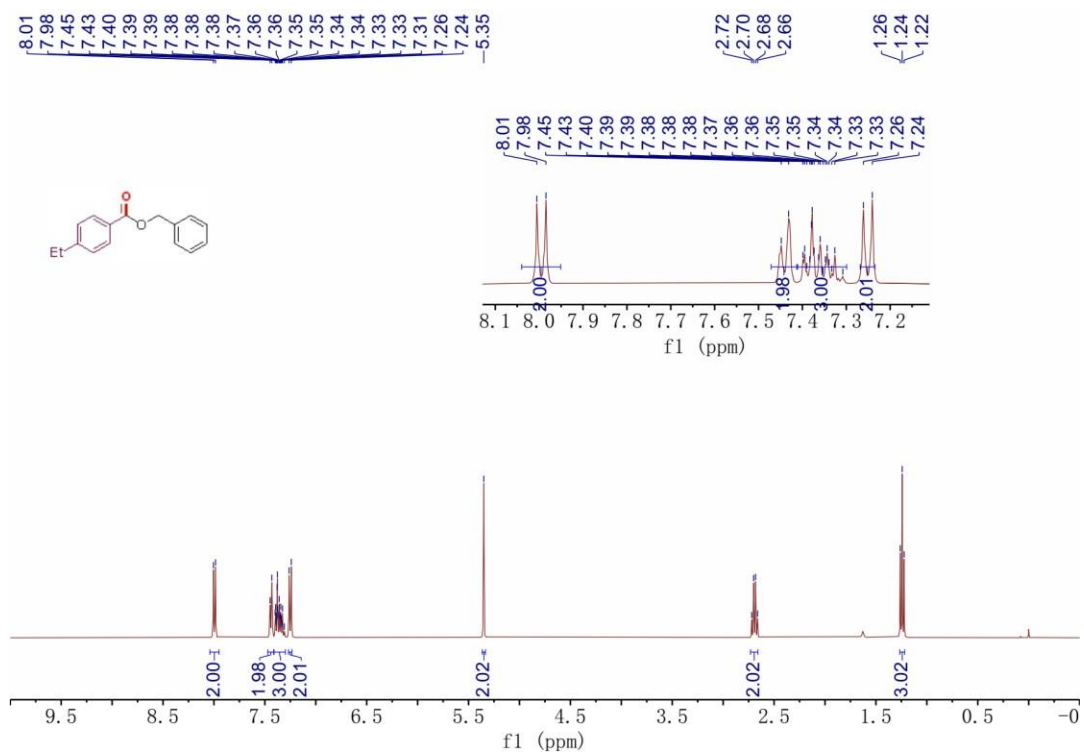
¹H NMR (400 MHz, CDCl₃) 37



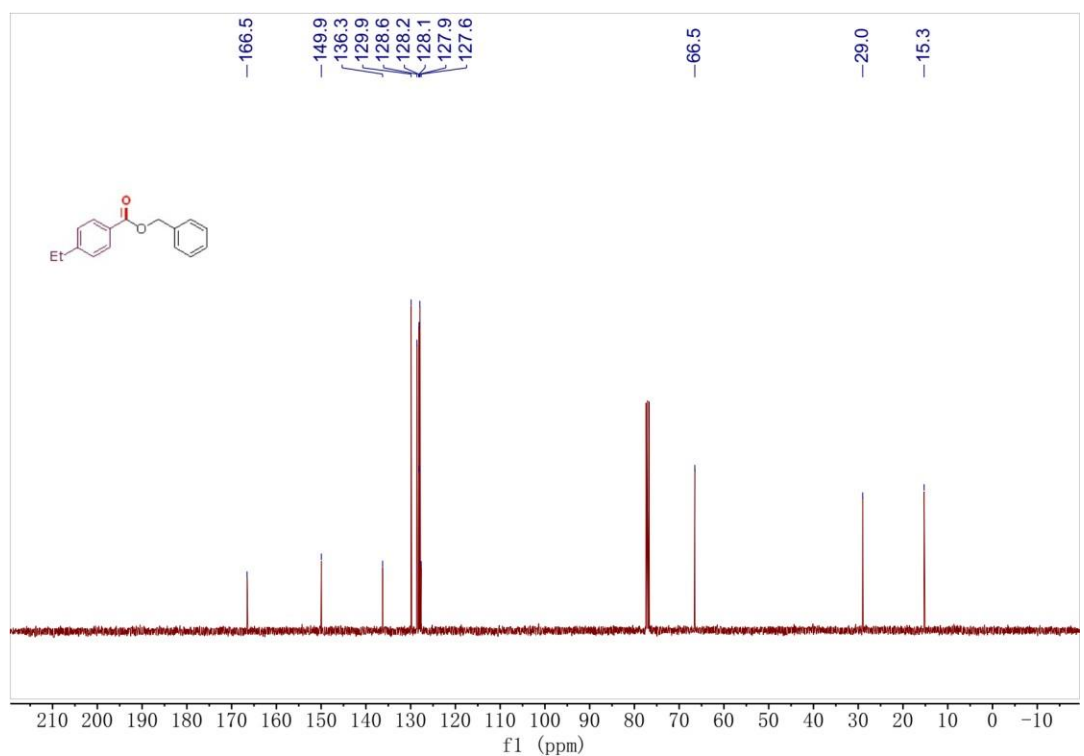
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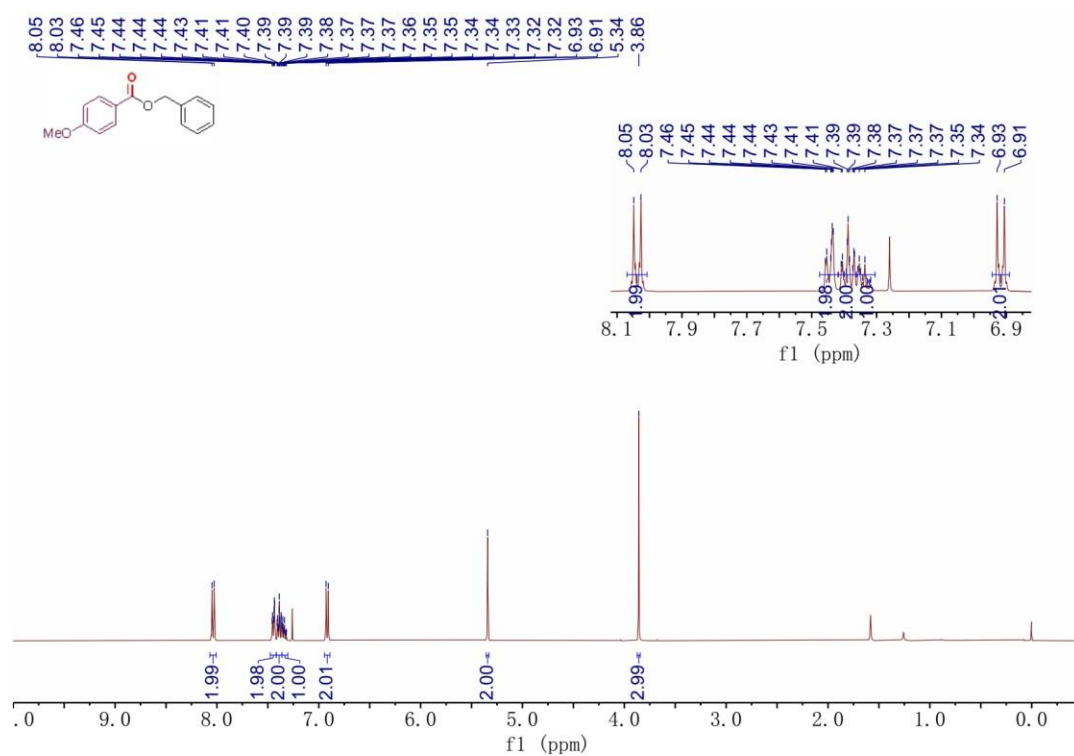
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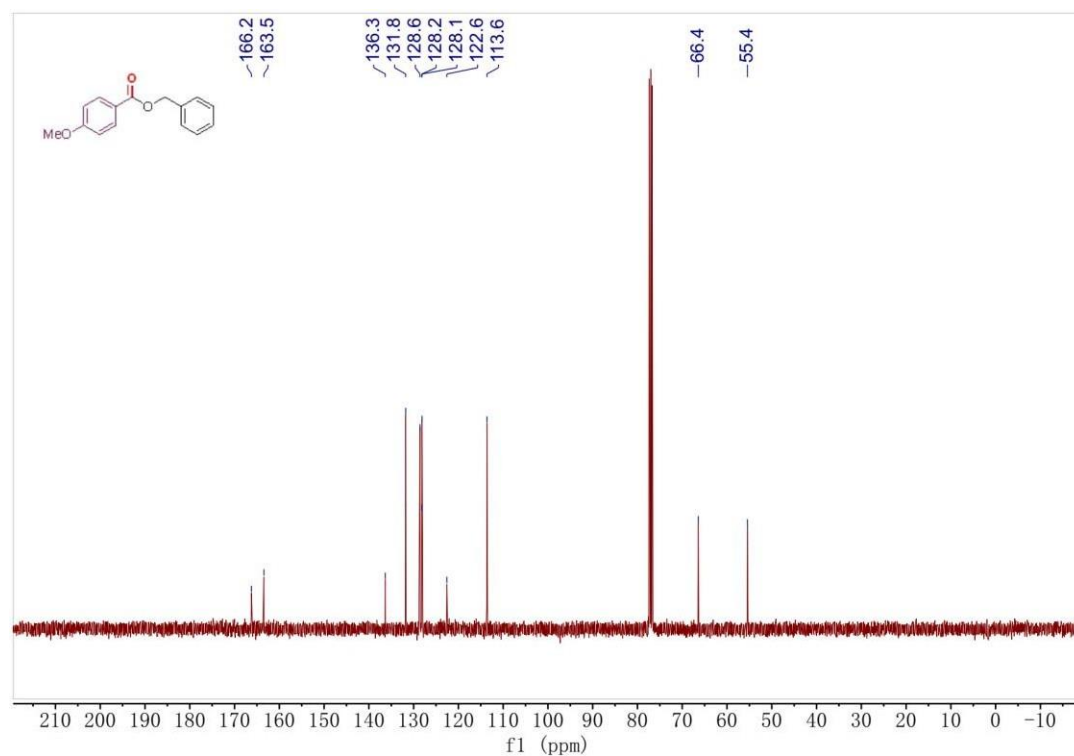
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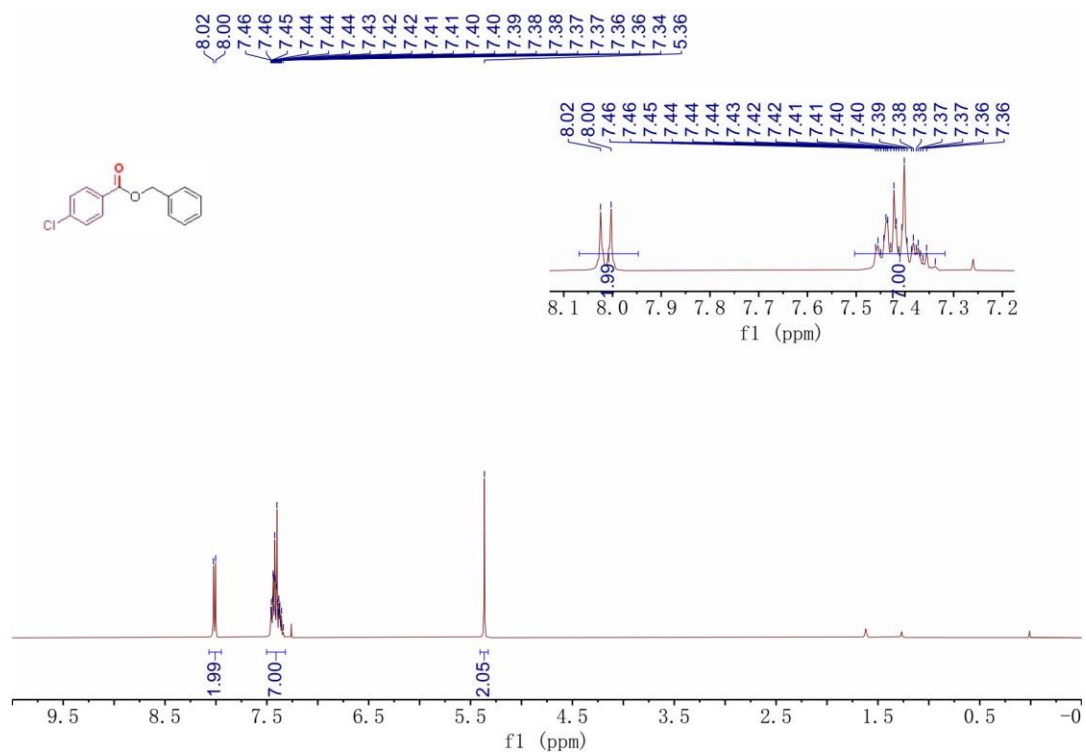
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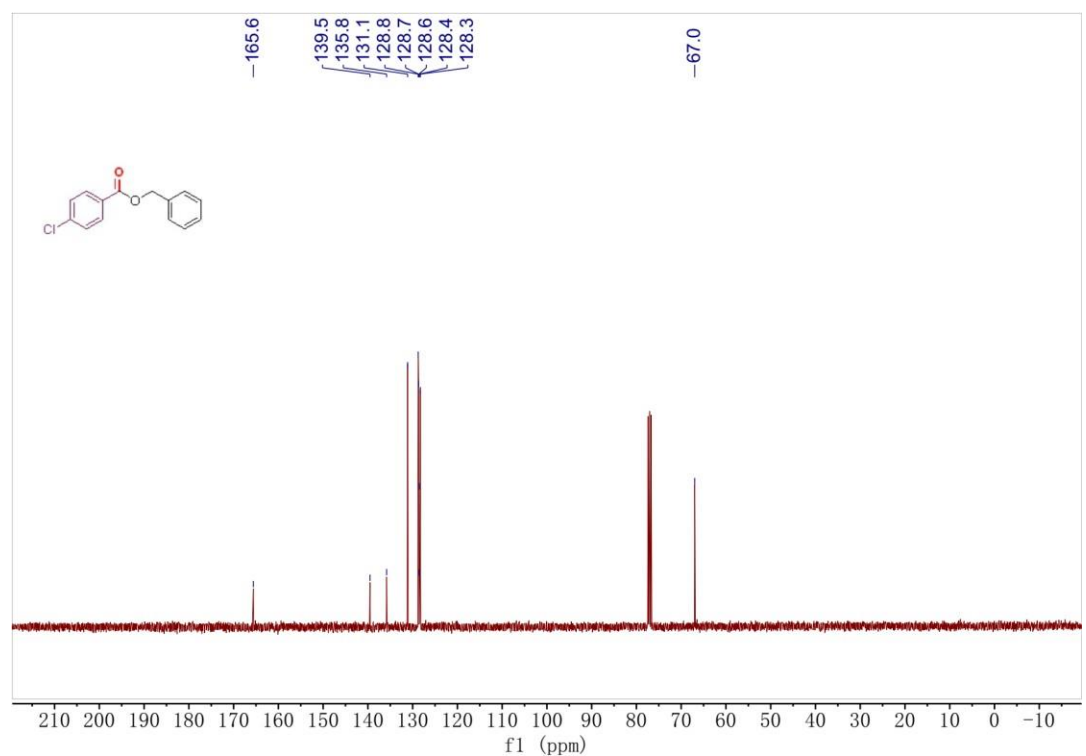
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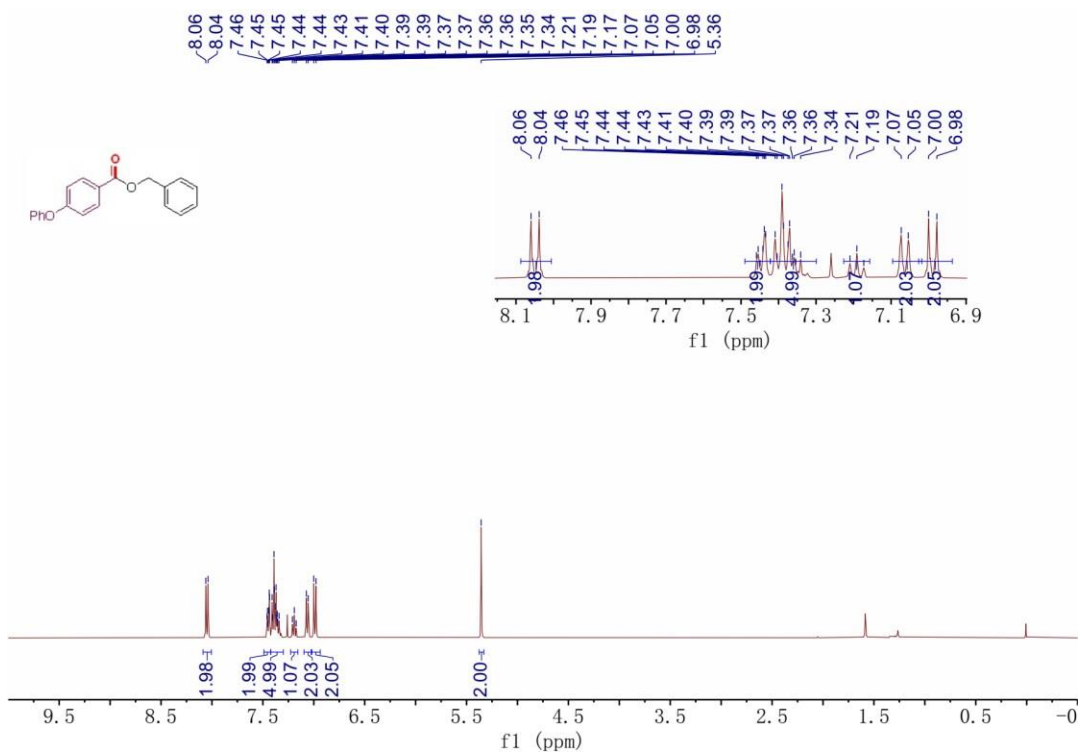
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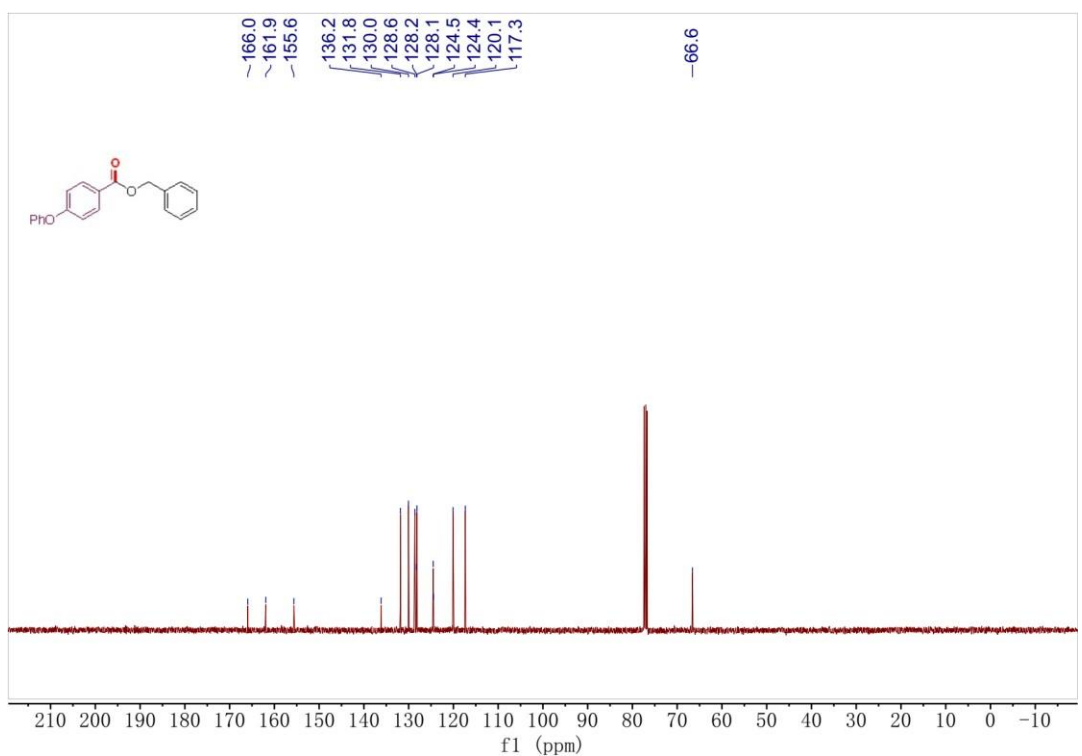
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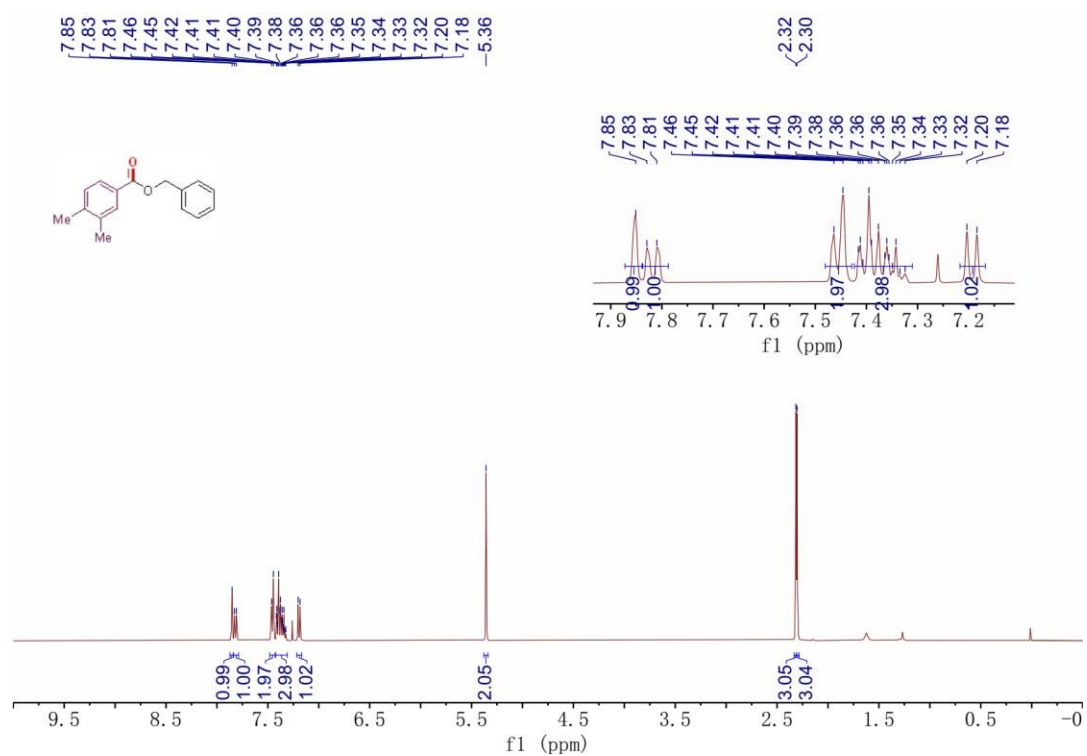
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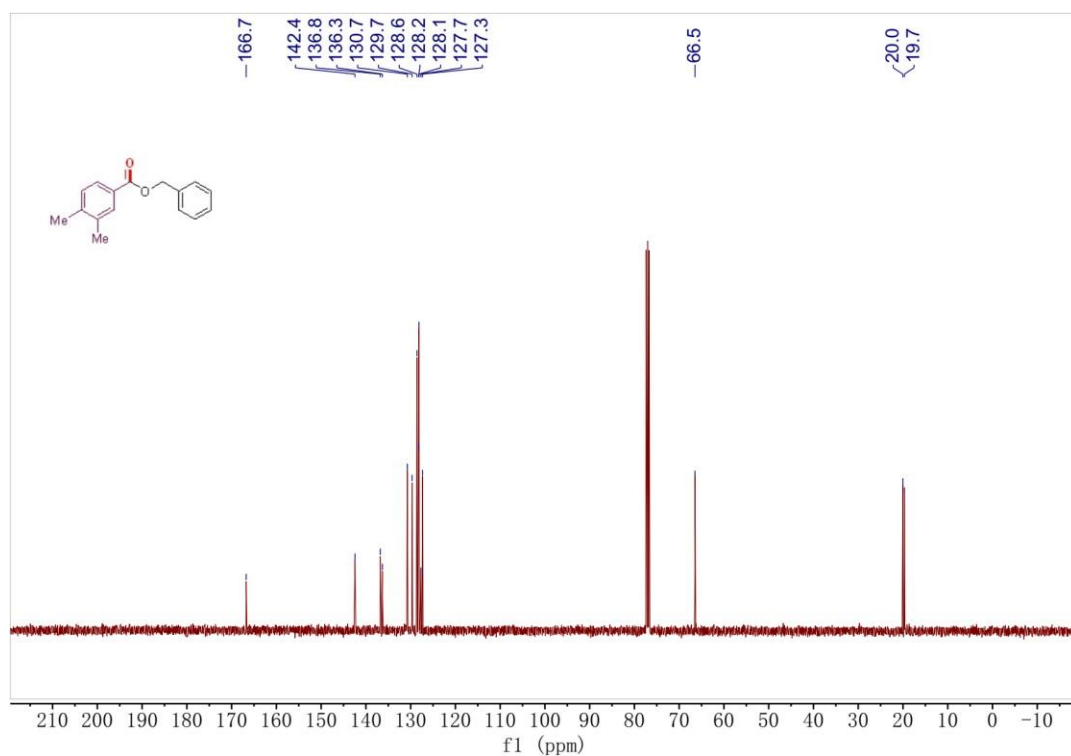
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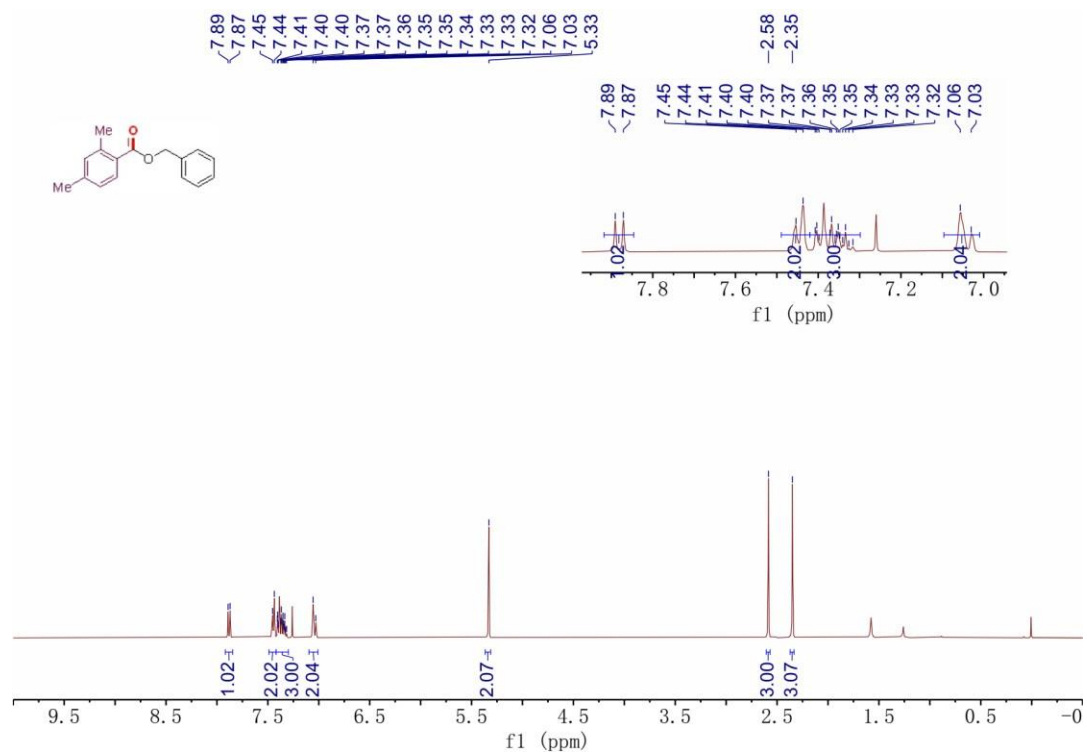
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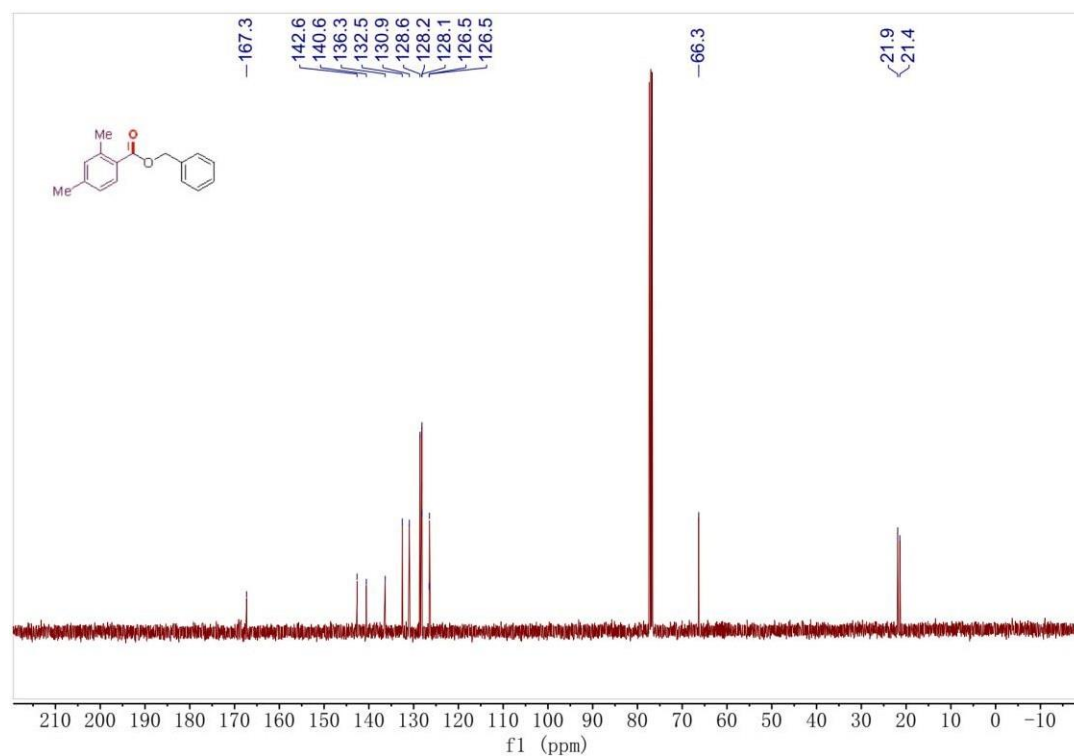
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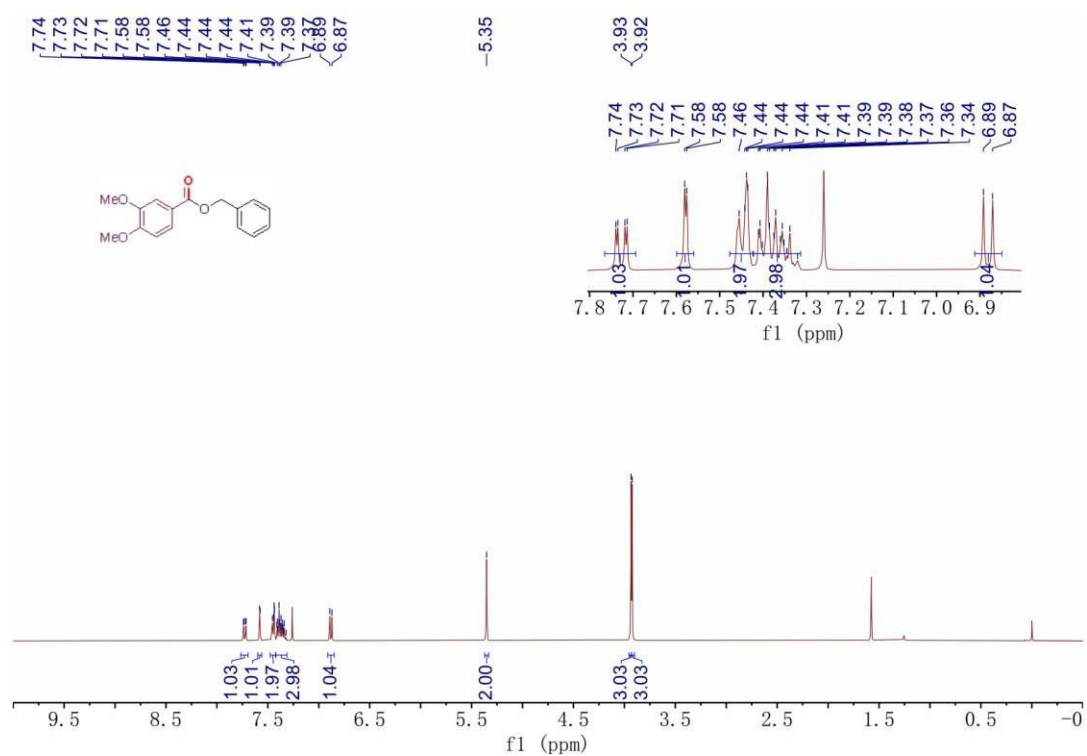
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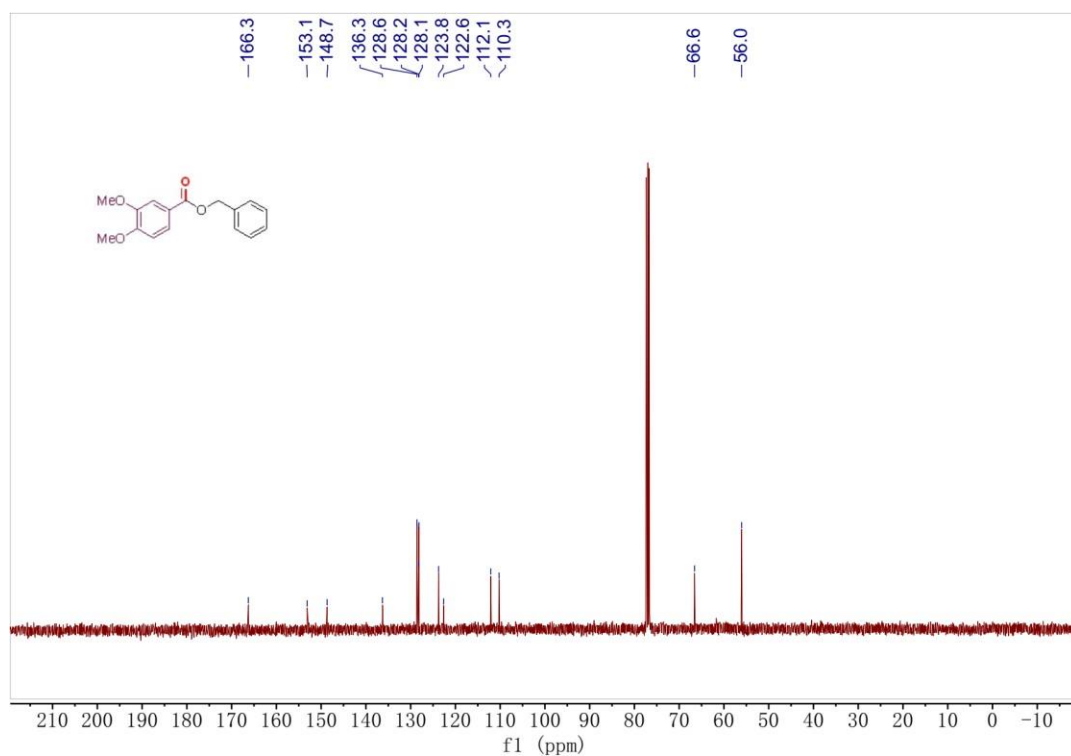
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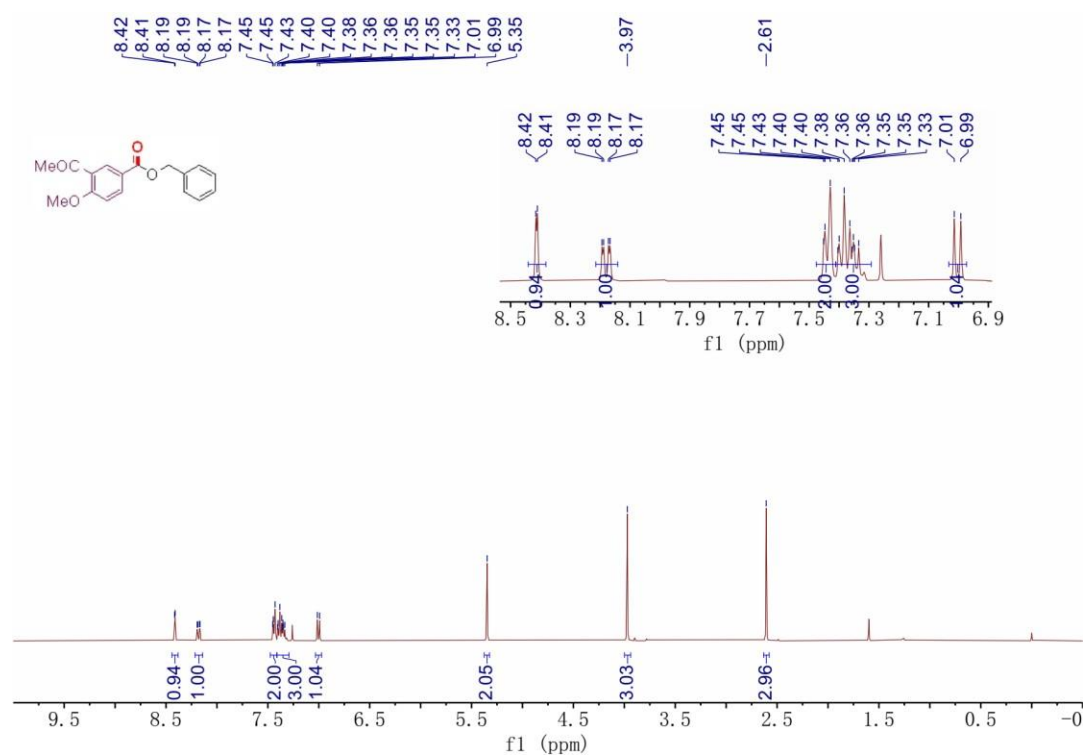
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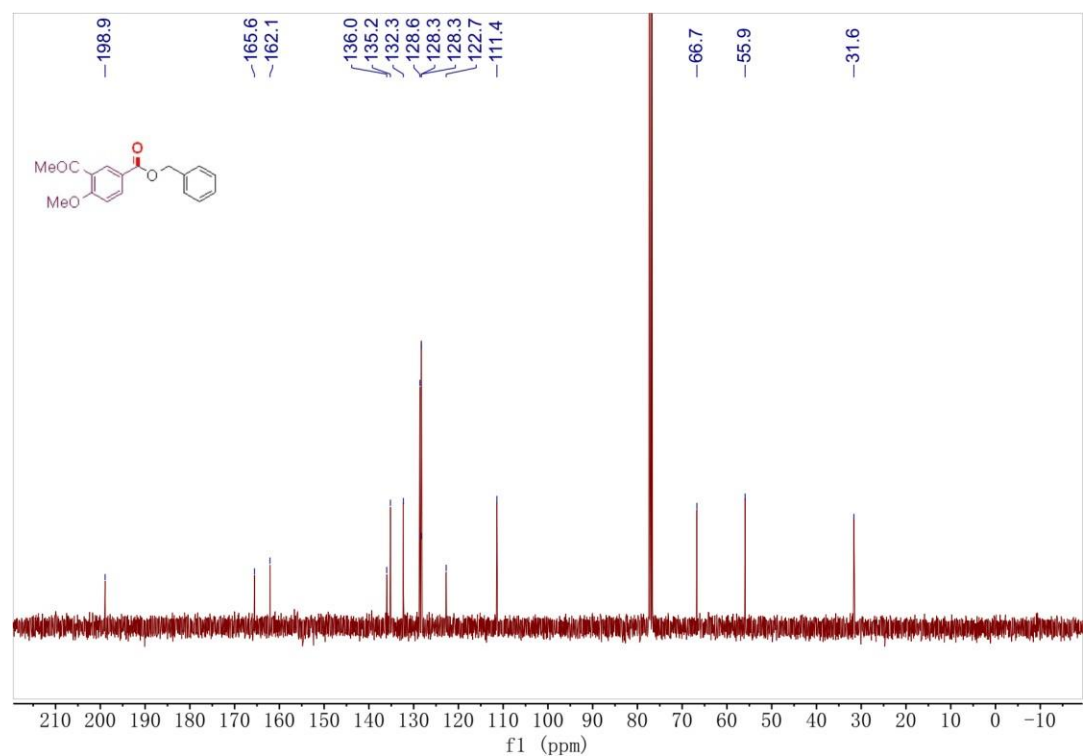
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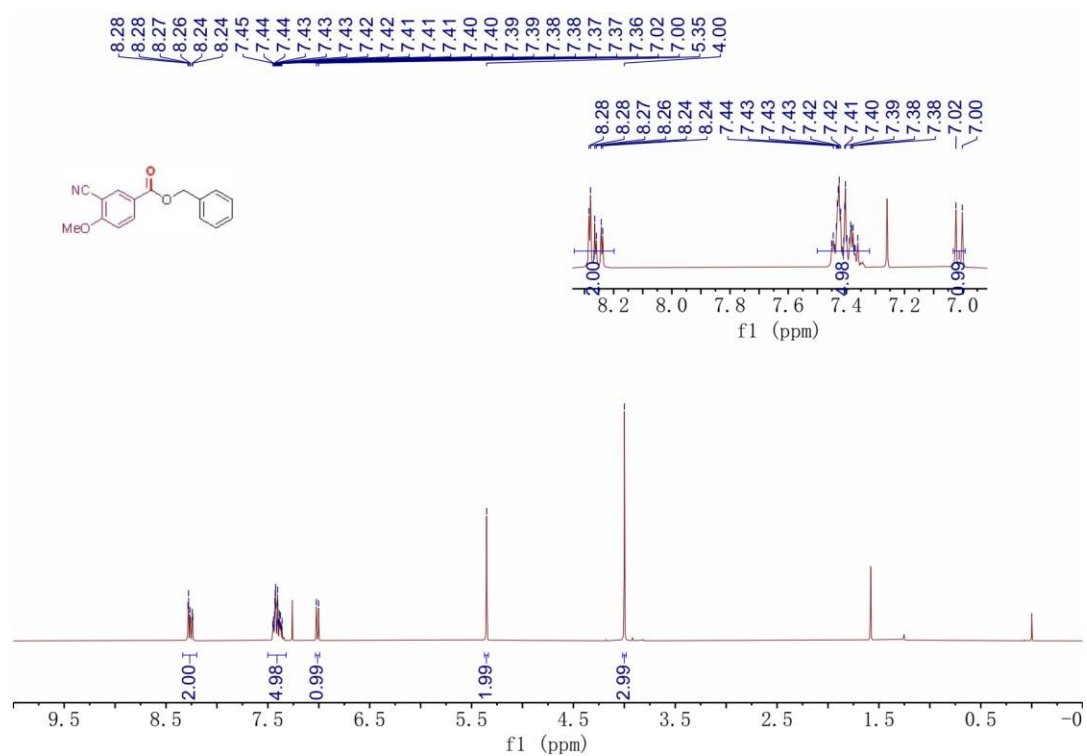
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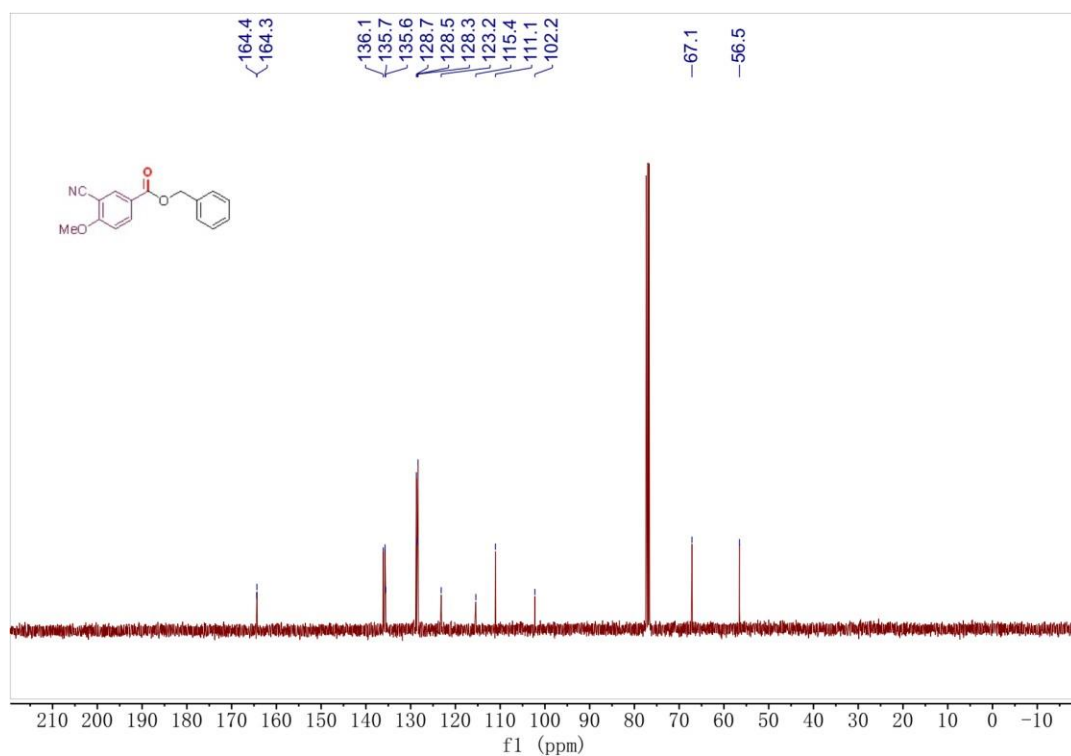
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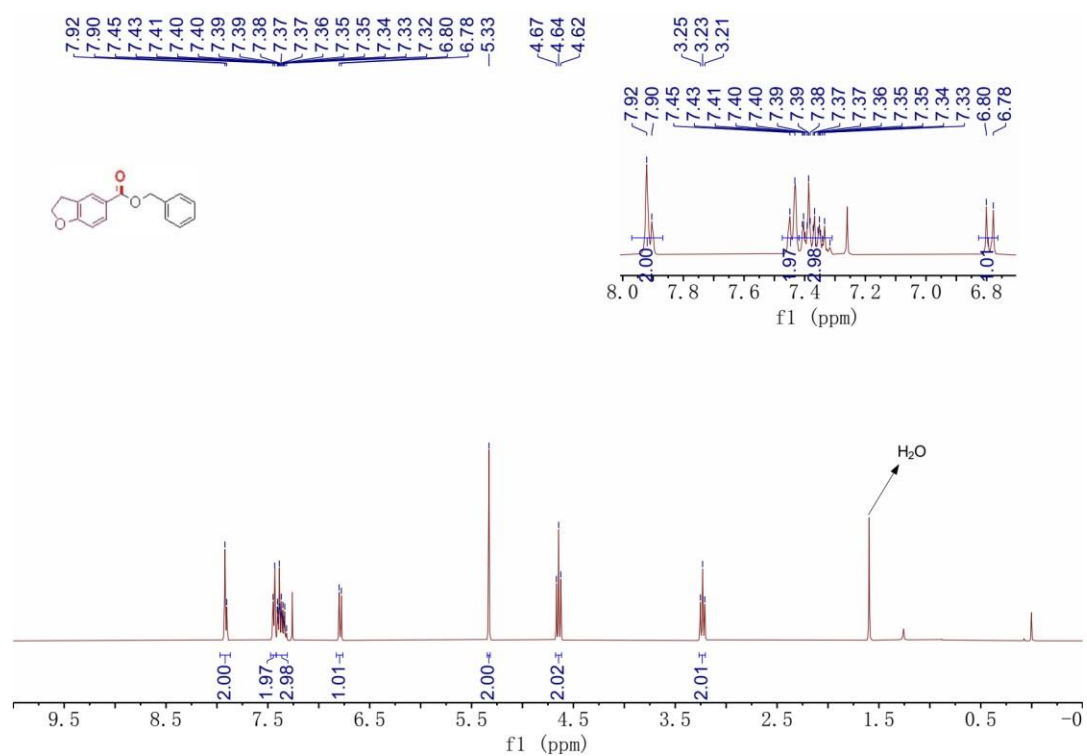
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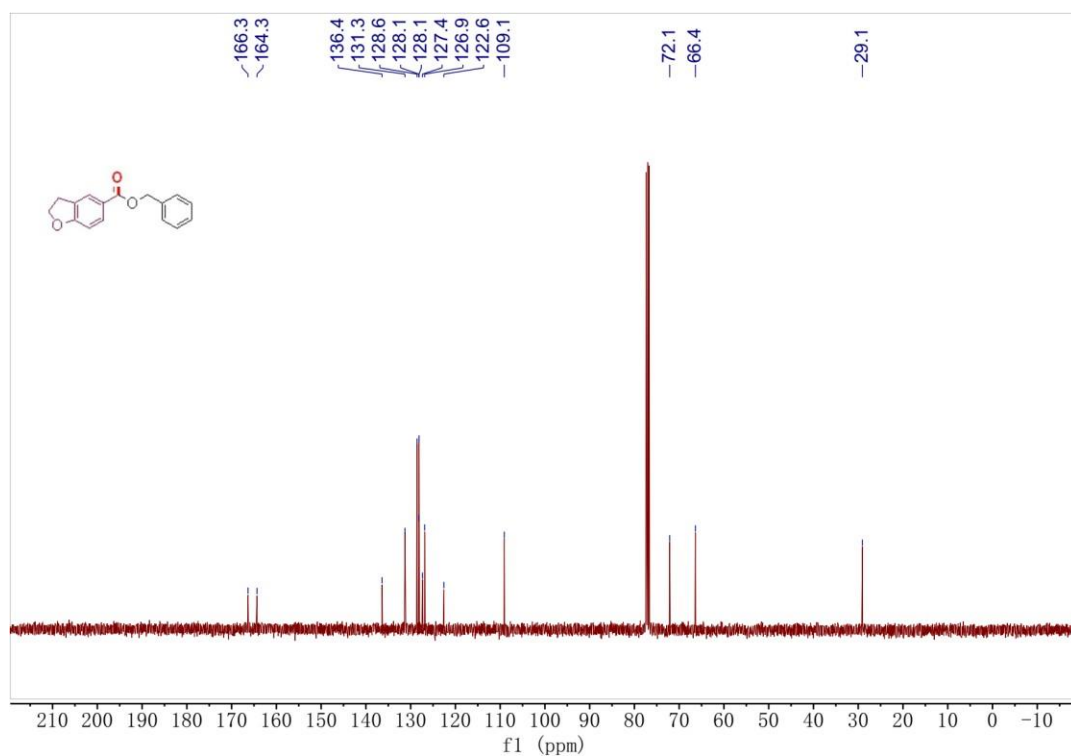
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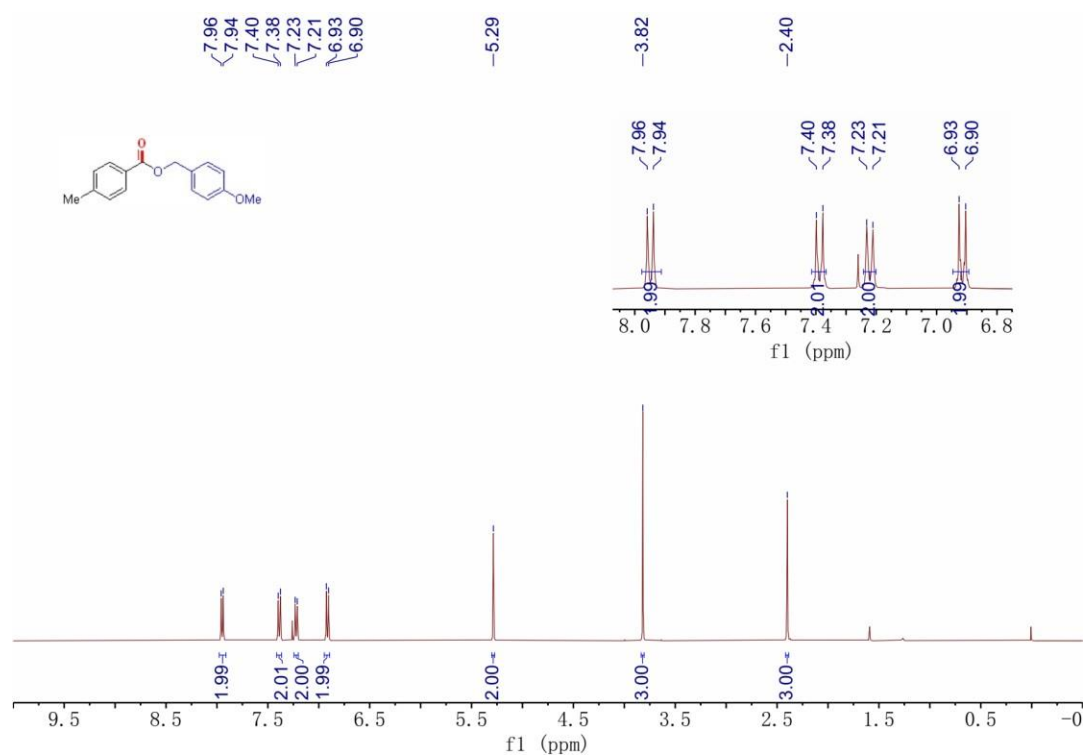
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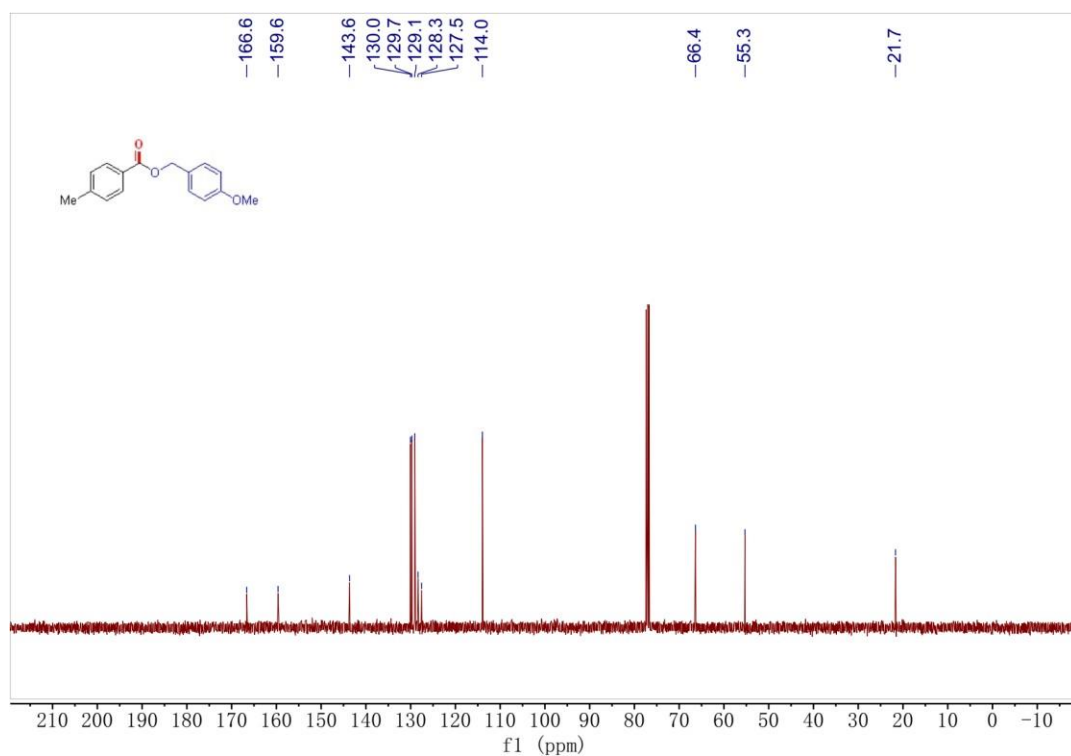
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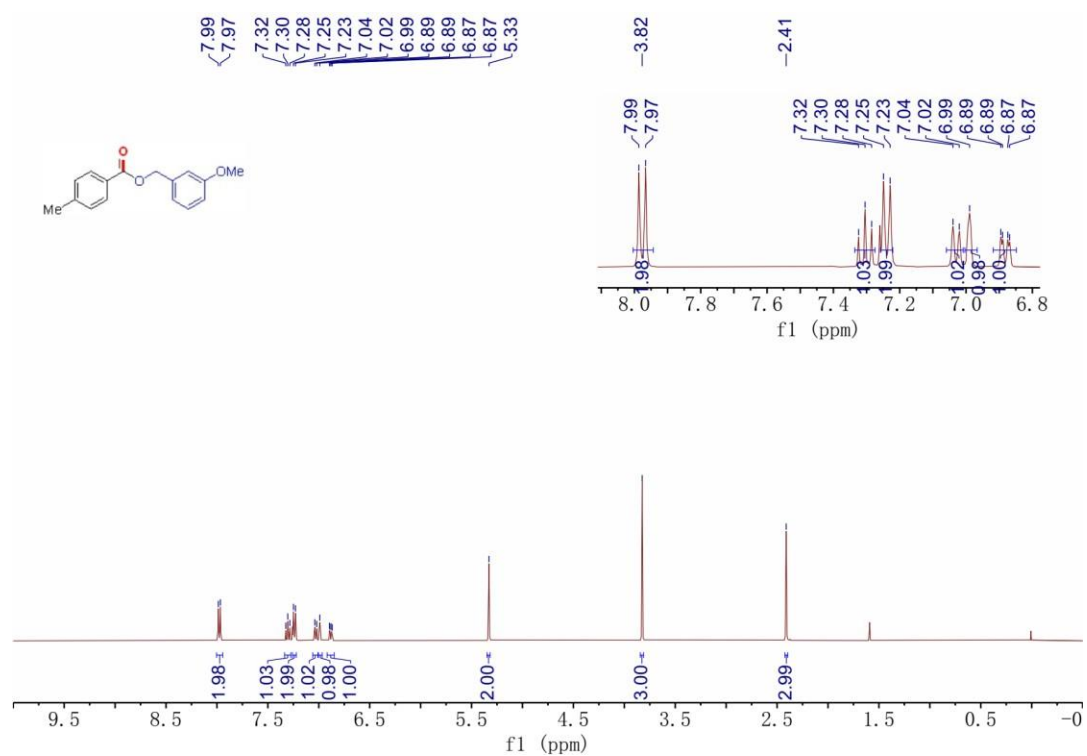
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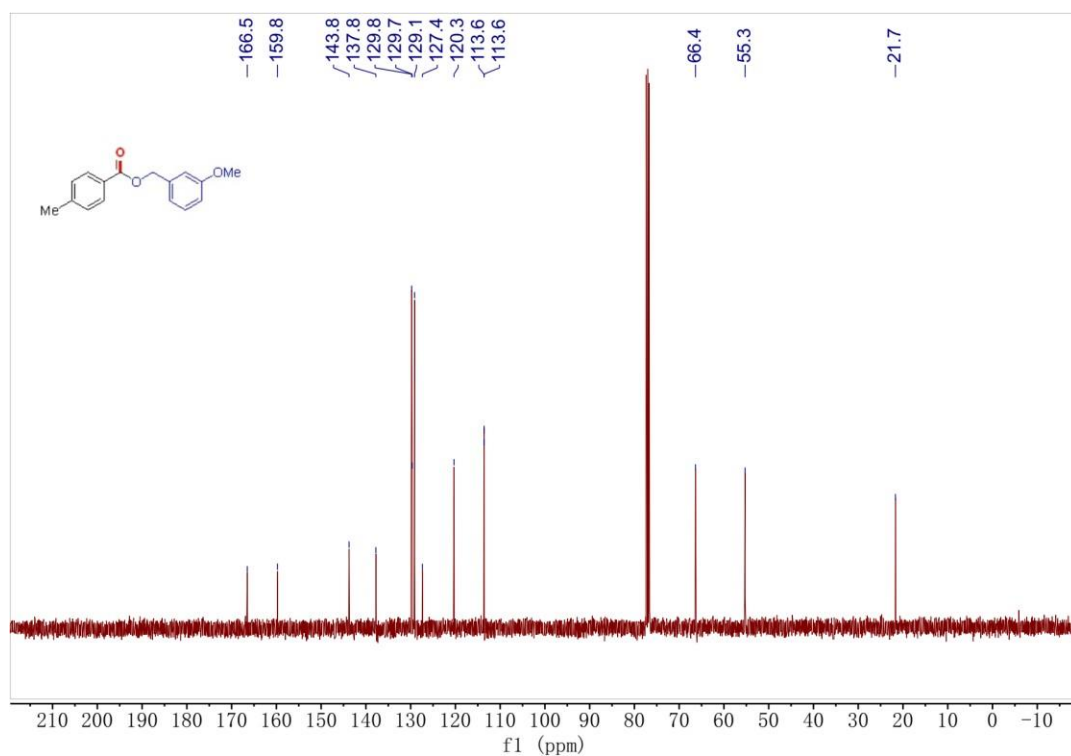
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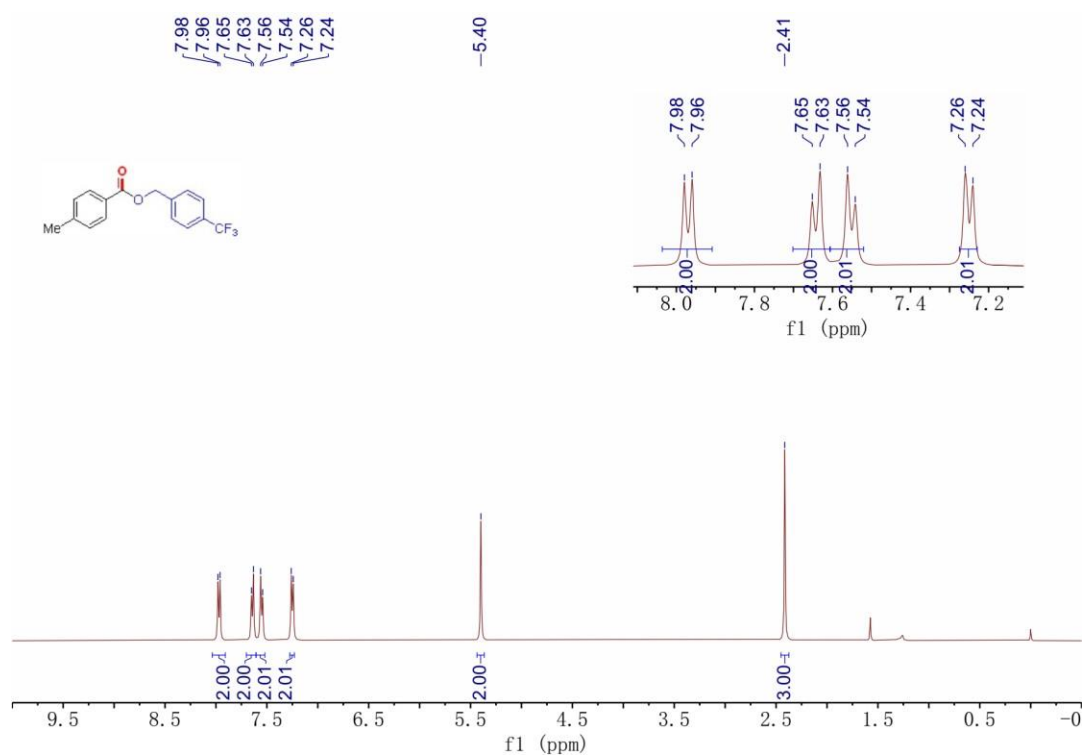
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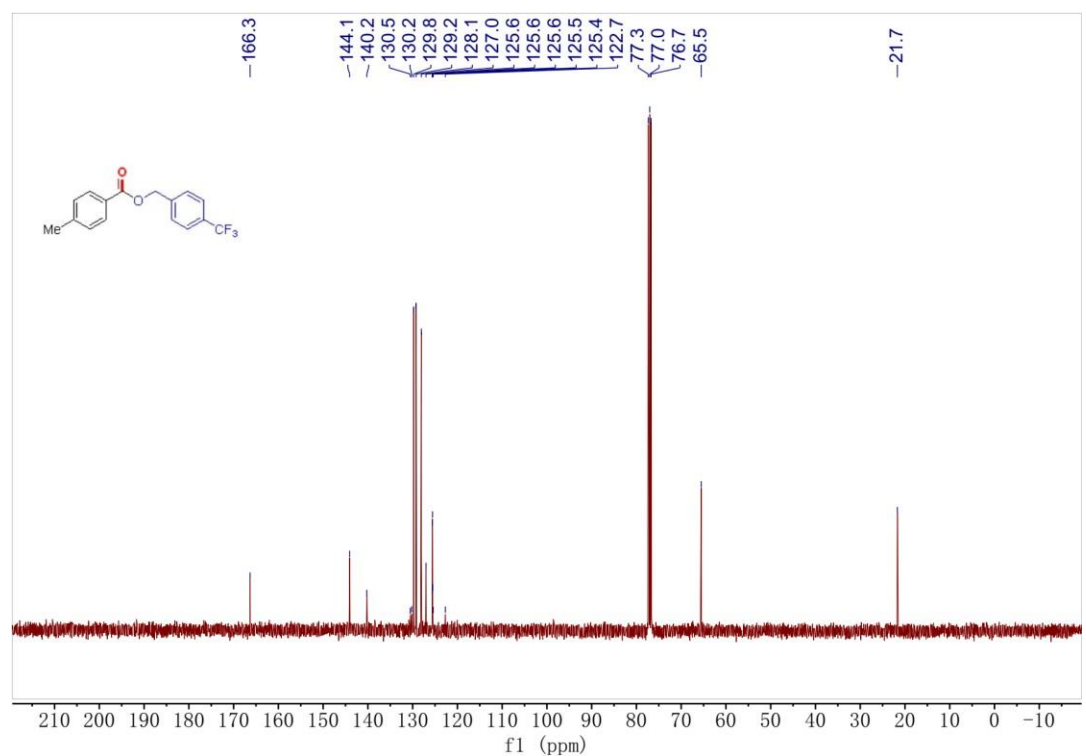
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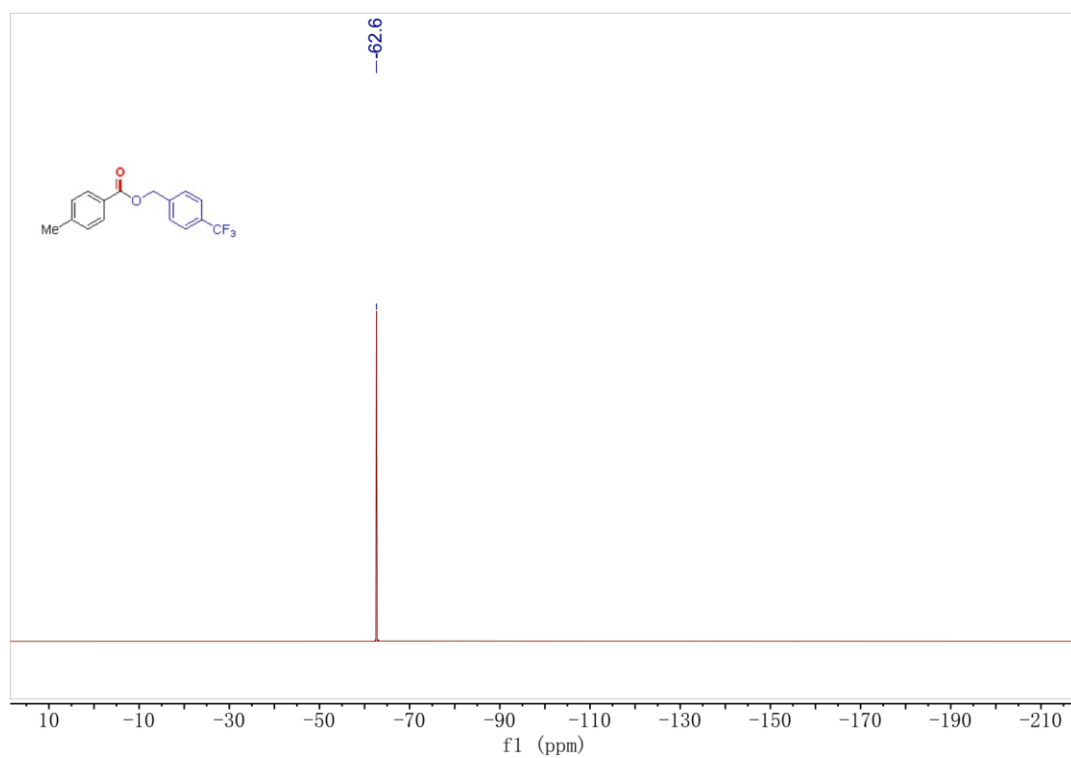
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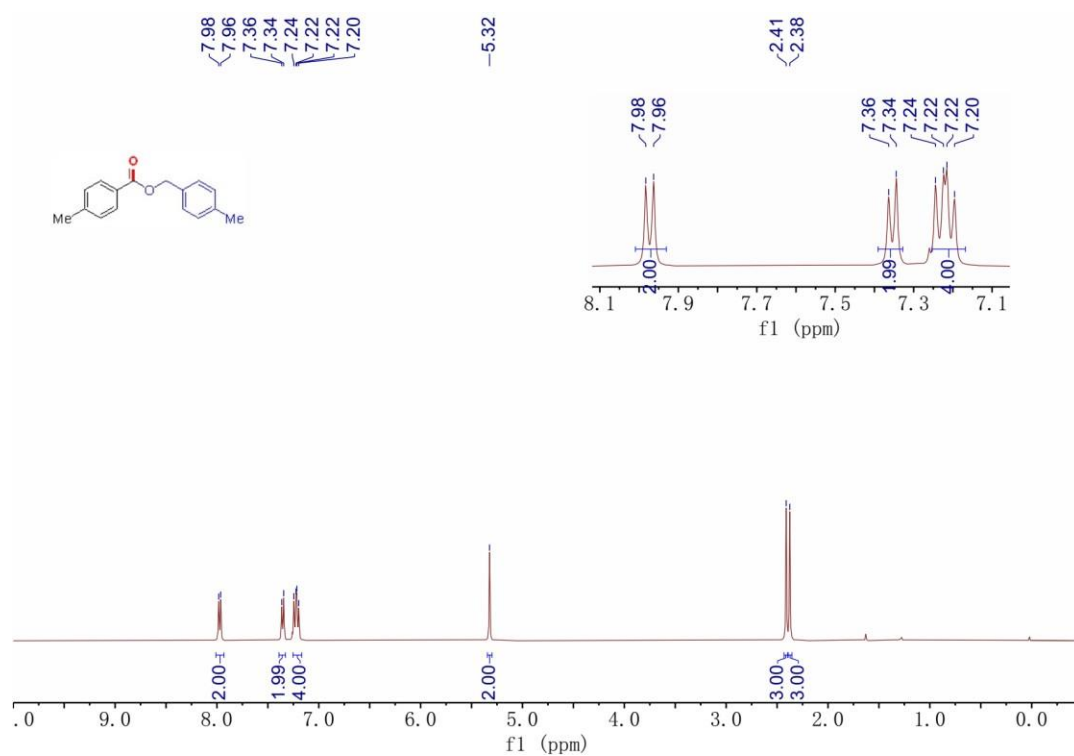
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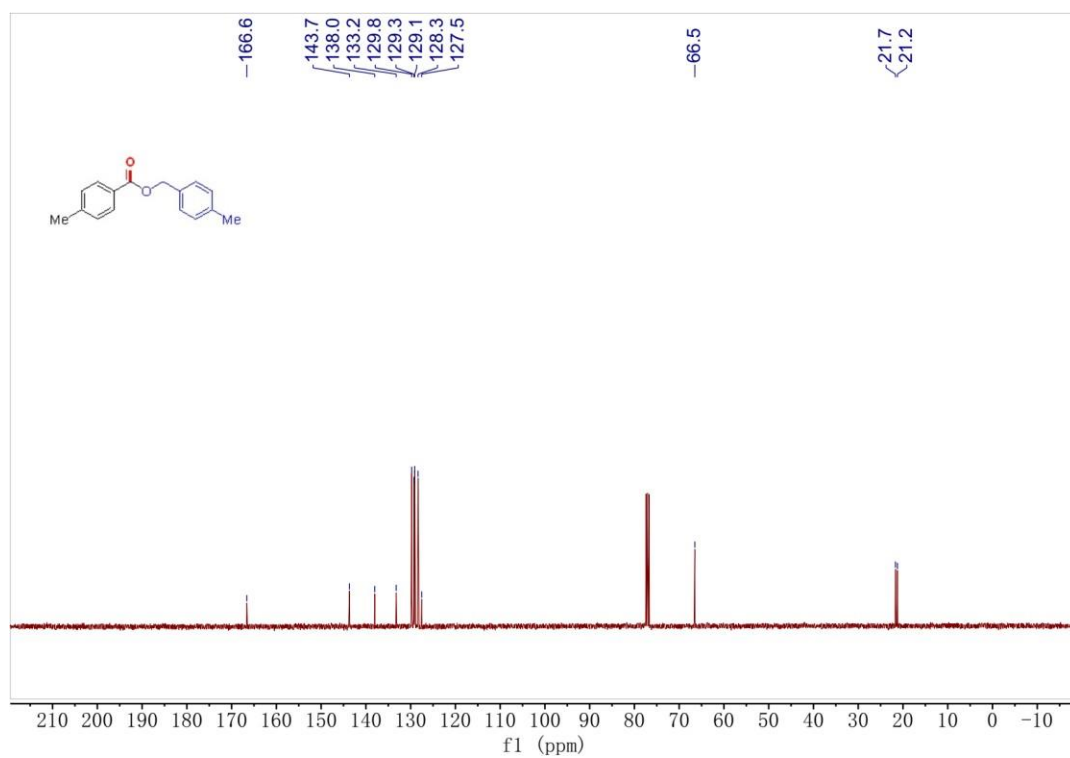
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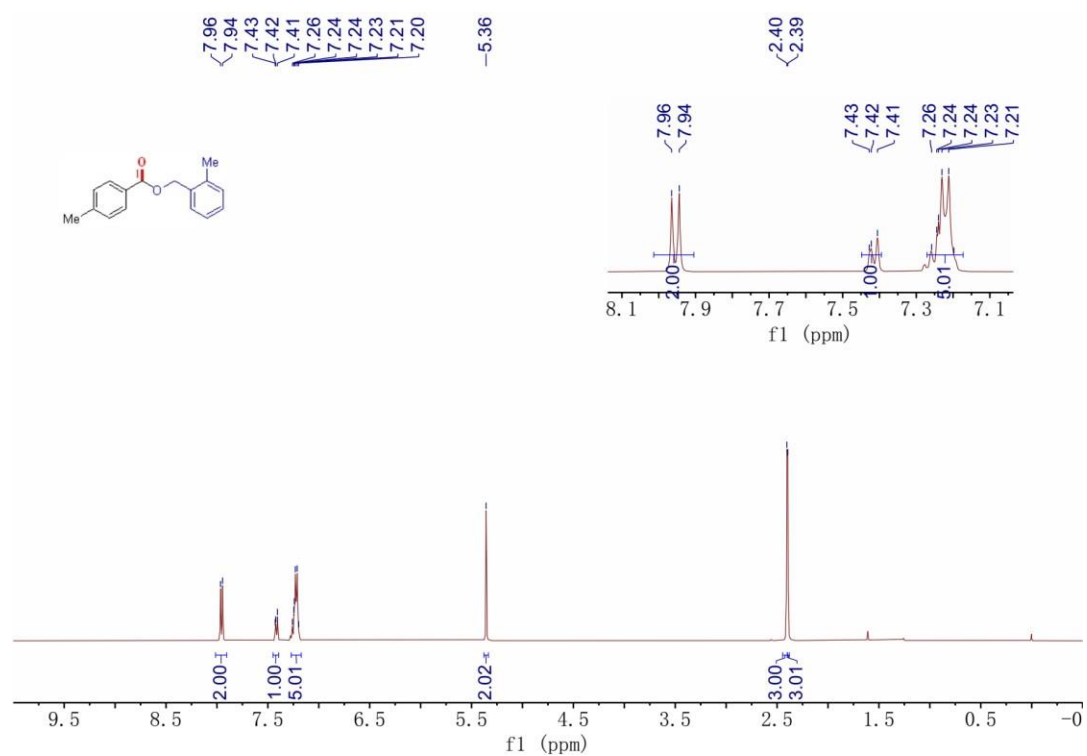
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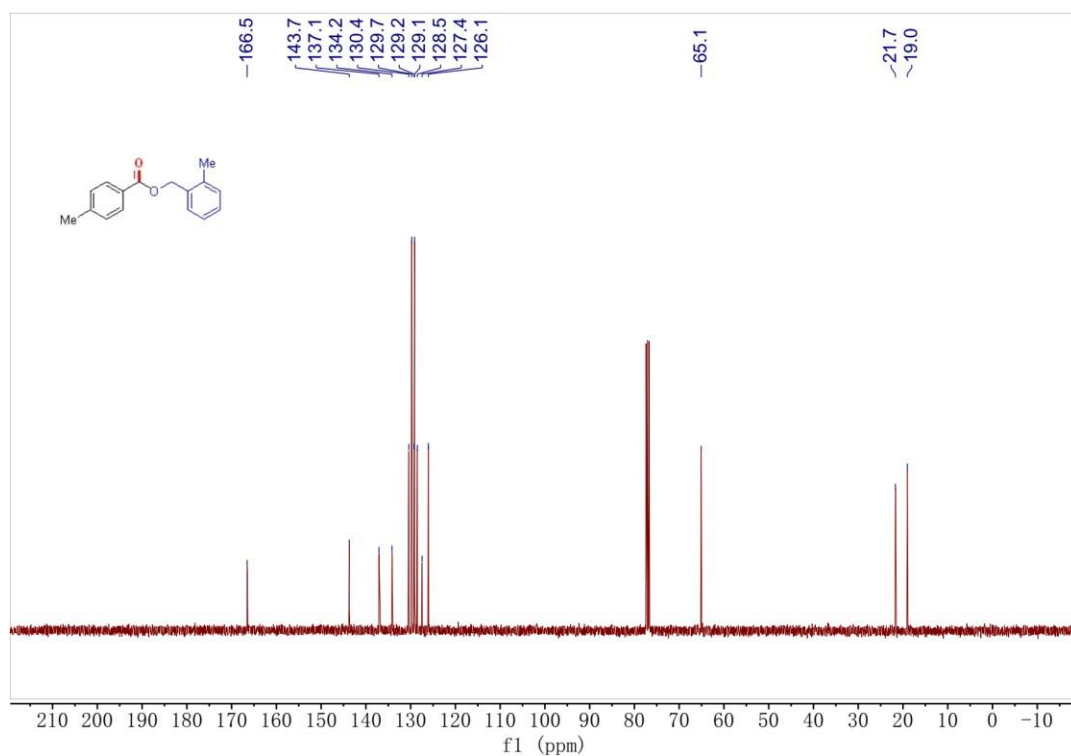
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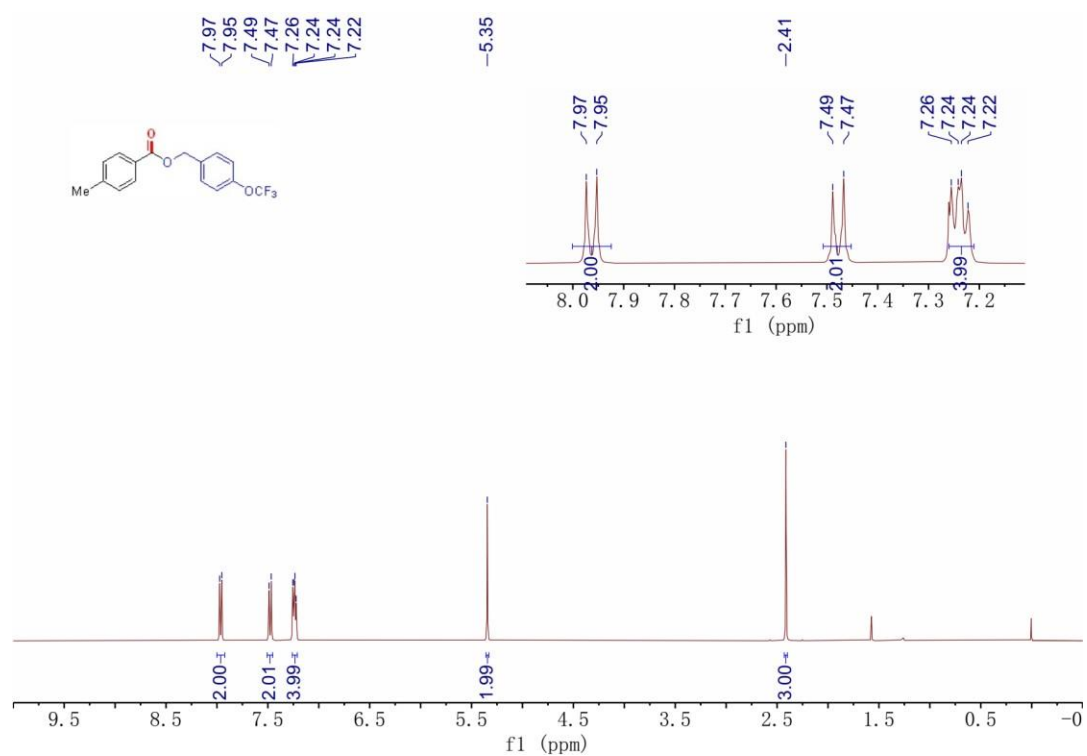
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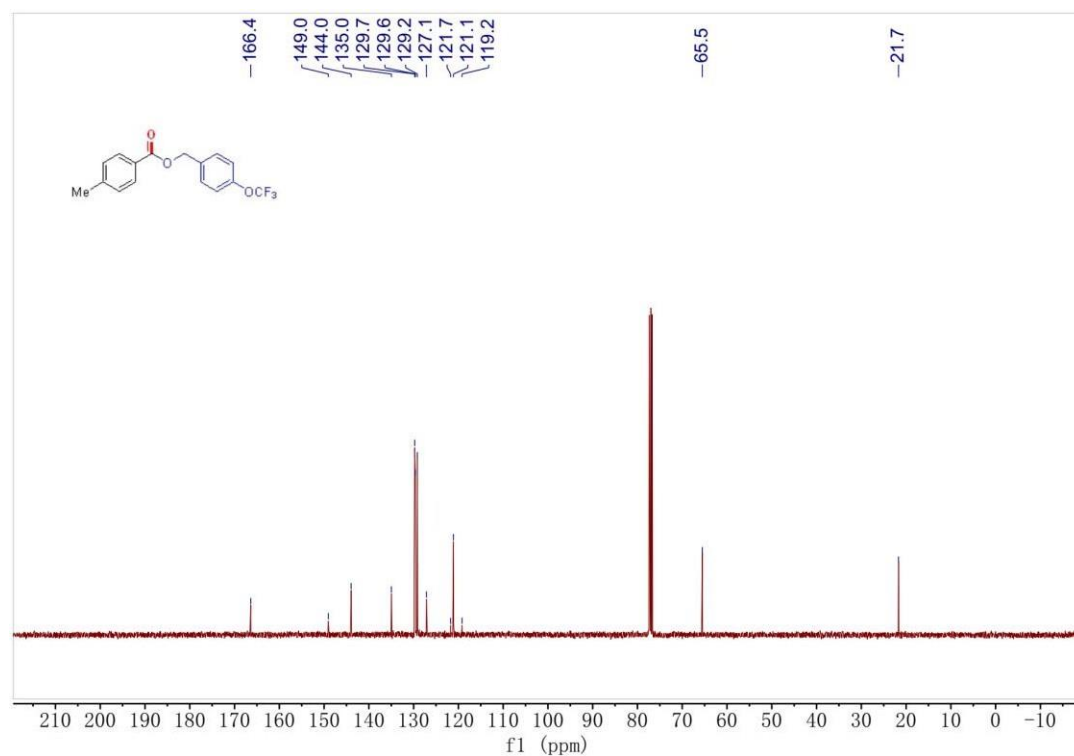
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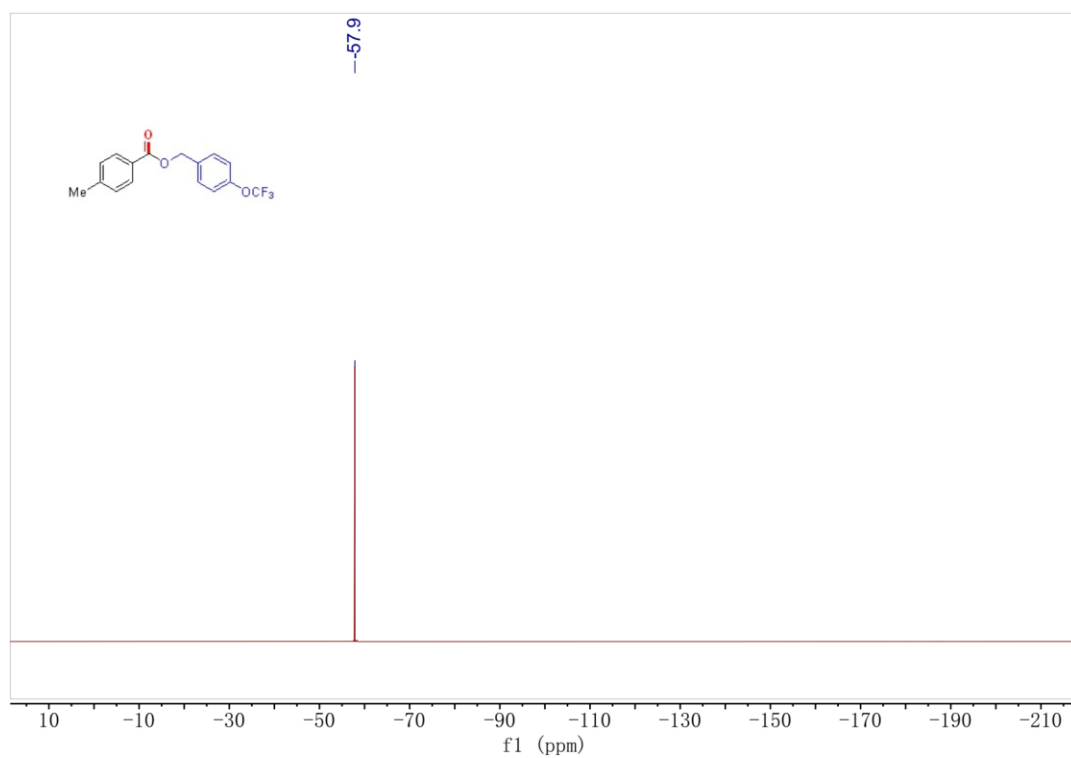
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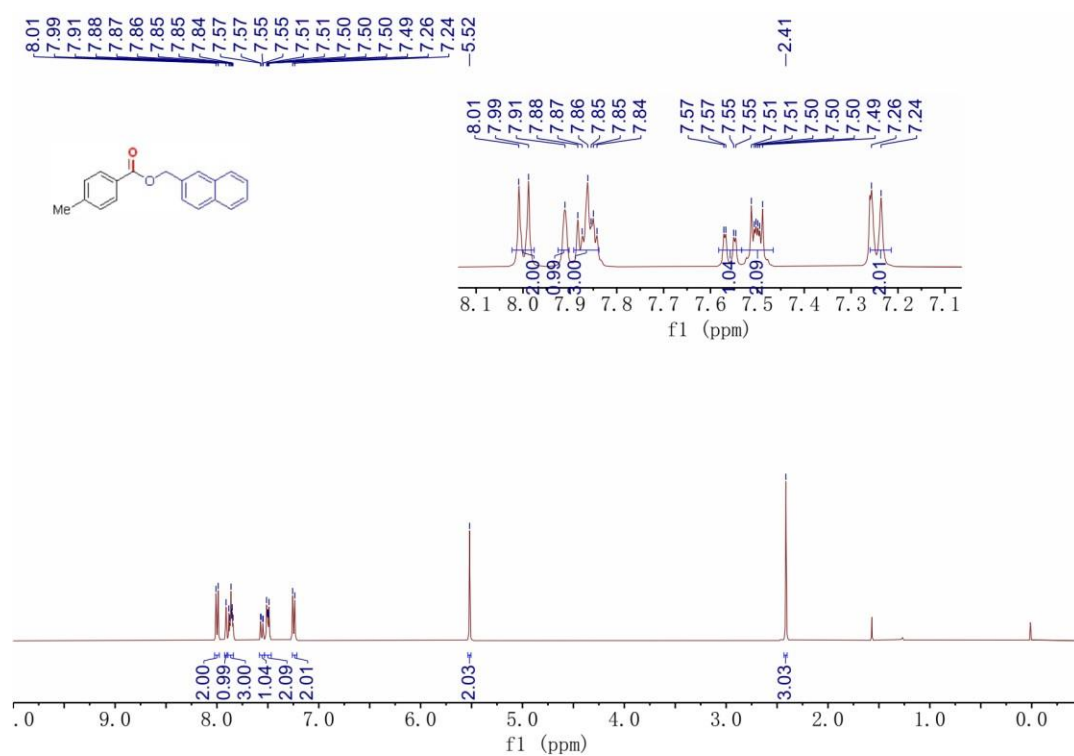
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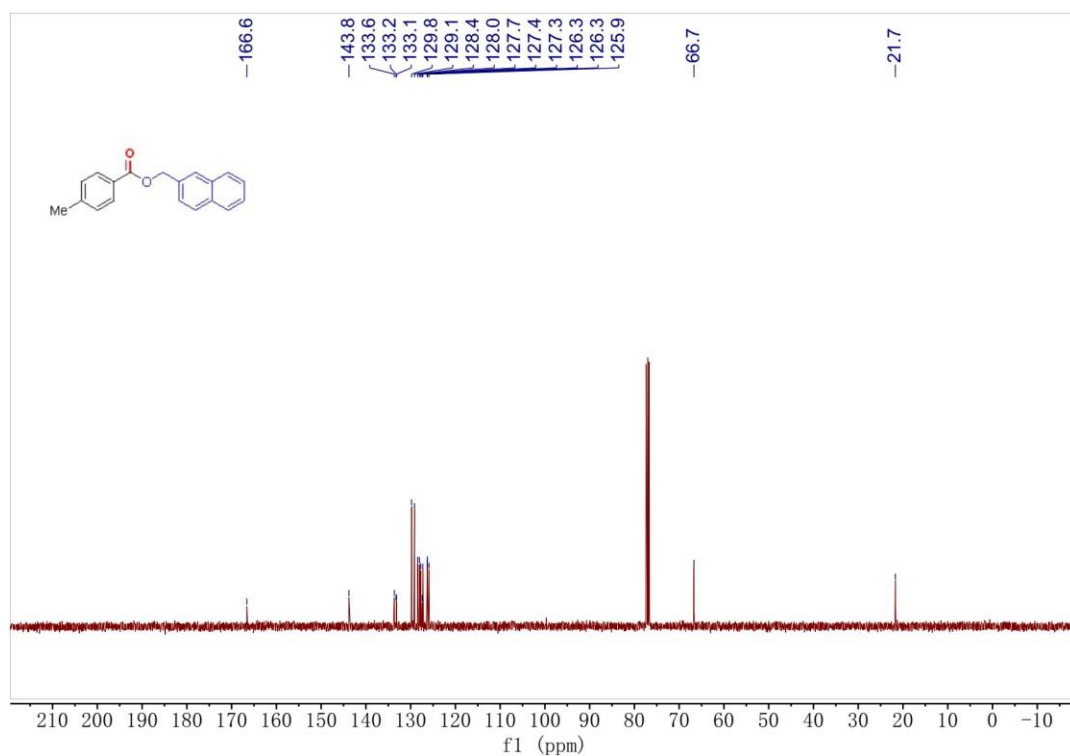
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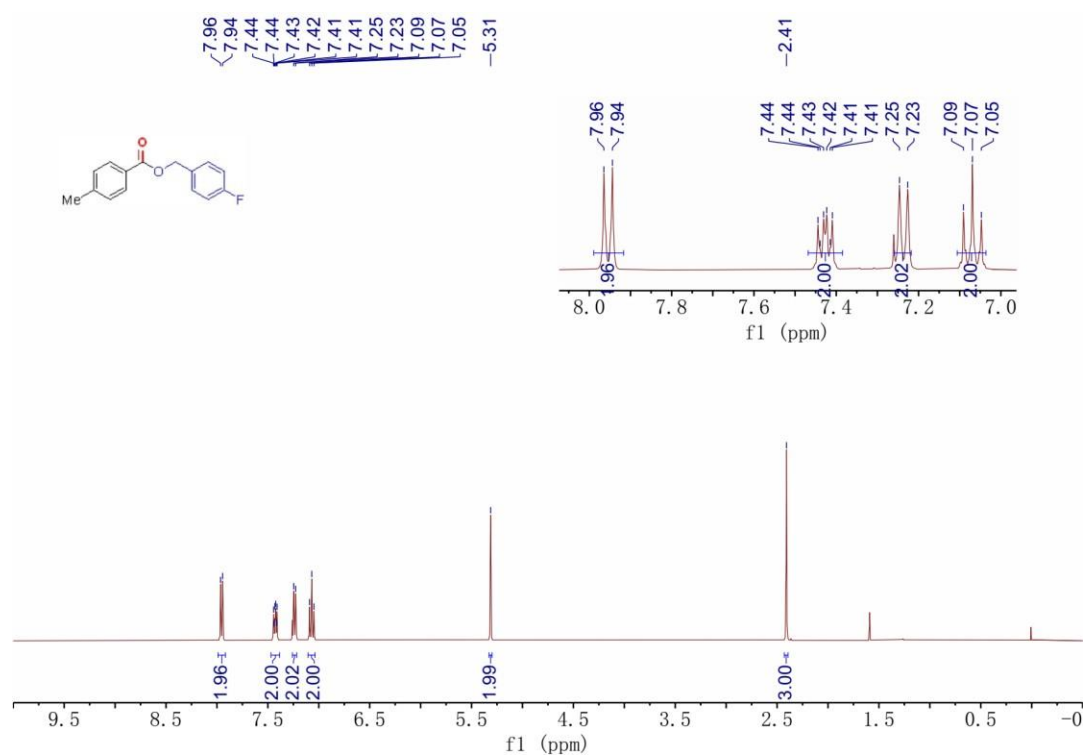
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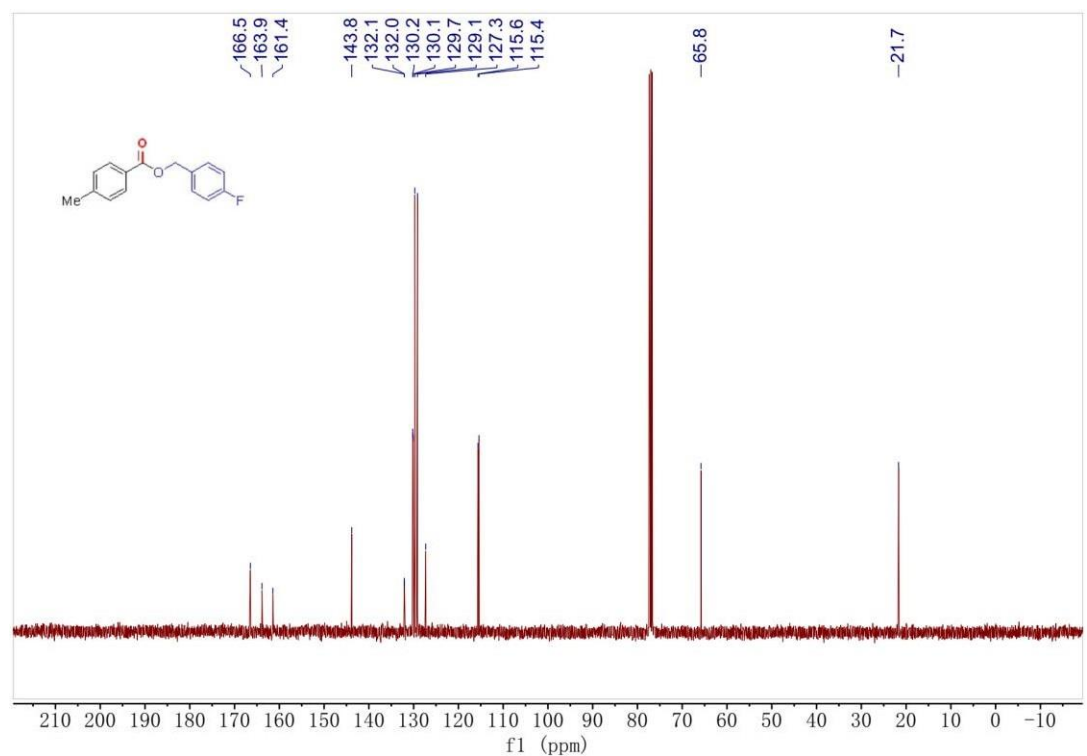
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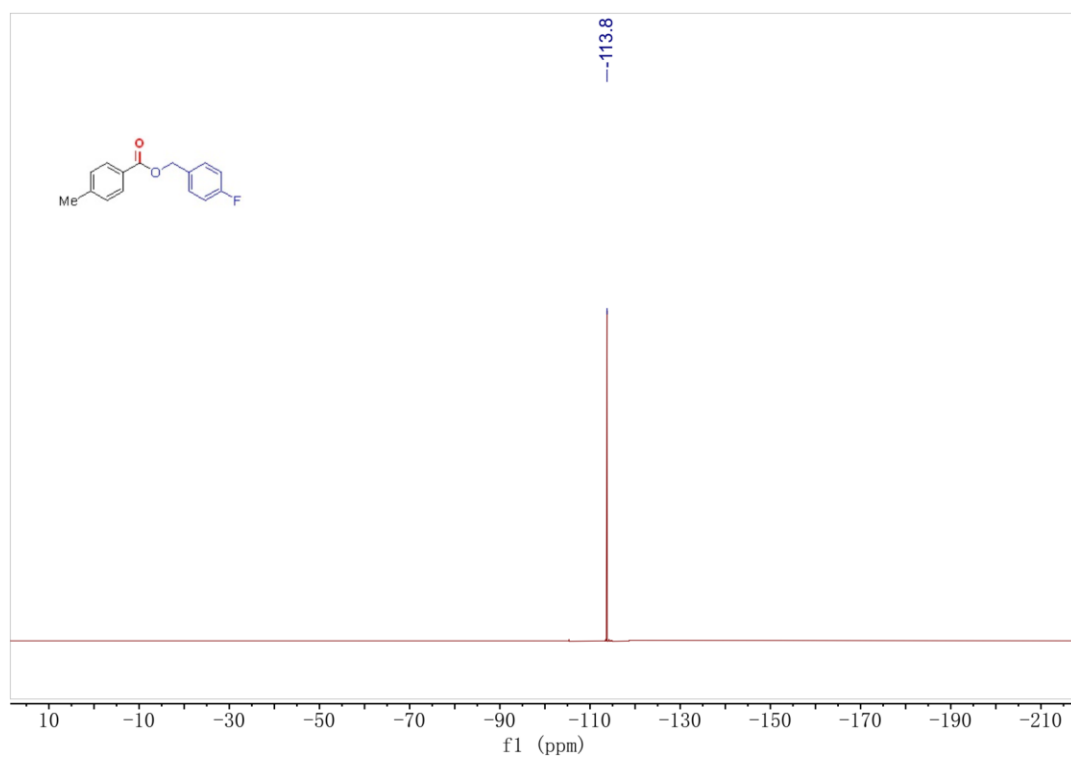
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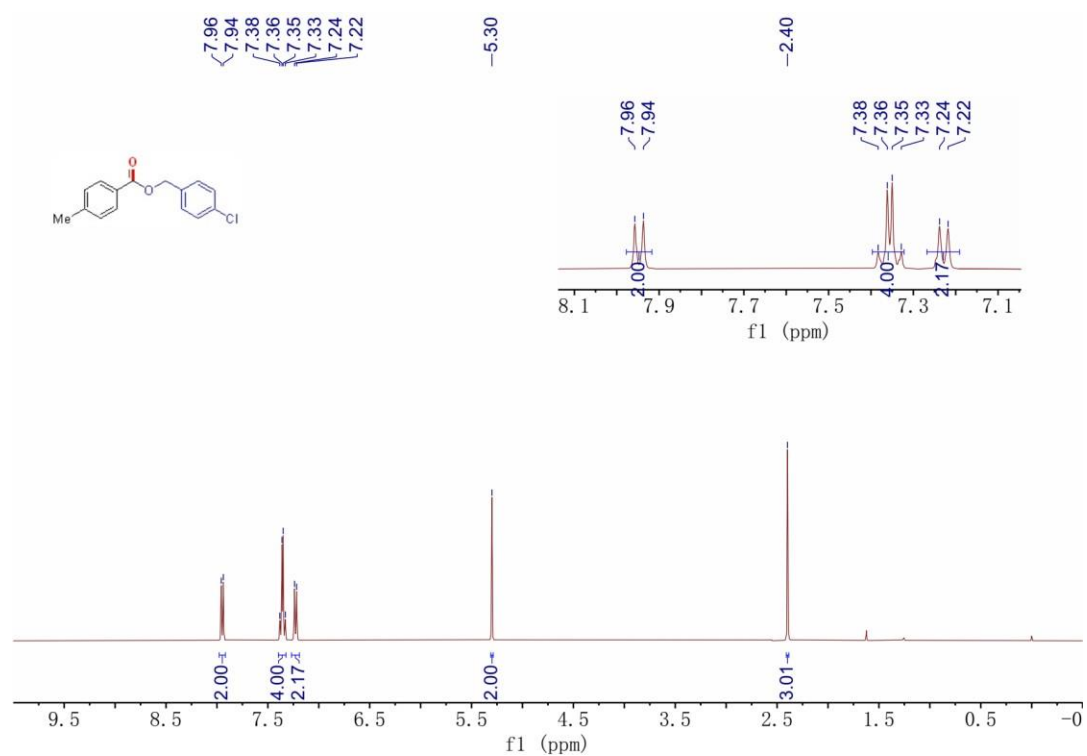
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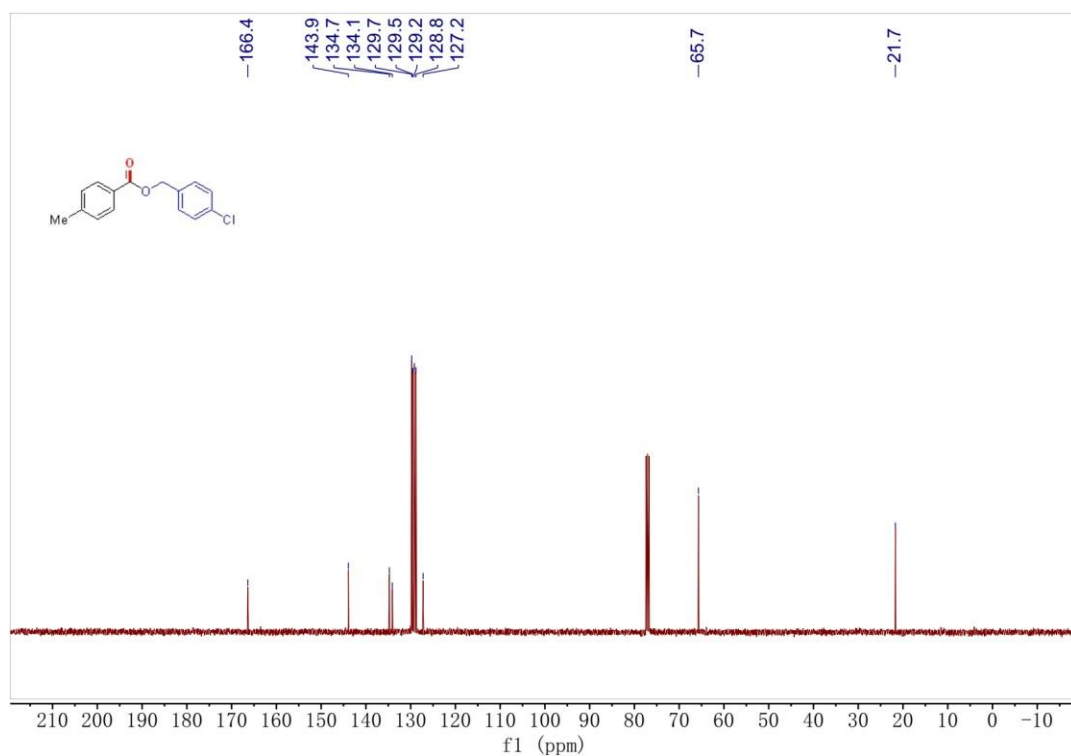
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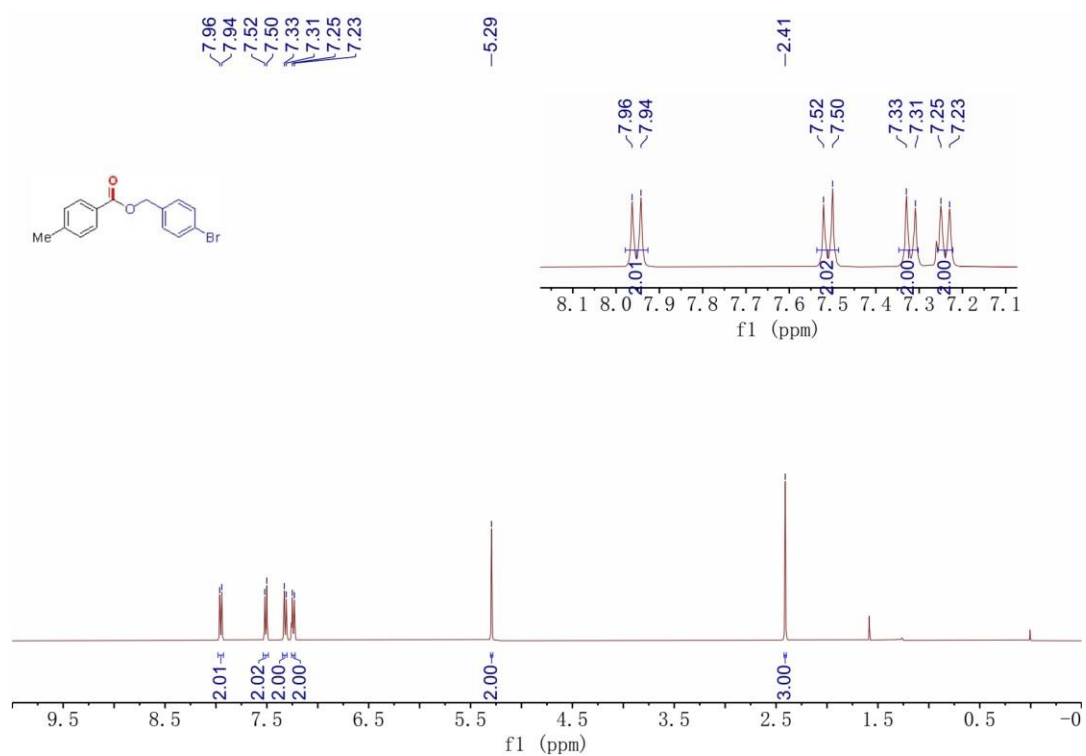
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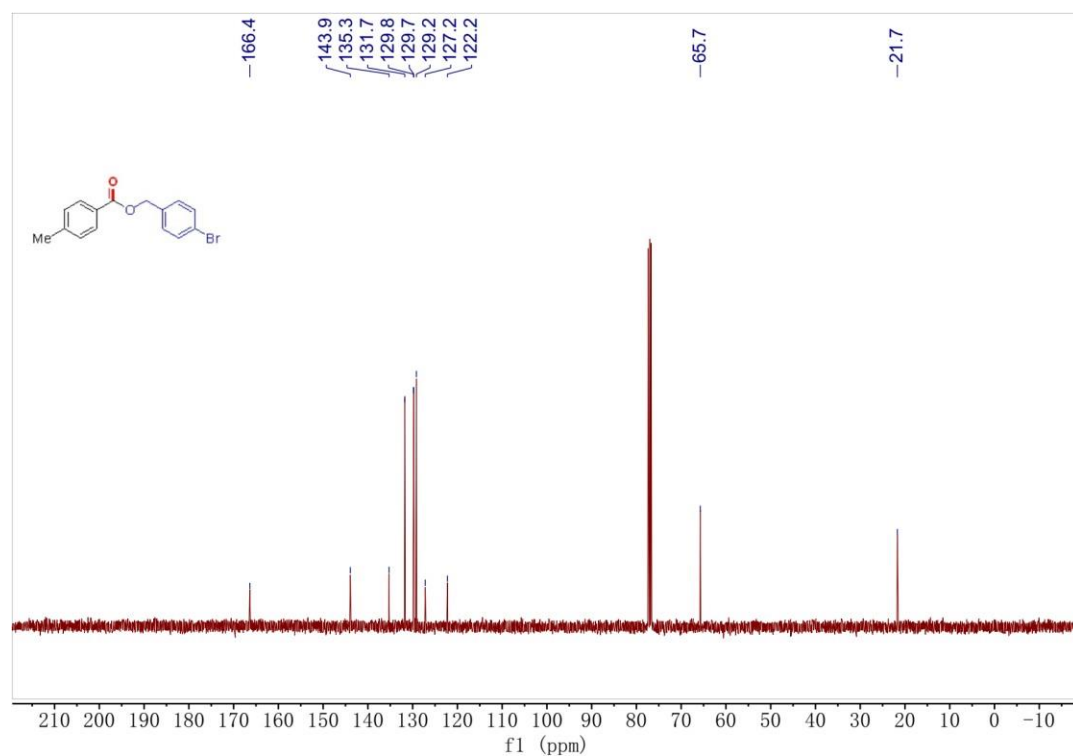
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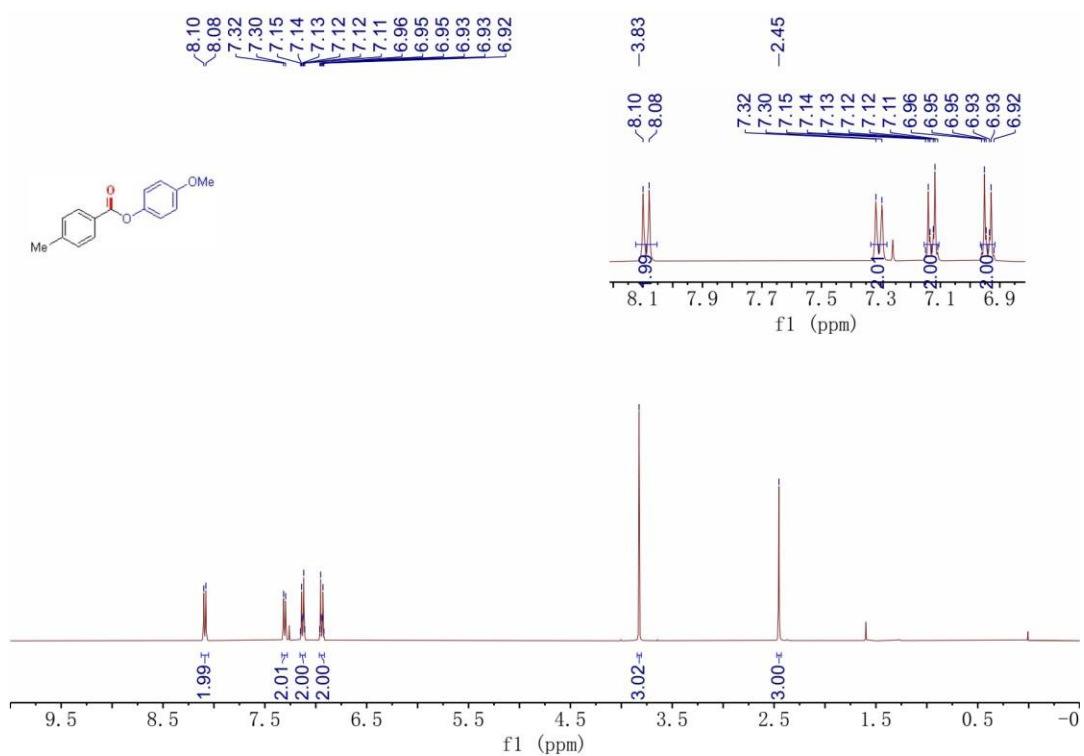
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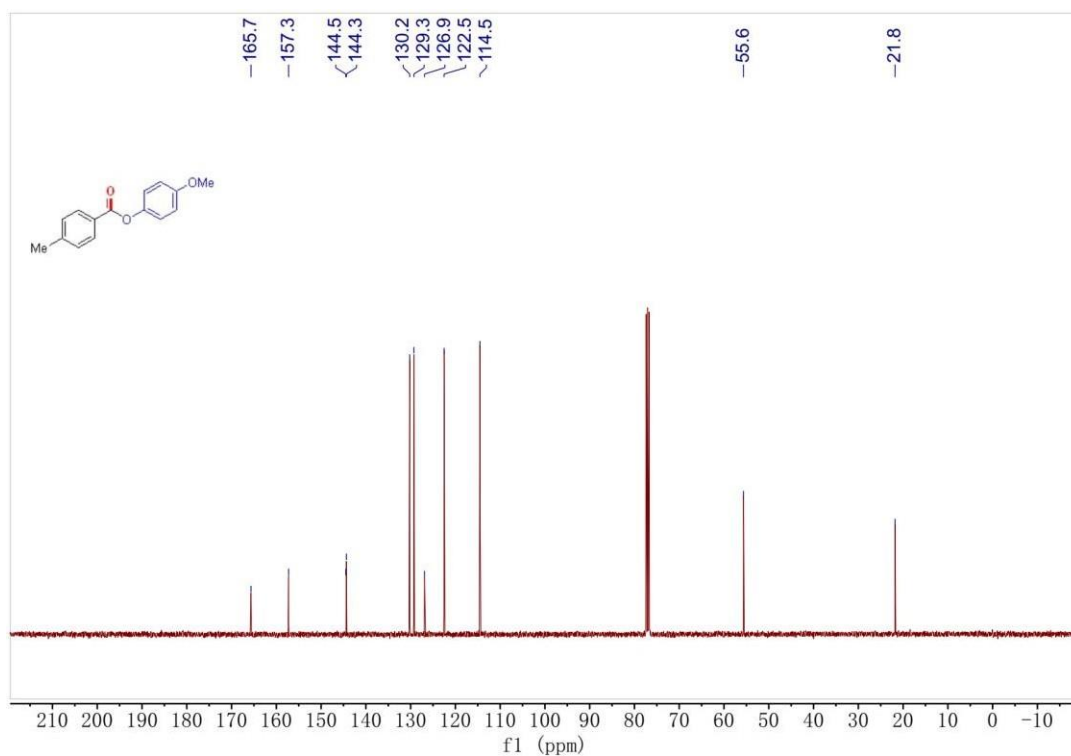
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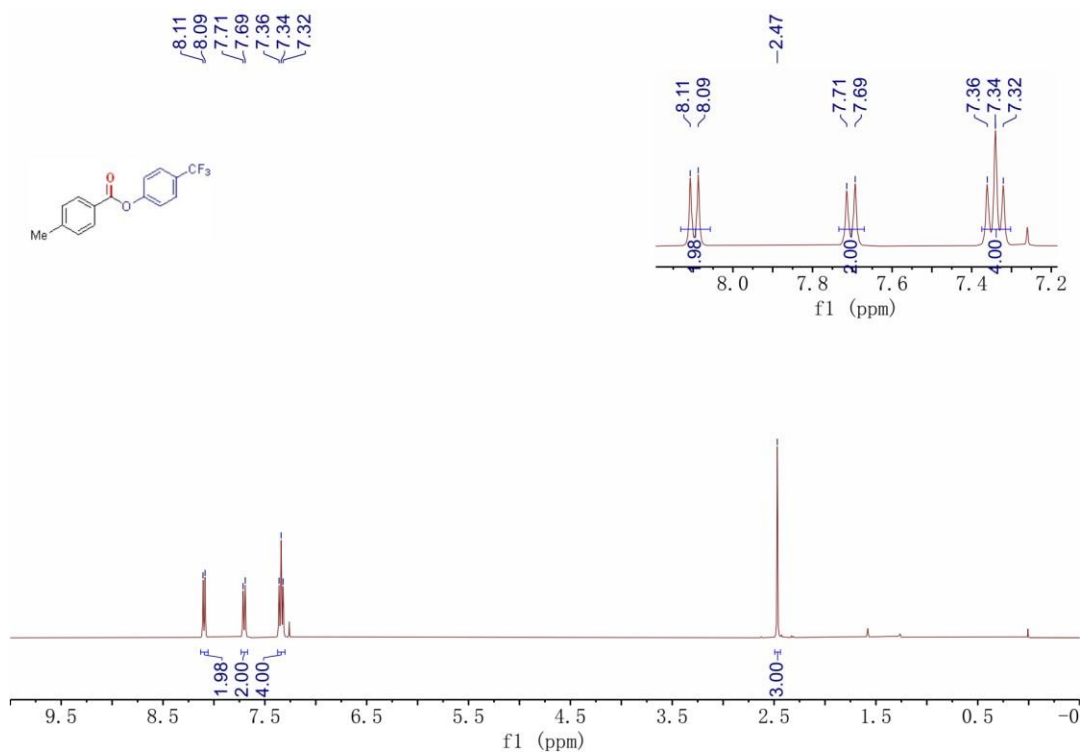
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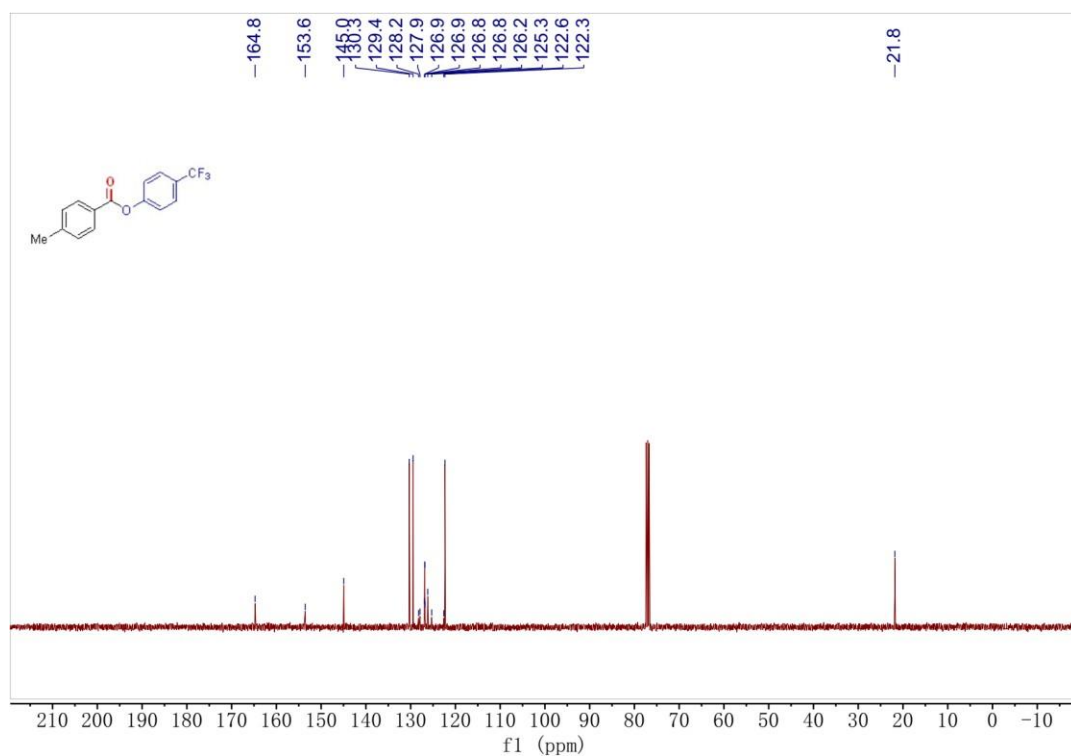
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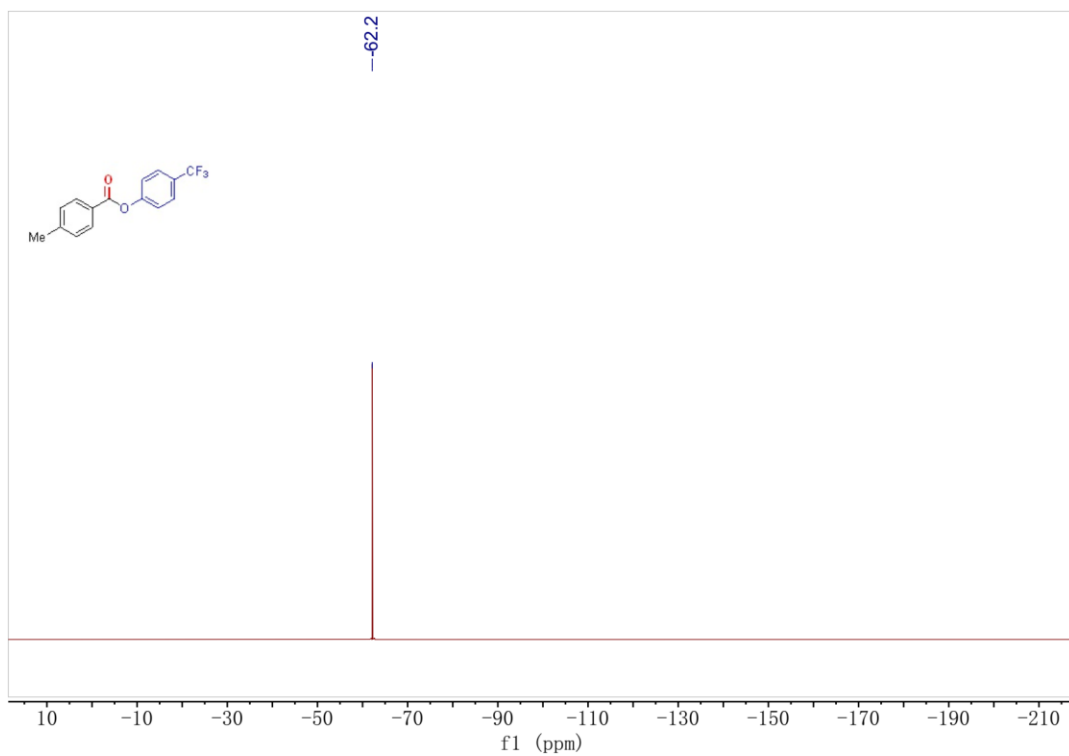
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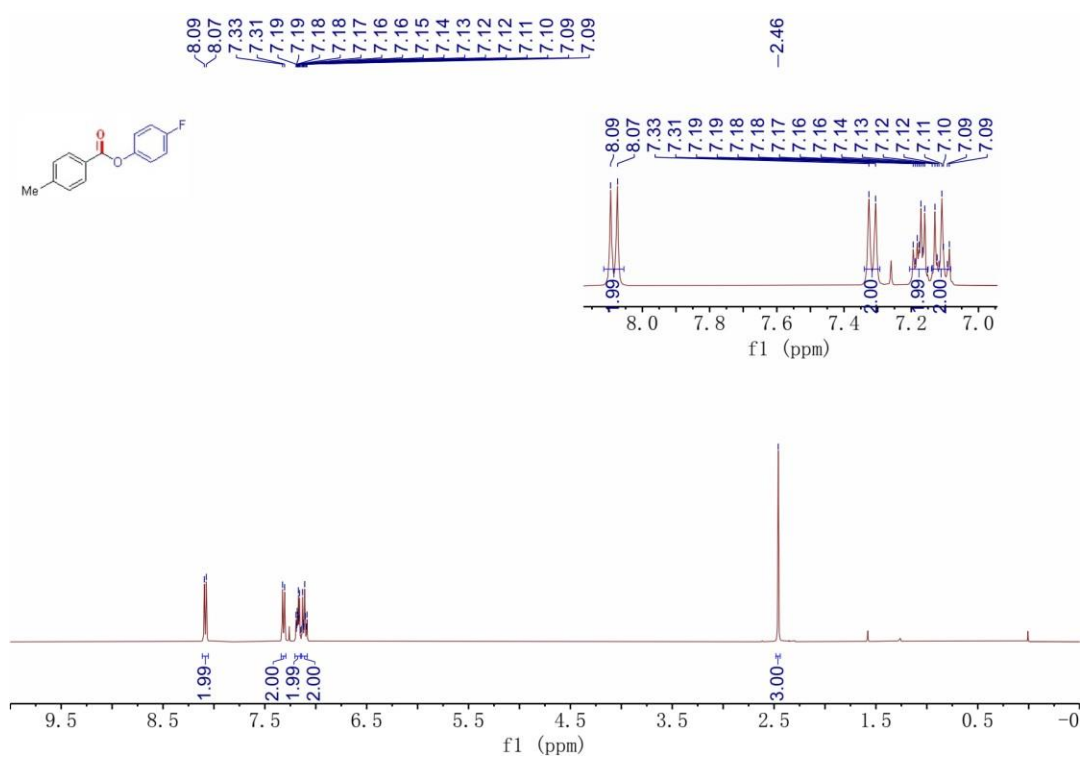
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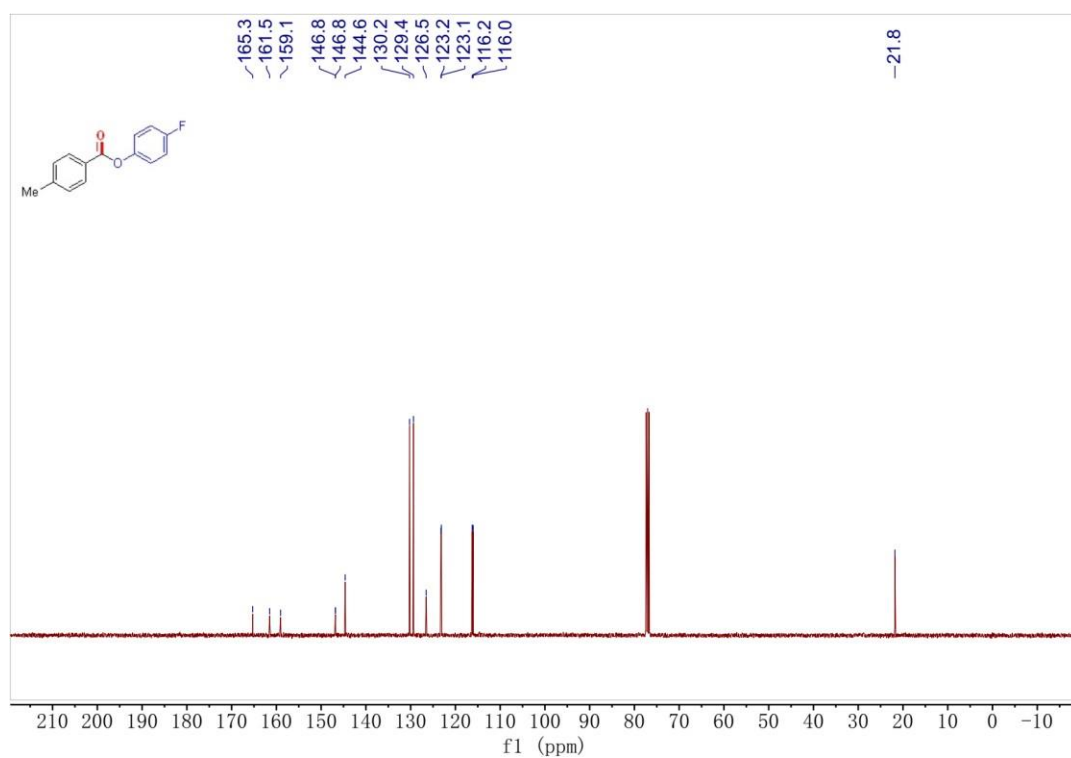
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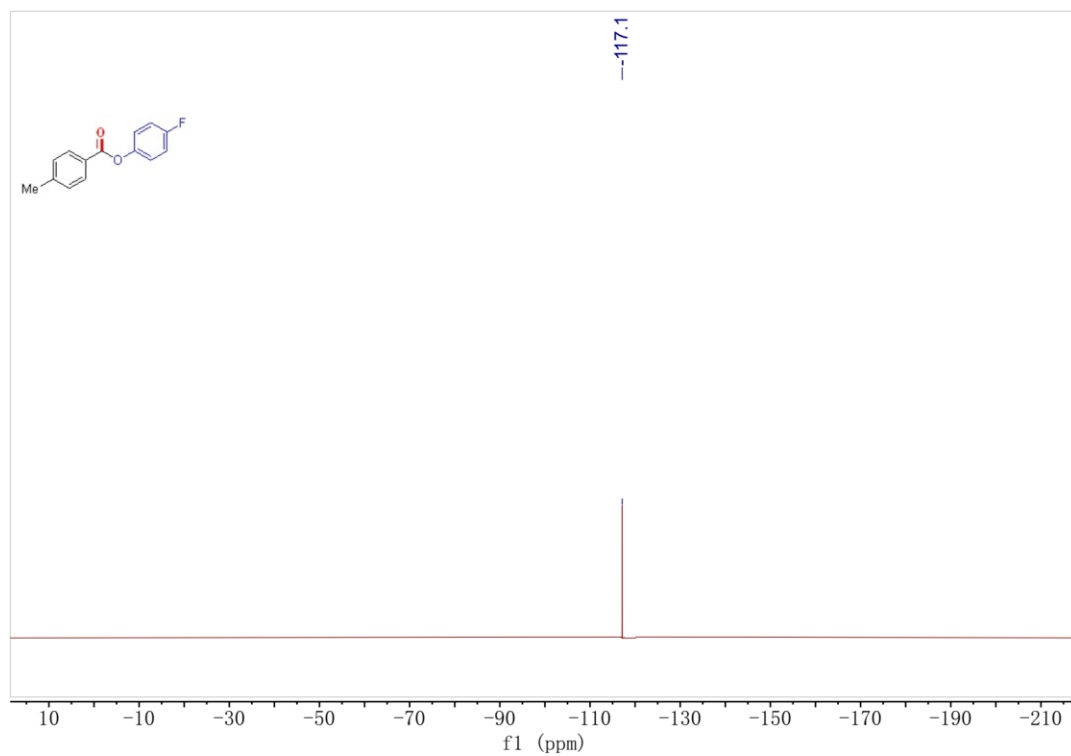
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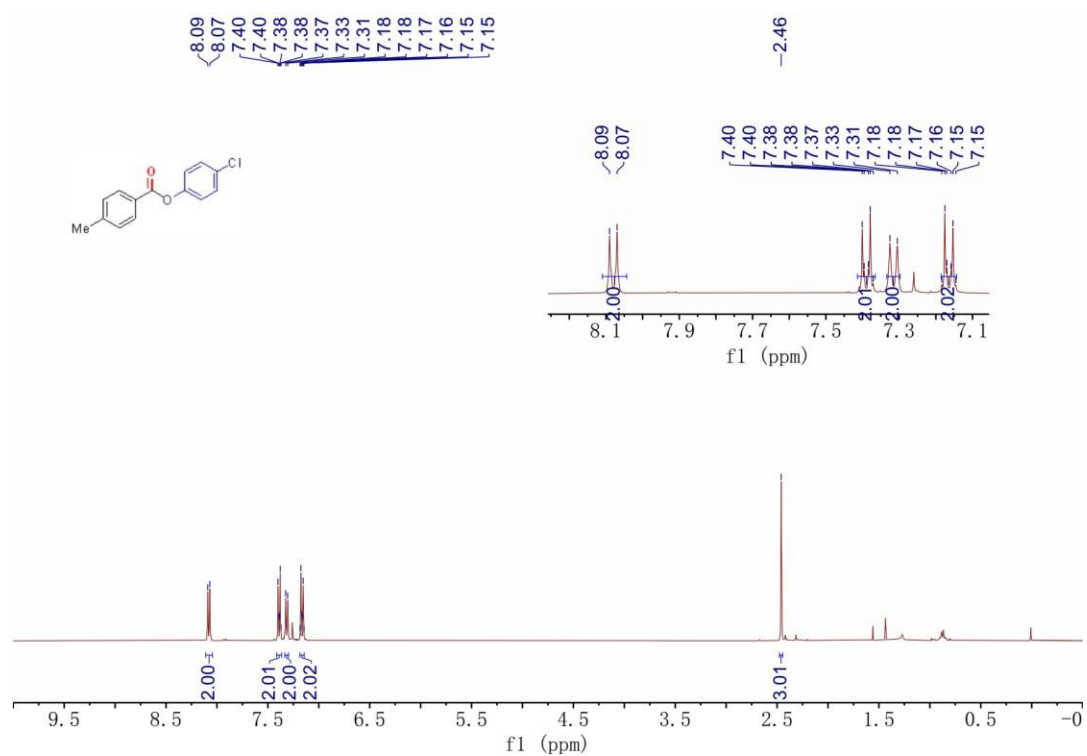
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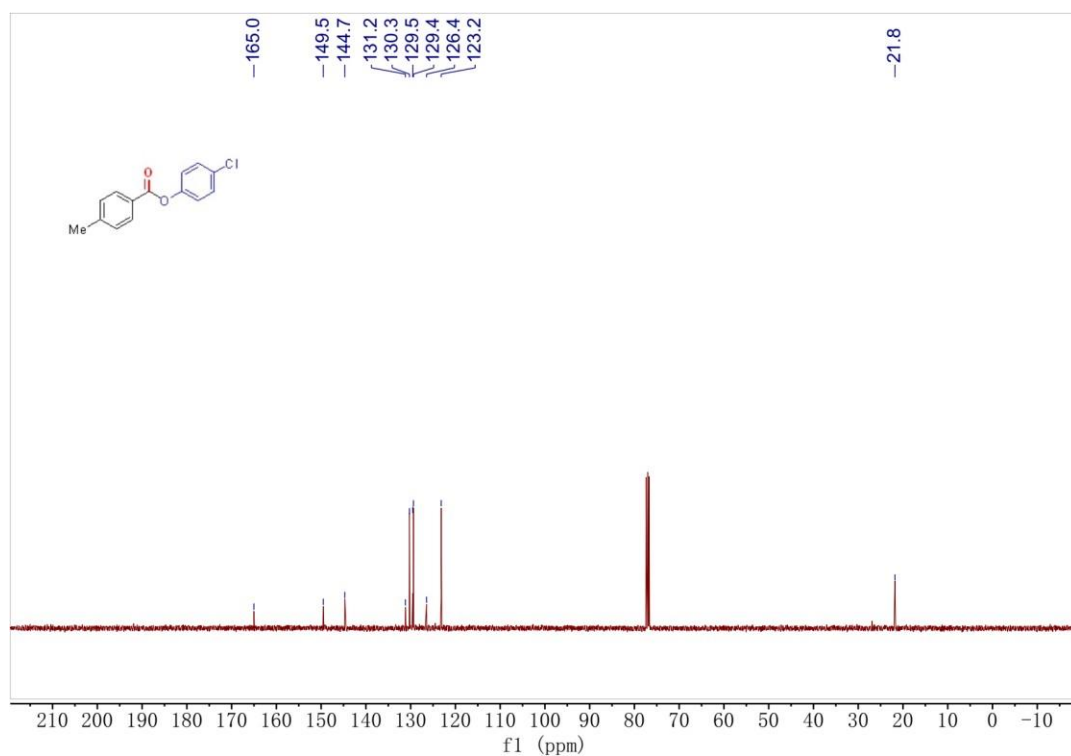
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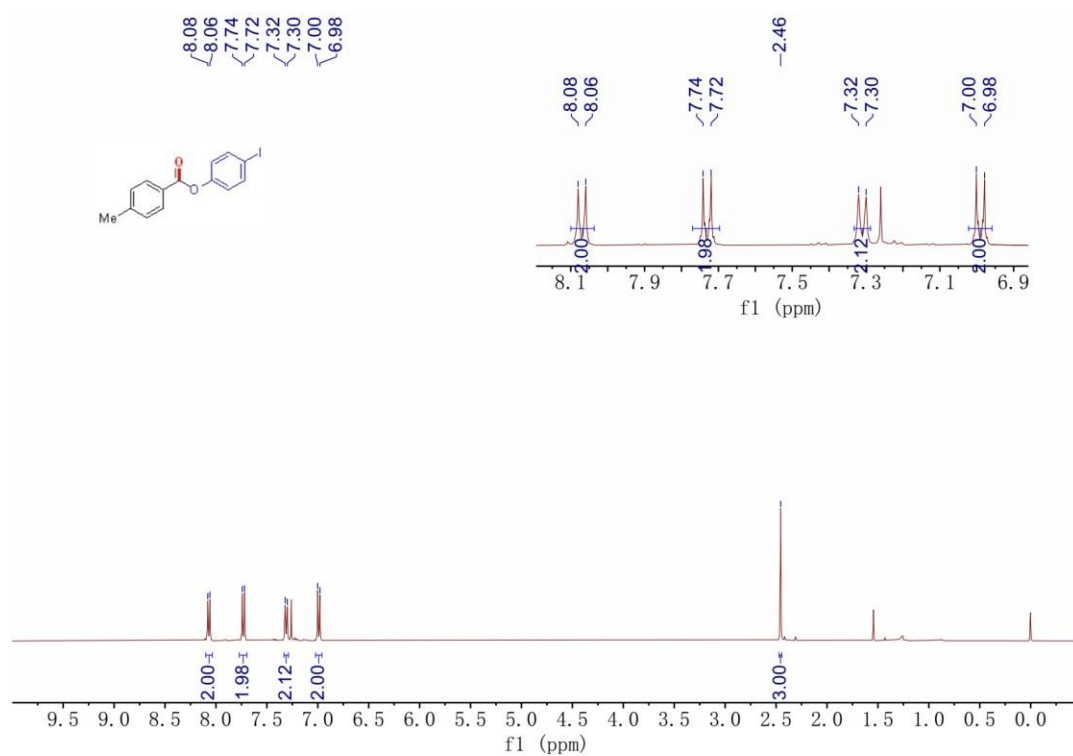
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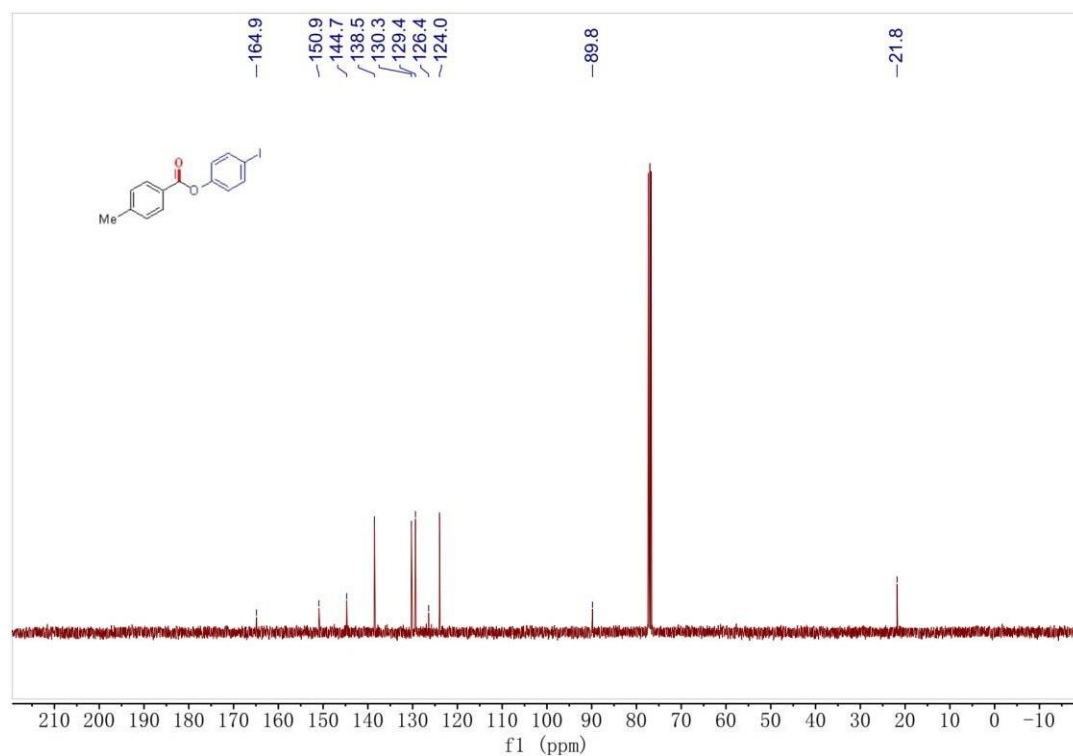
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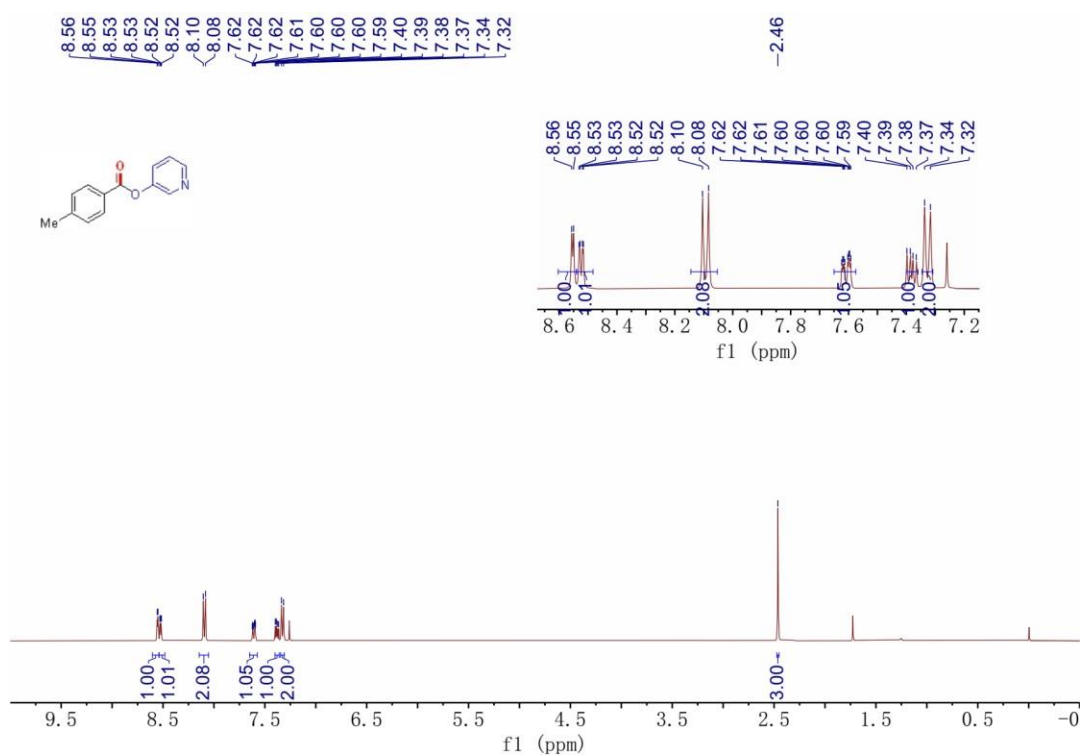
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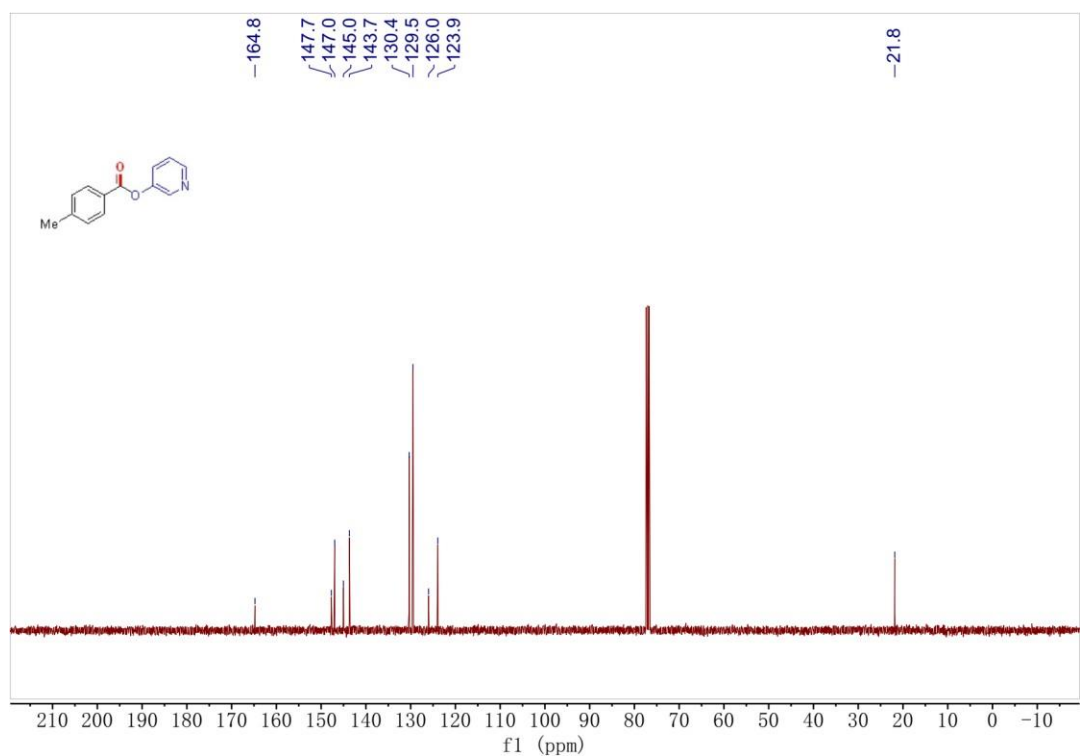
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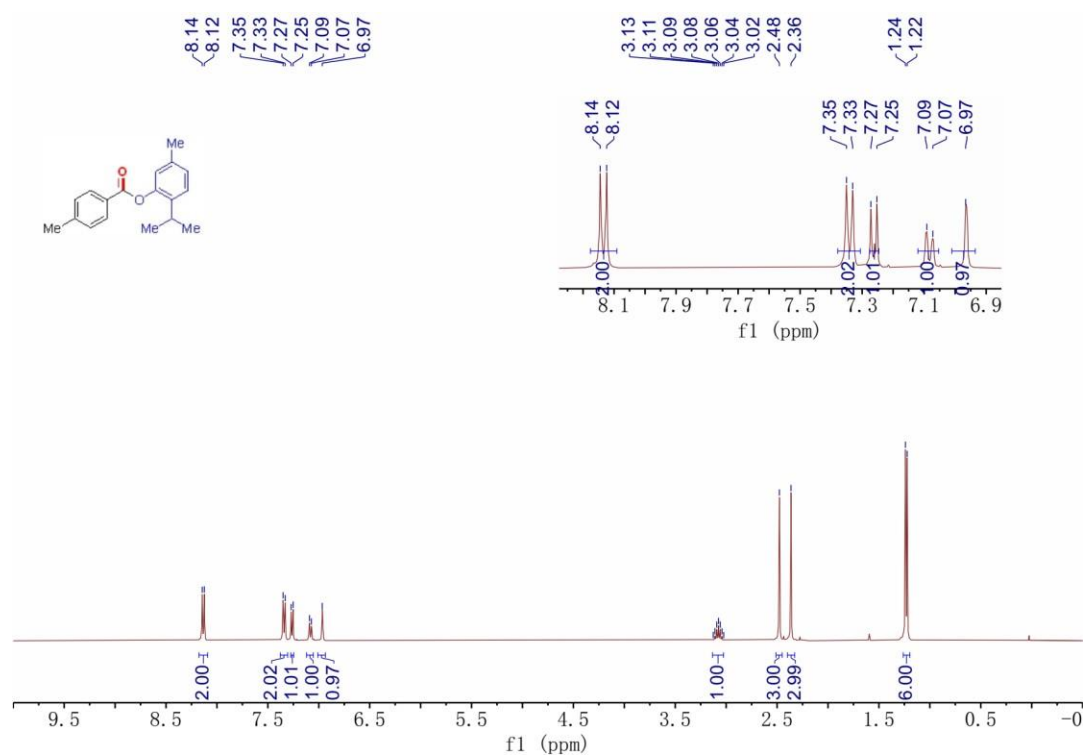
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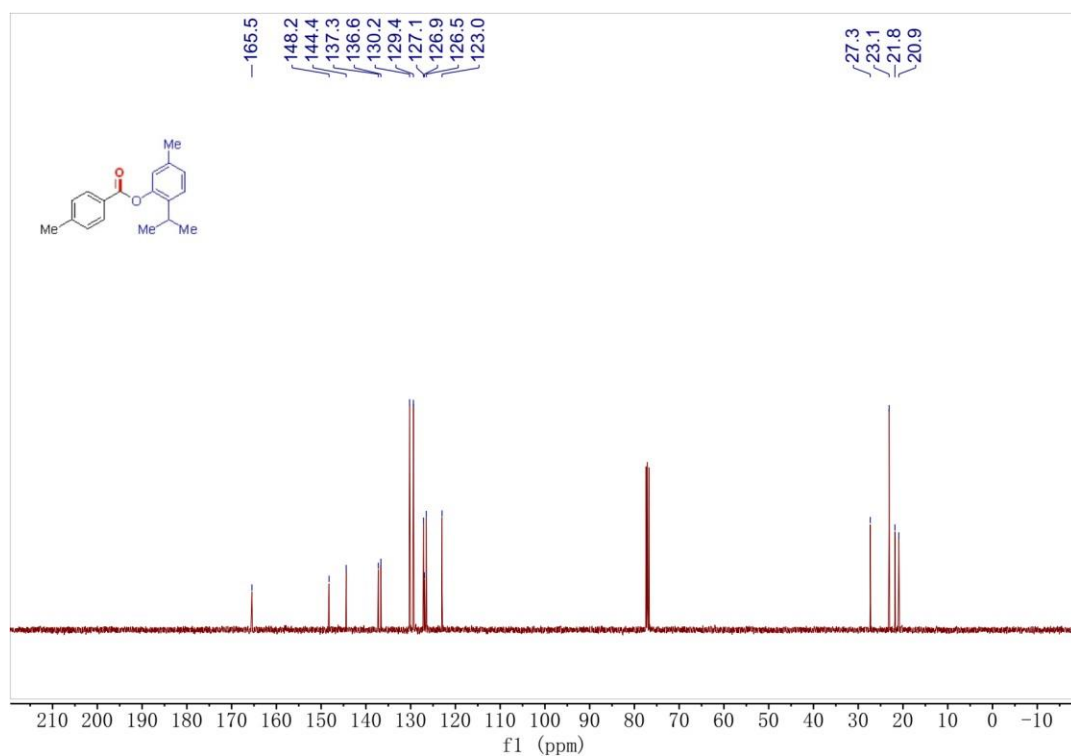
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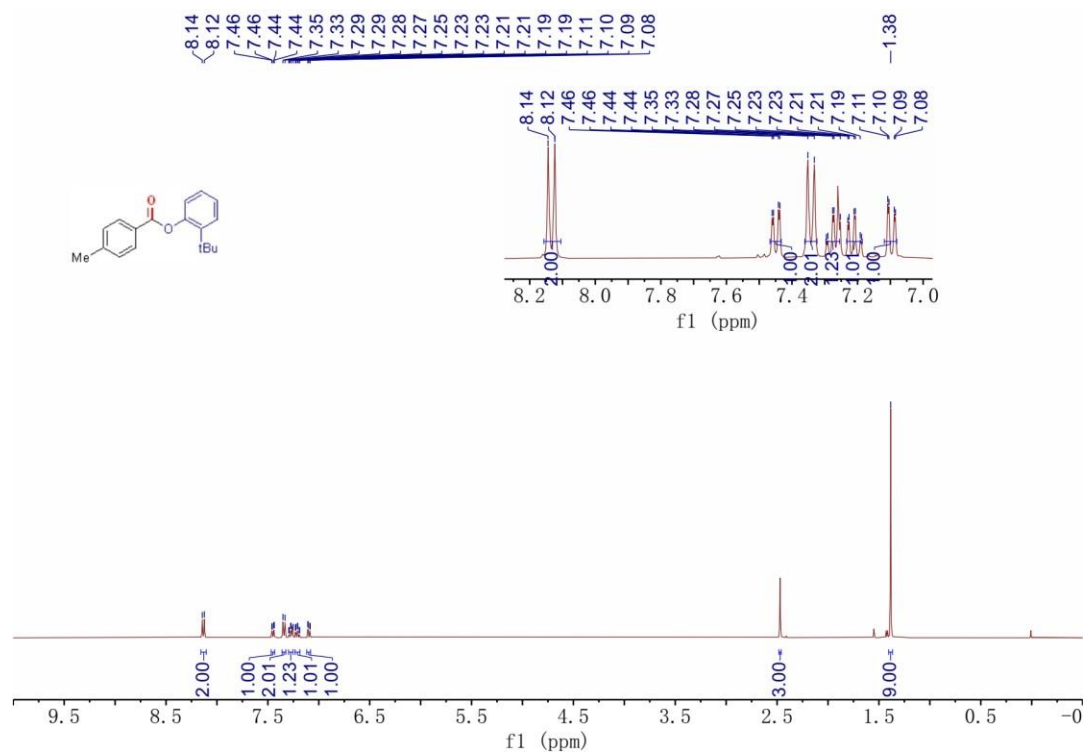
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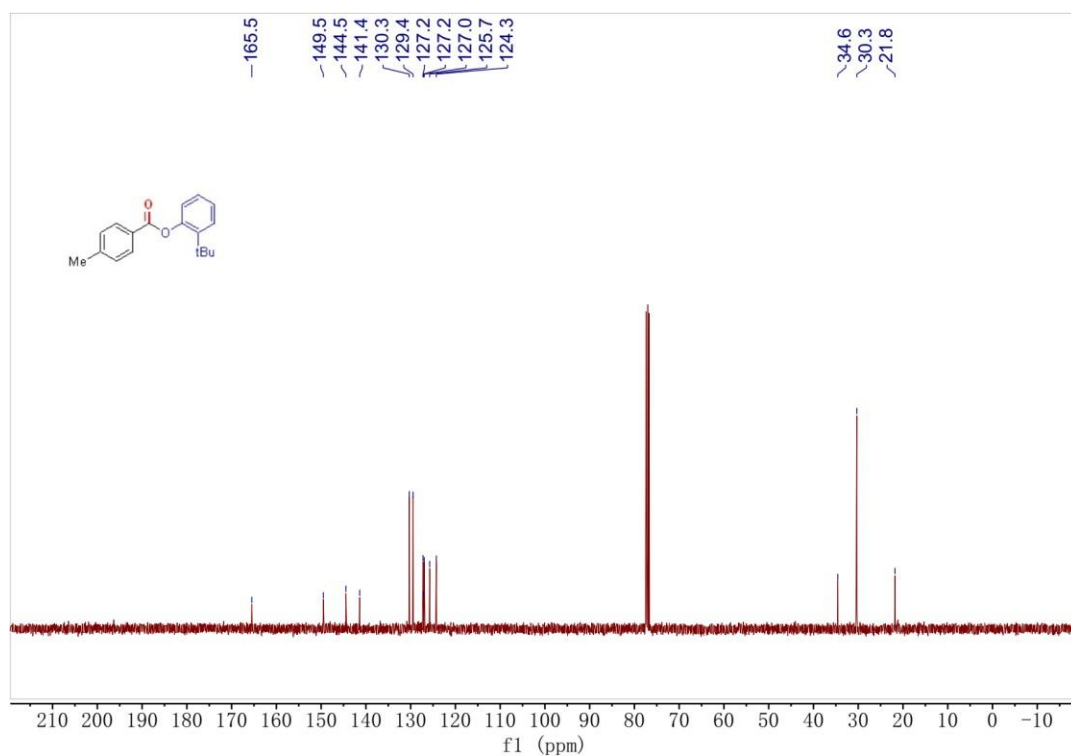
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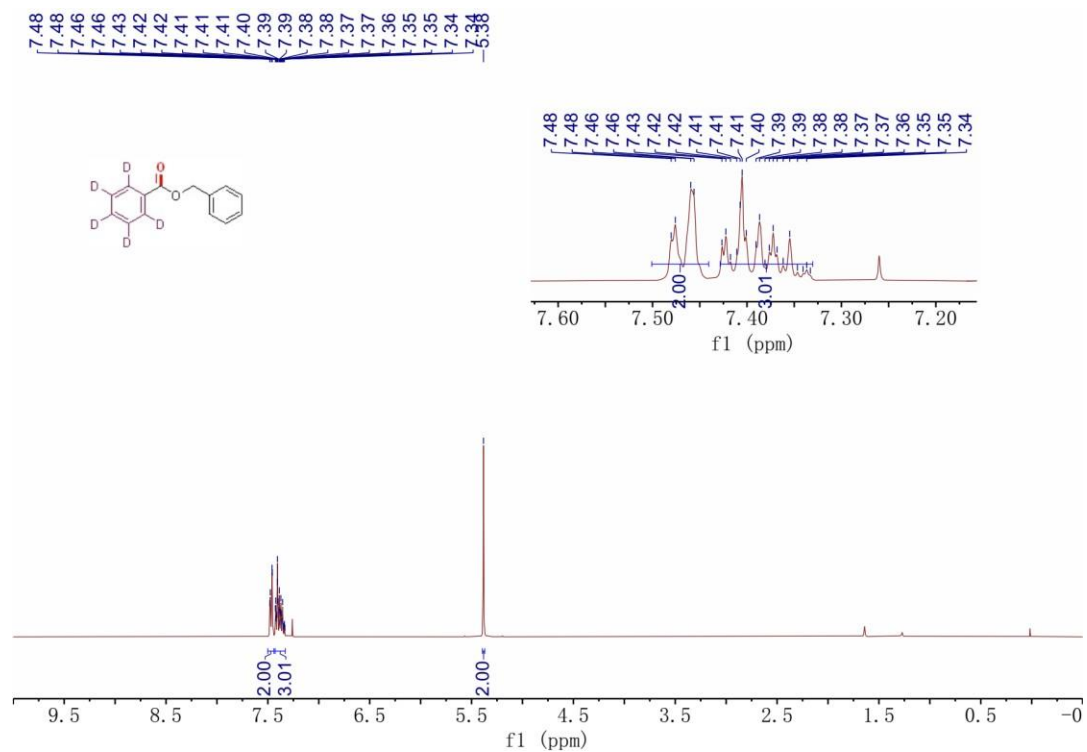
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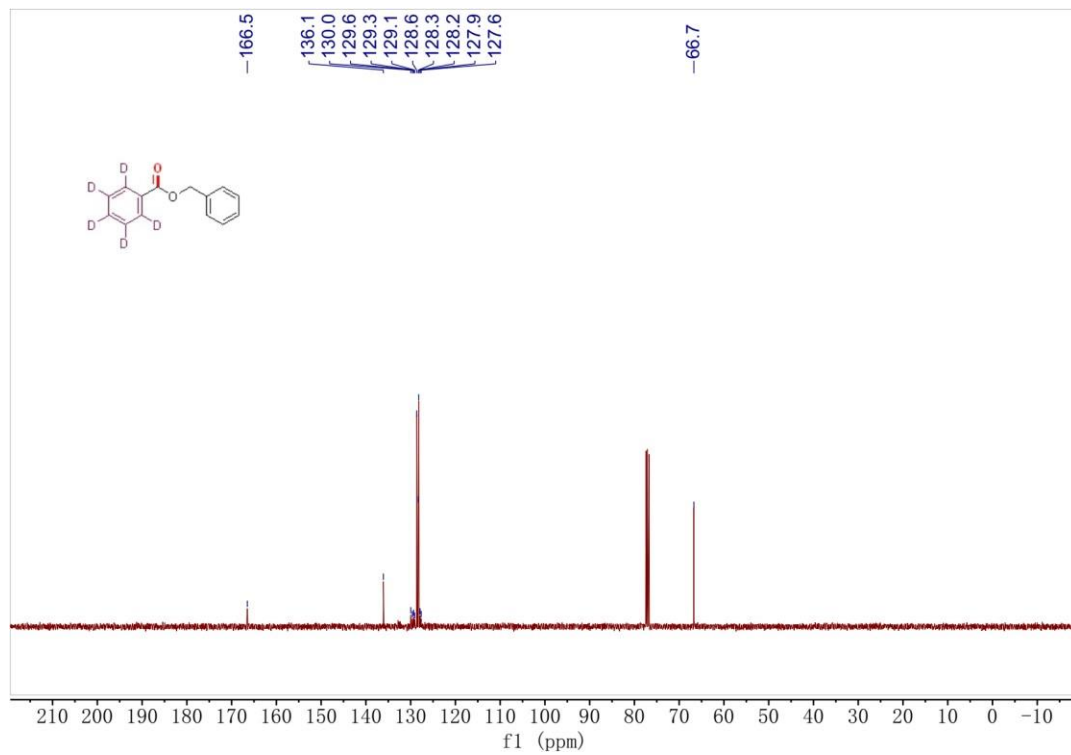
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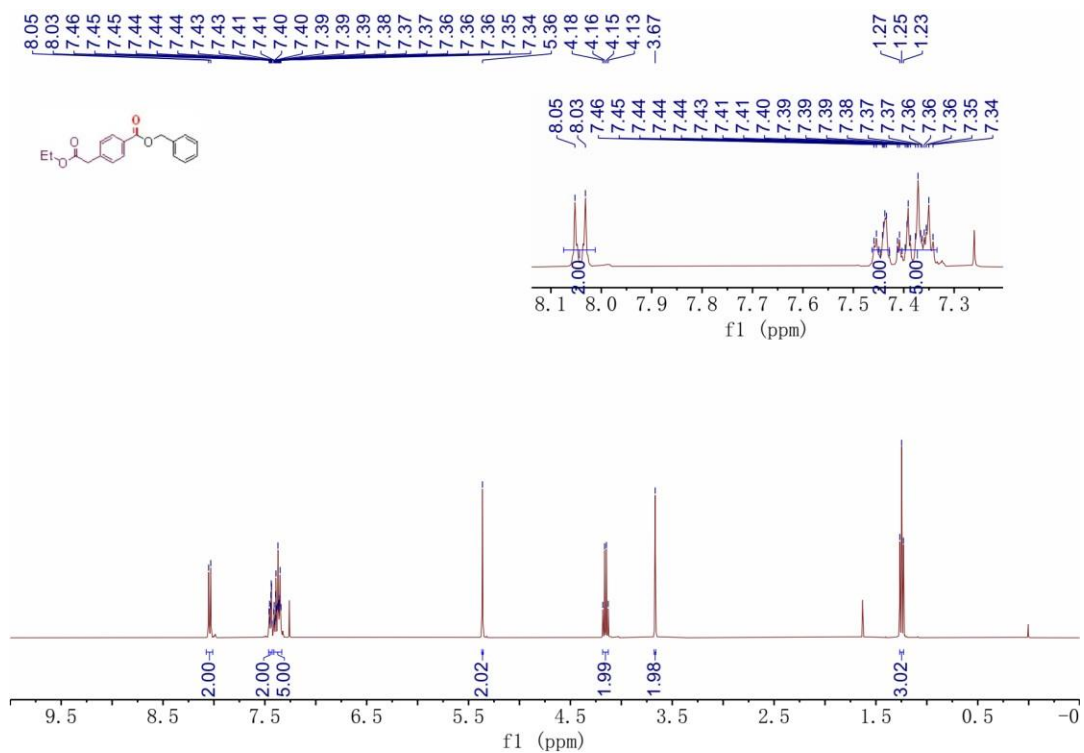
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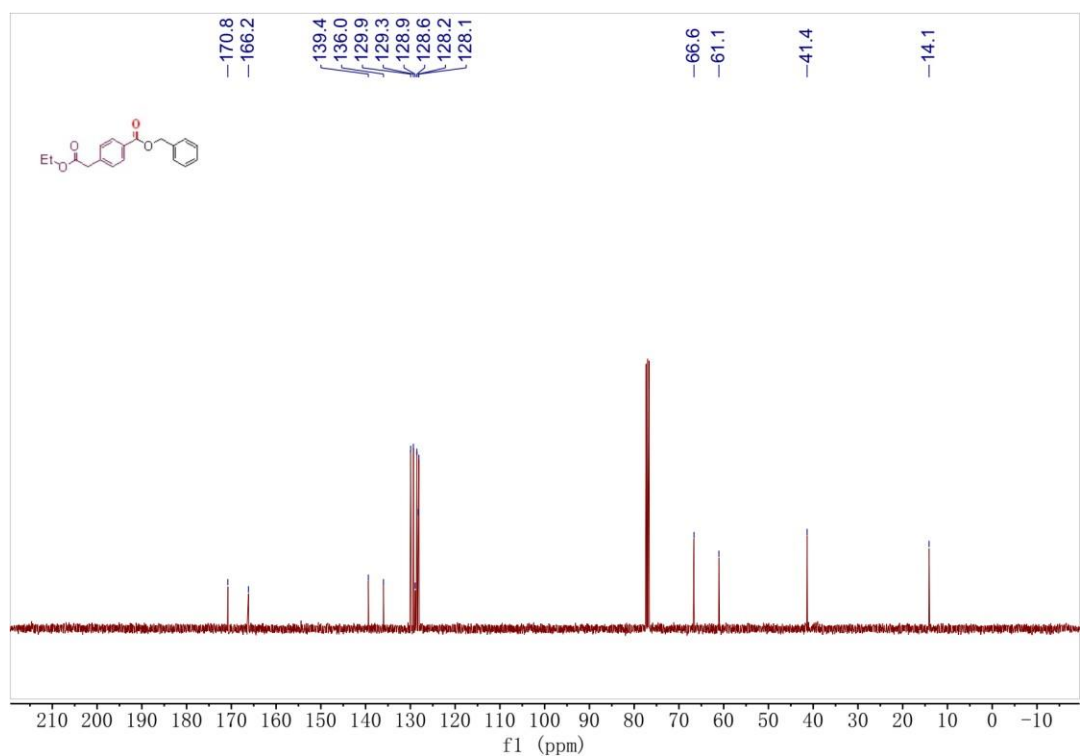
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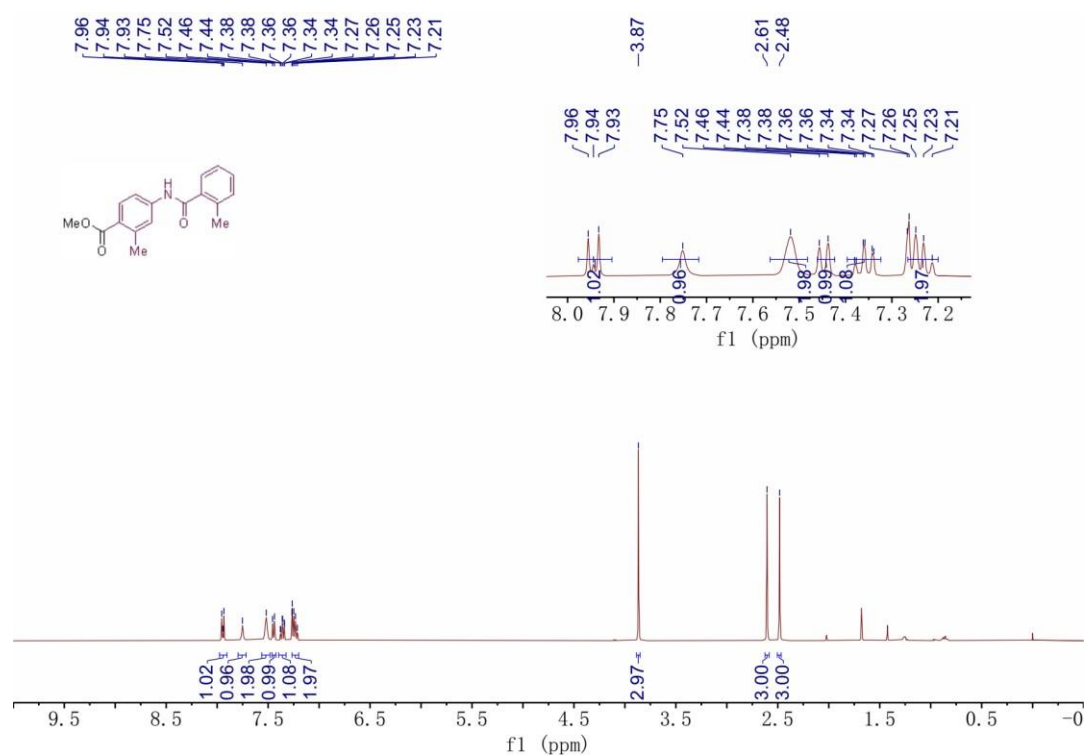
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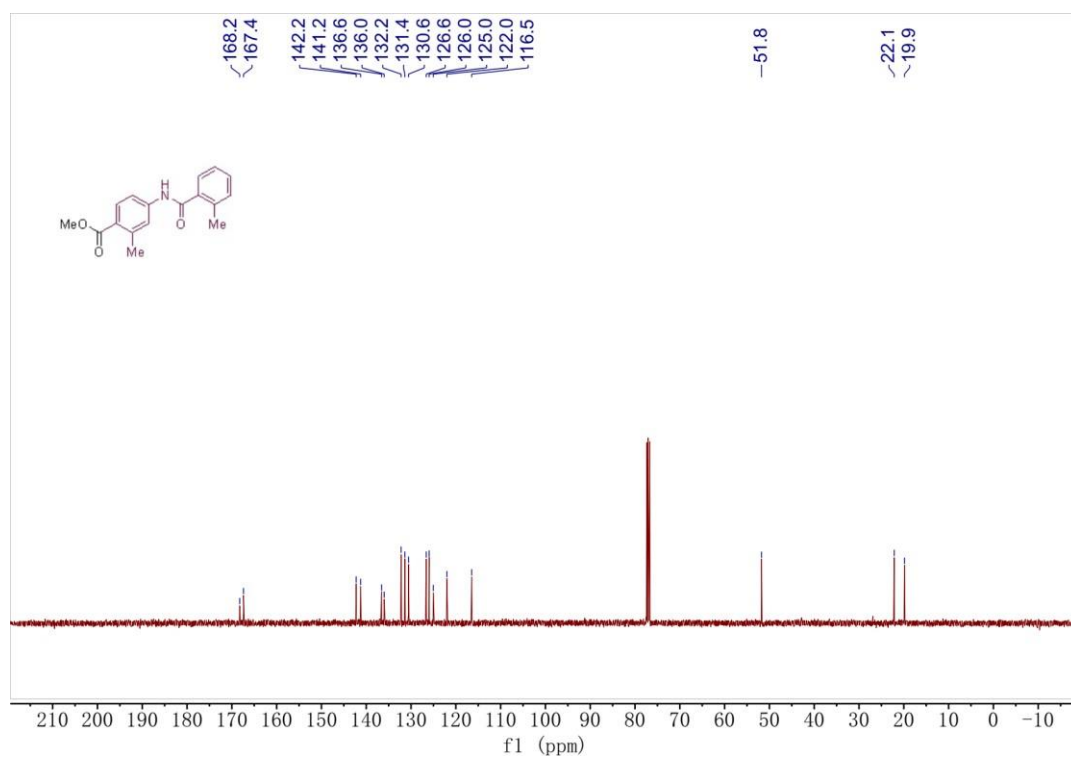
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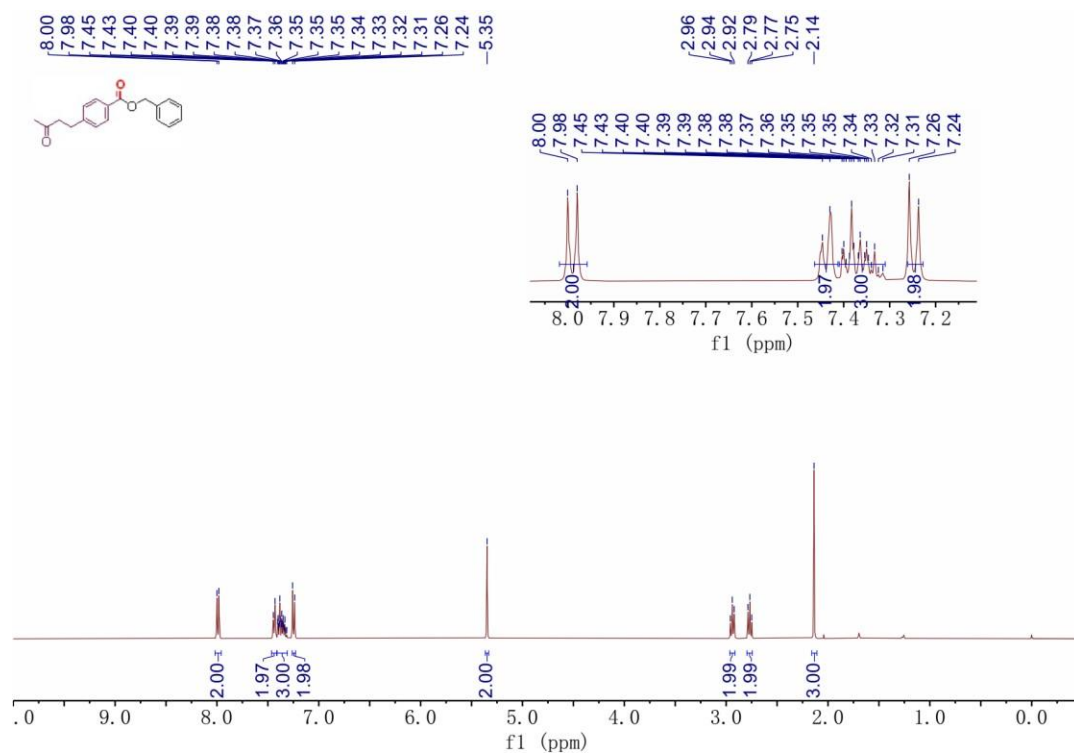
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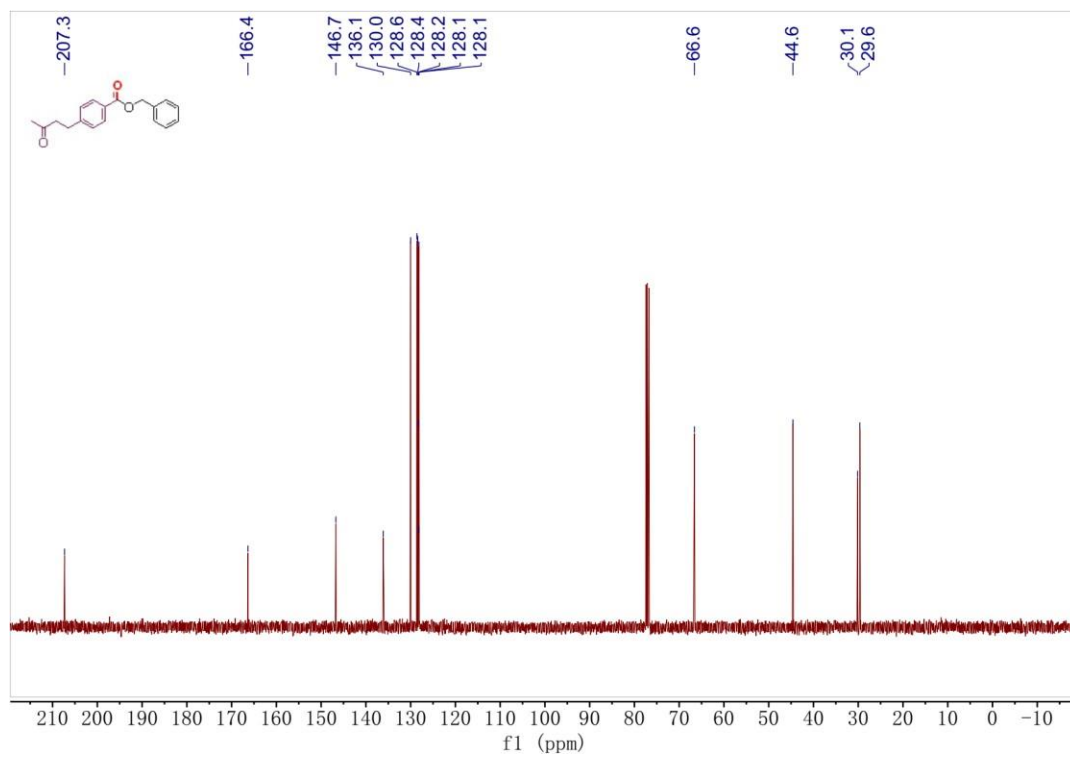
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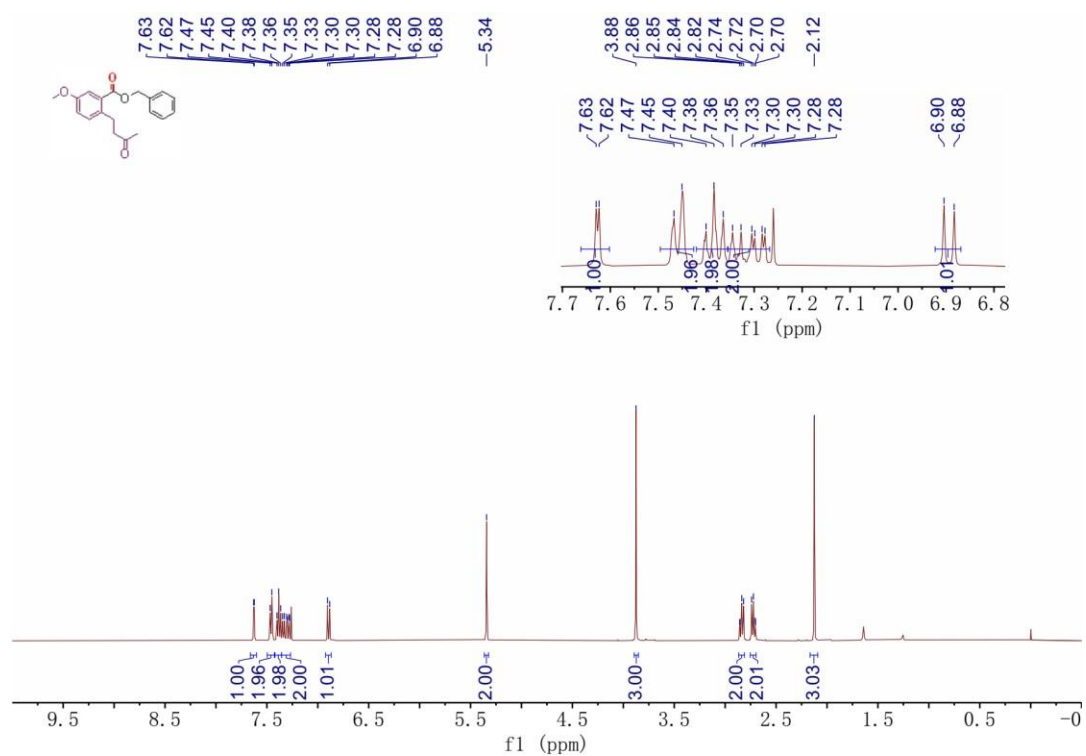
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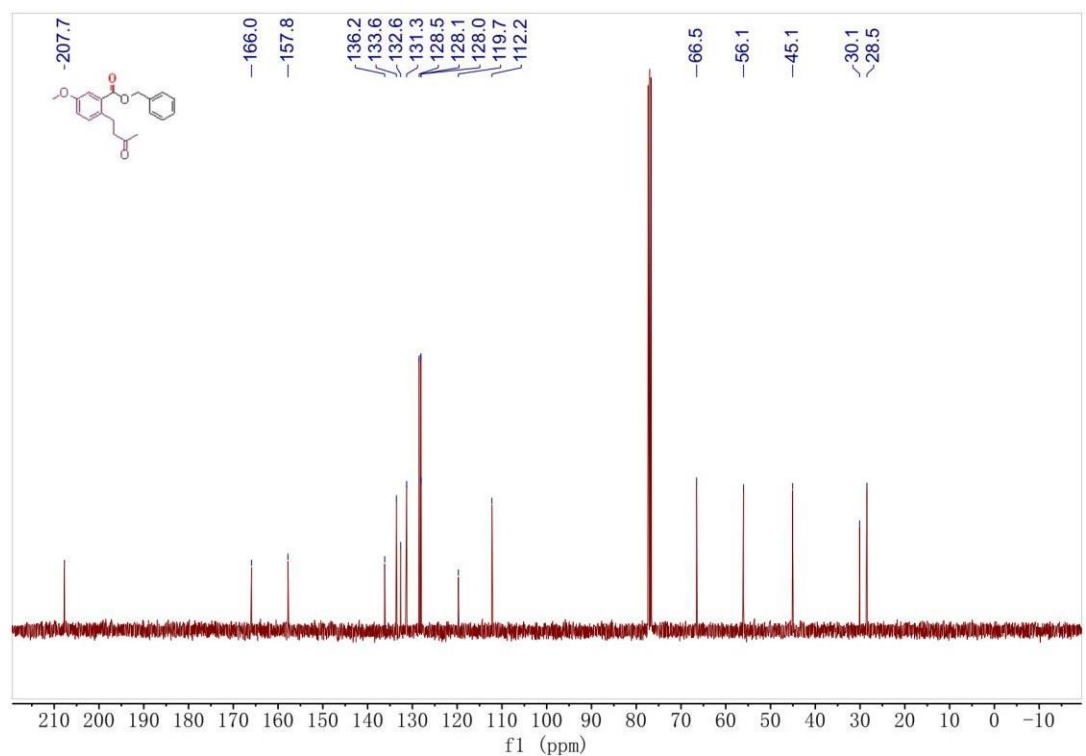
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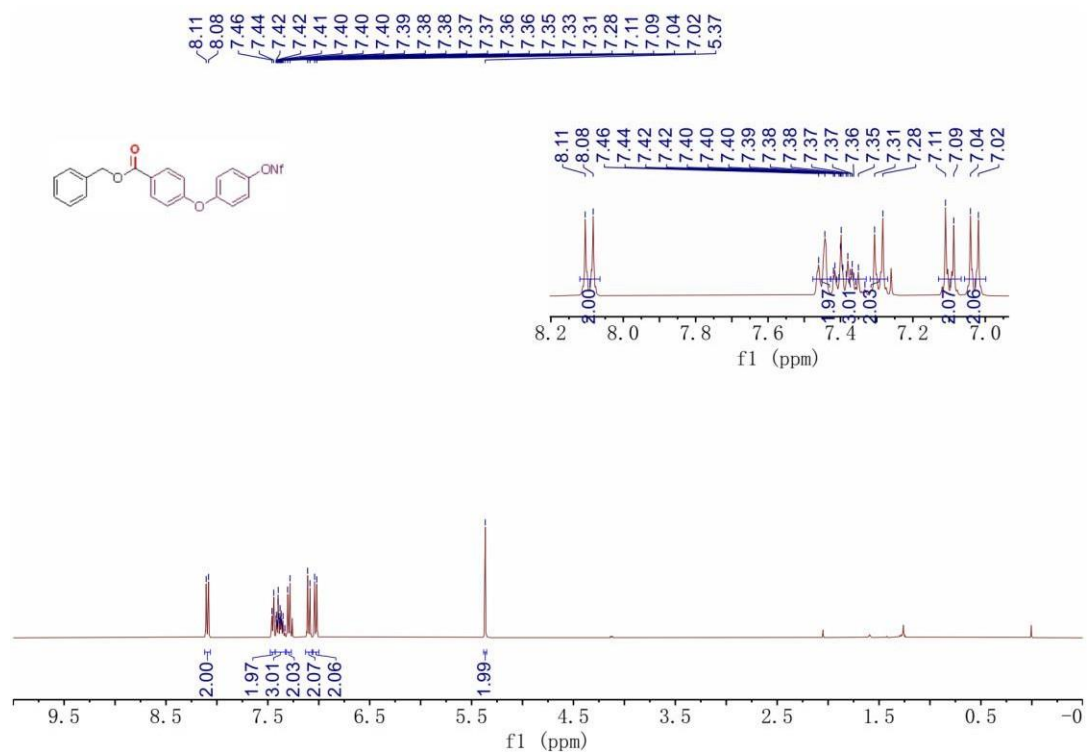
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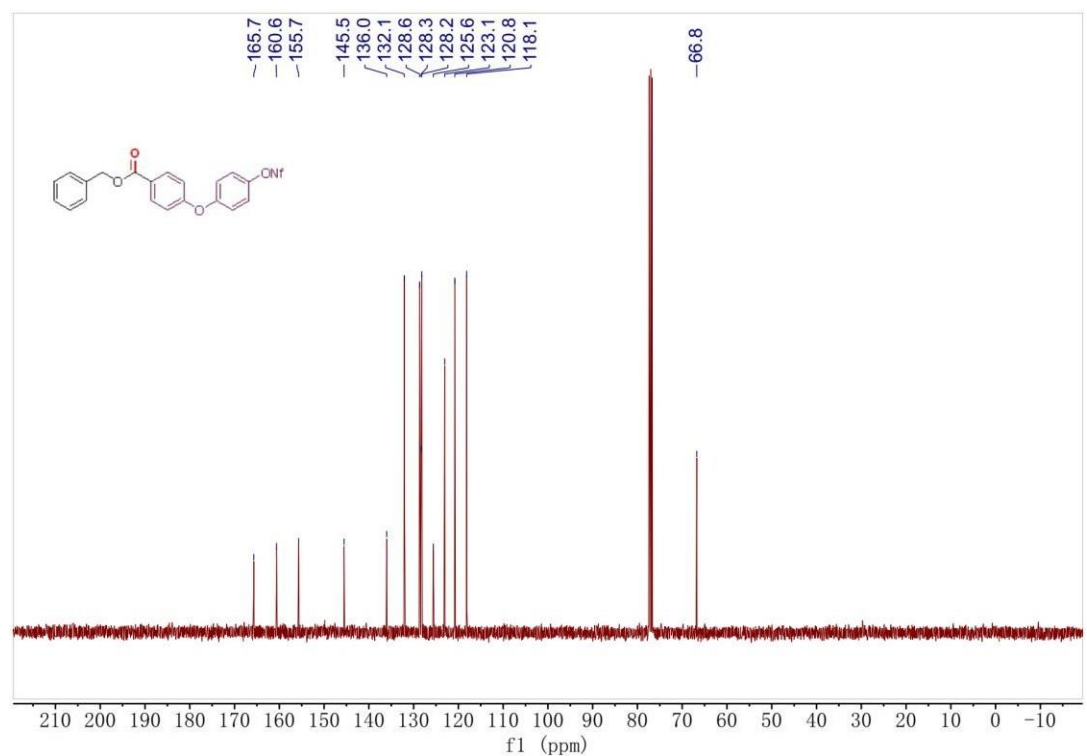
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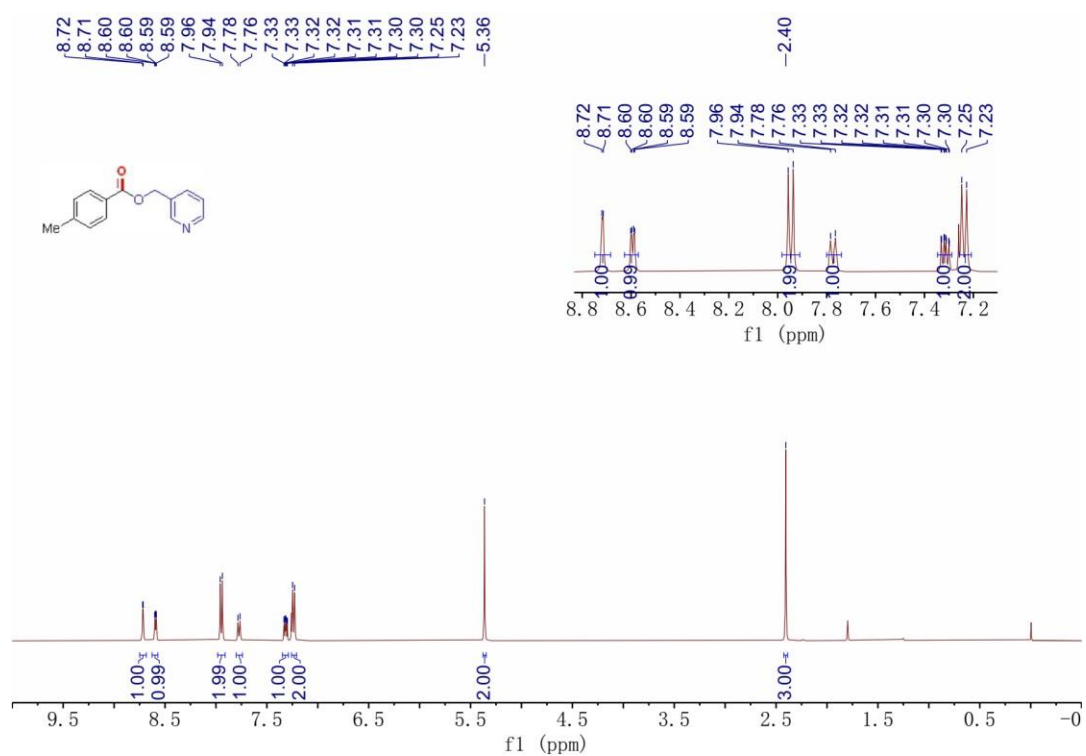
¹H NMR (400 MHz, CDCl₃) 376



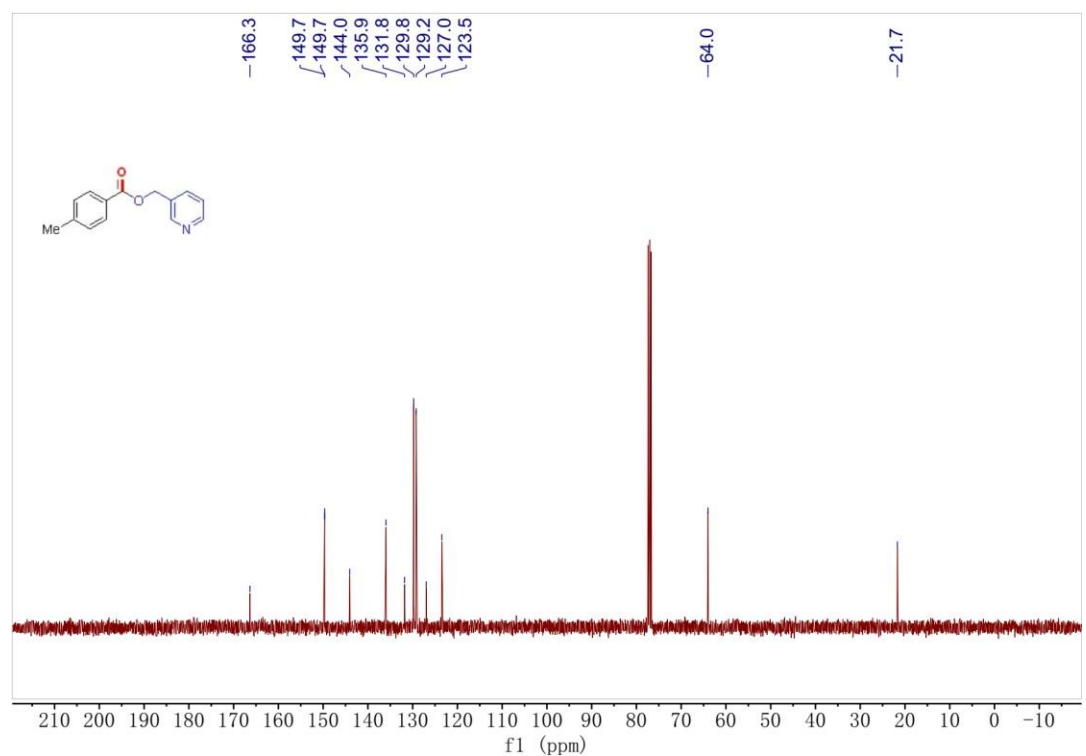
¹³C NMR (101 MHz, CDCl₃) 39



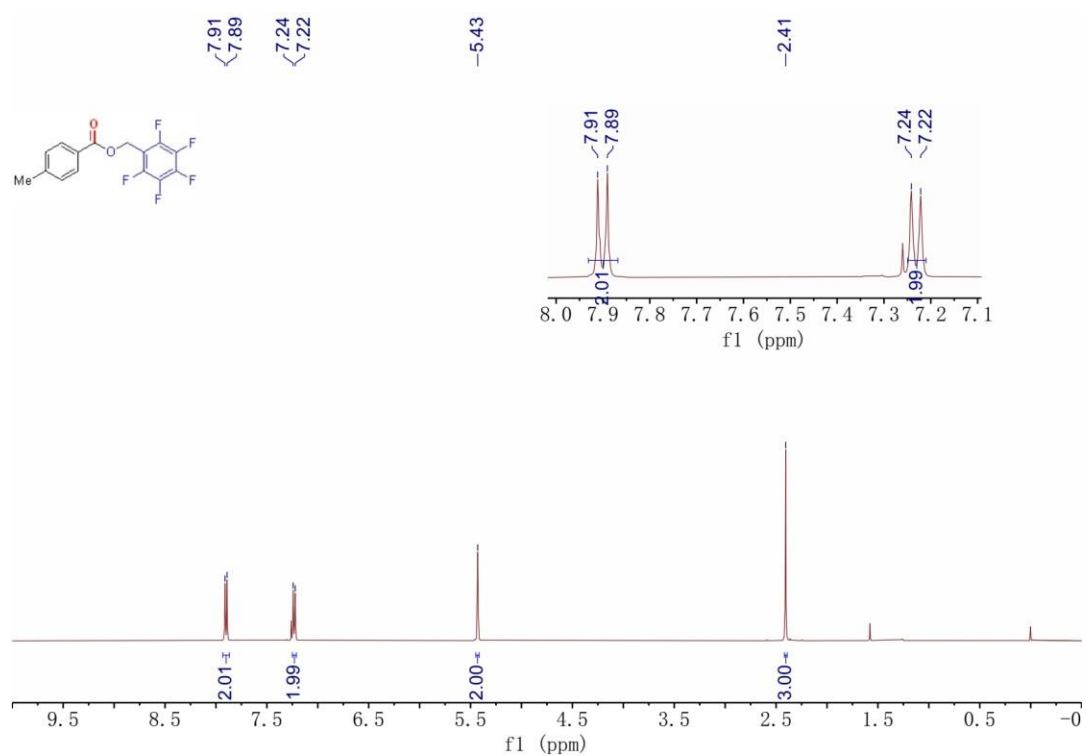
¹H NMR (400 MHz, CDCl₃) 40



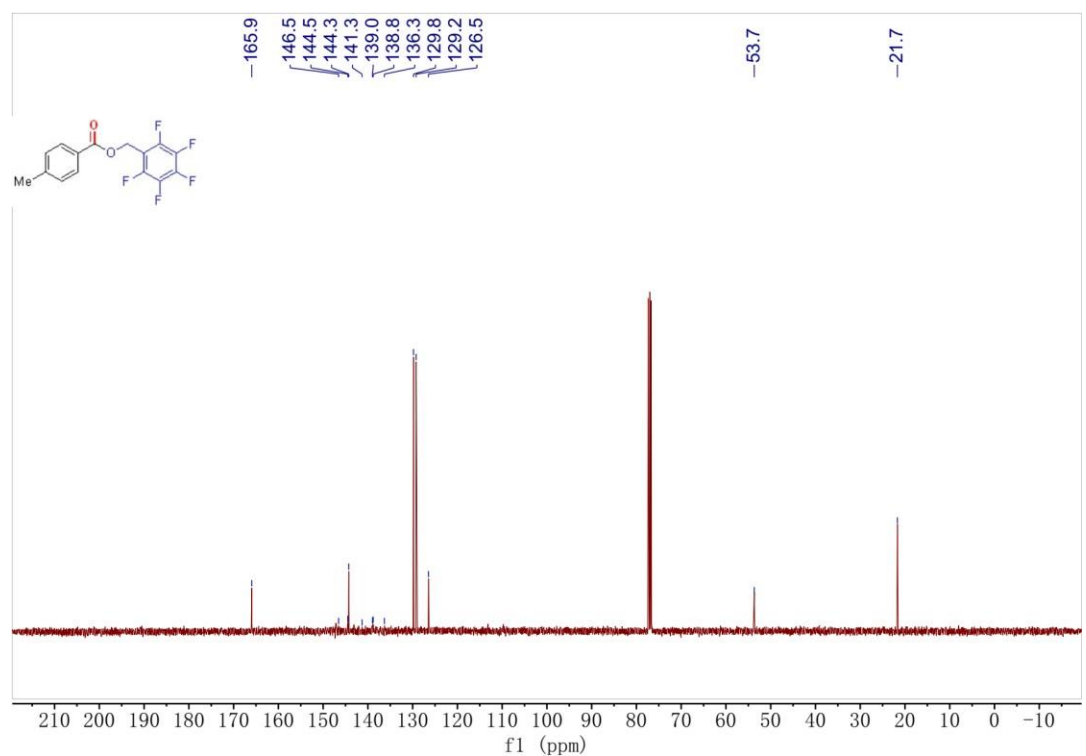
¹³C NMR (101 MHz, CDCl₃) 40



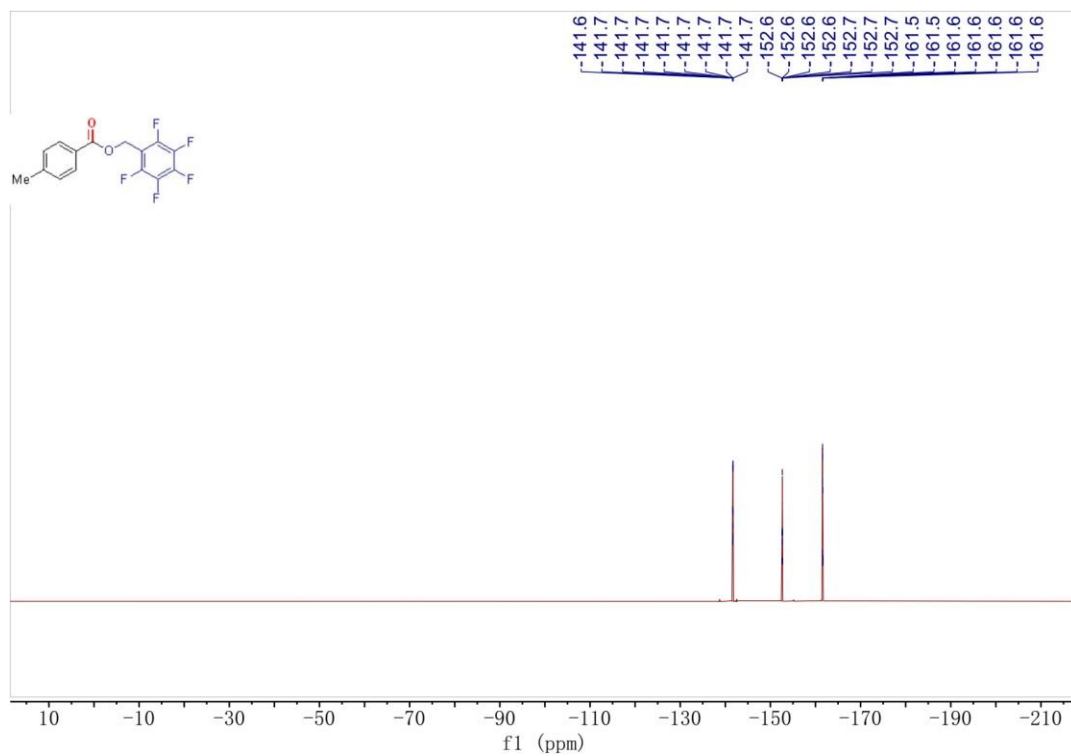
¹H NMR (400 MHz, CDCl₃) 41



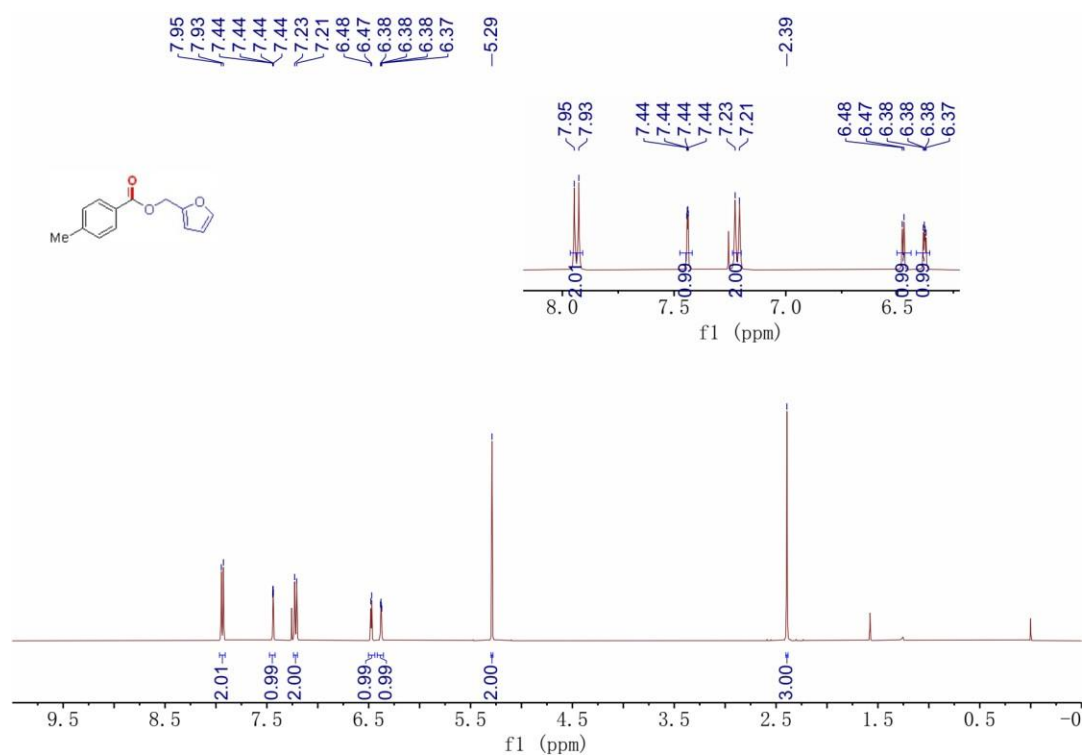
¹³C NMR (101 MHz, CDCl₃) 41



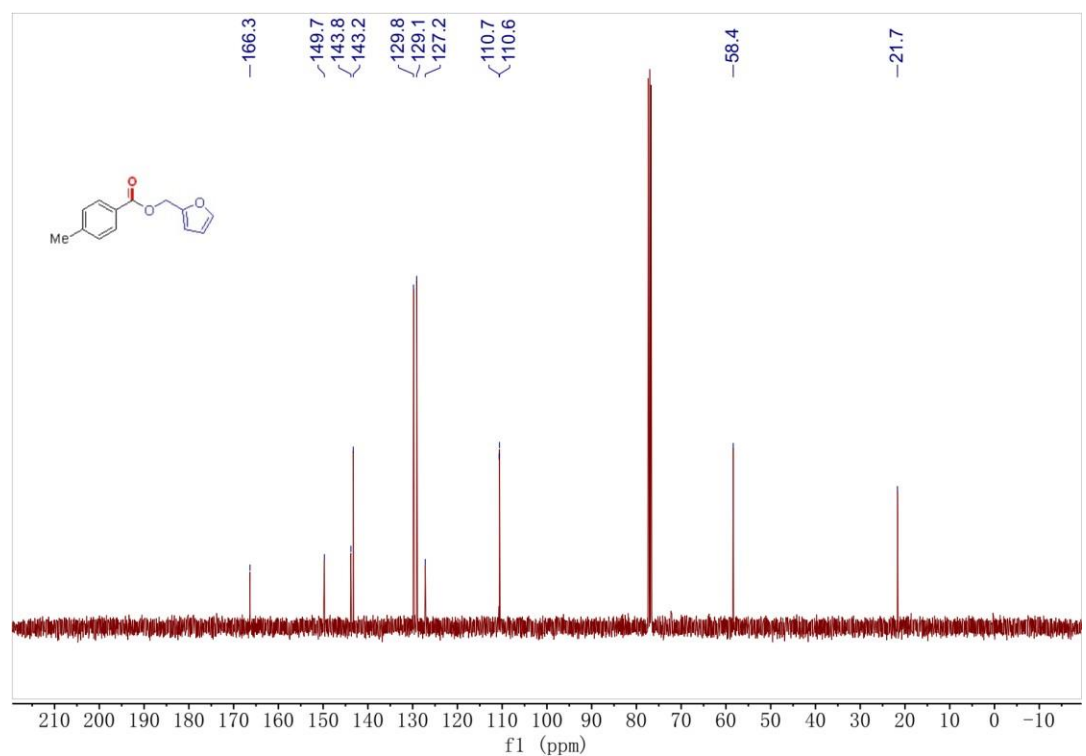
¹⁹F NMR (376 MHz, CDCl₃) 41



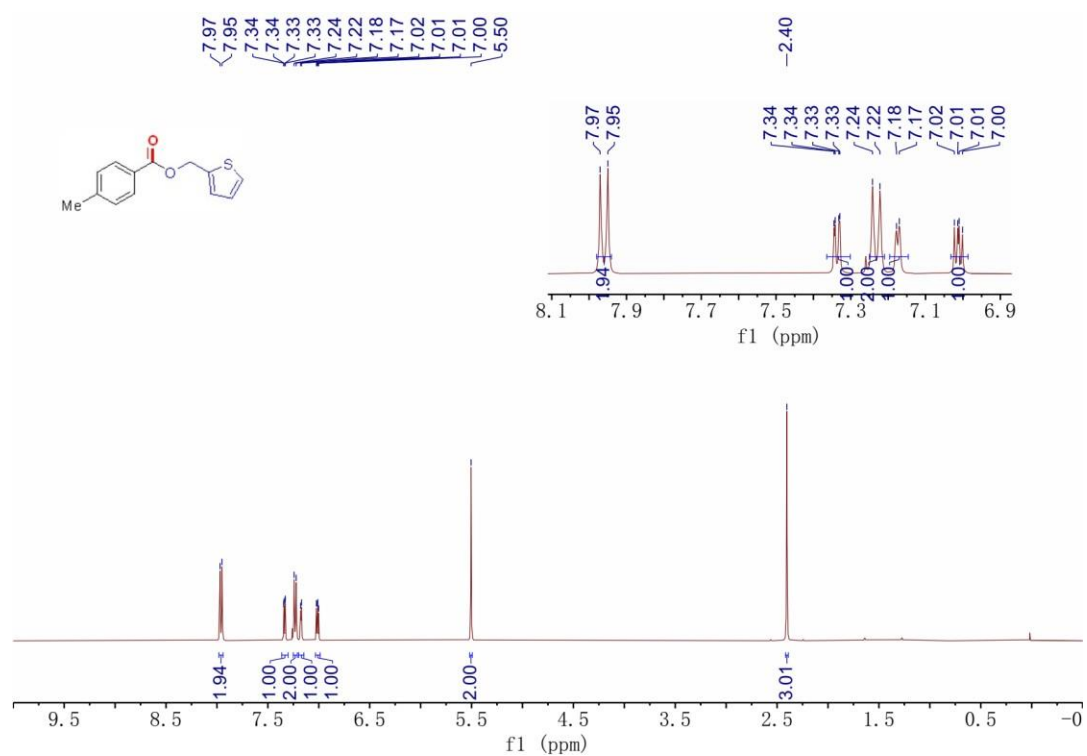
¹H NMR (400 MHz, CDCl₃) 480



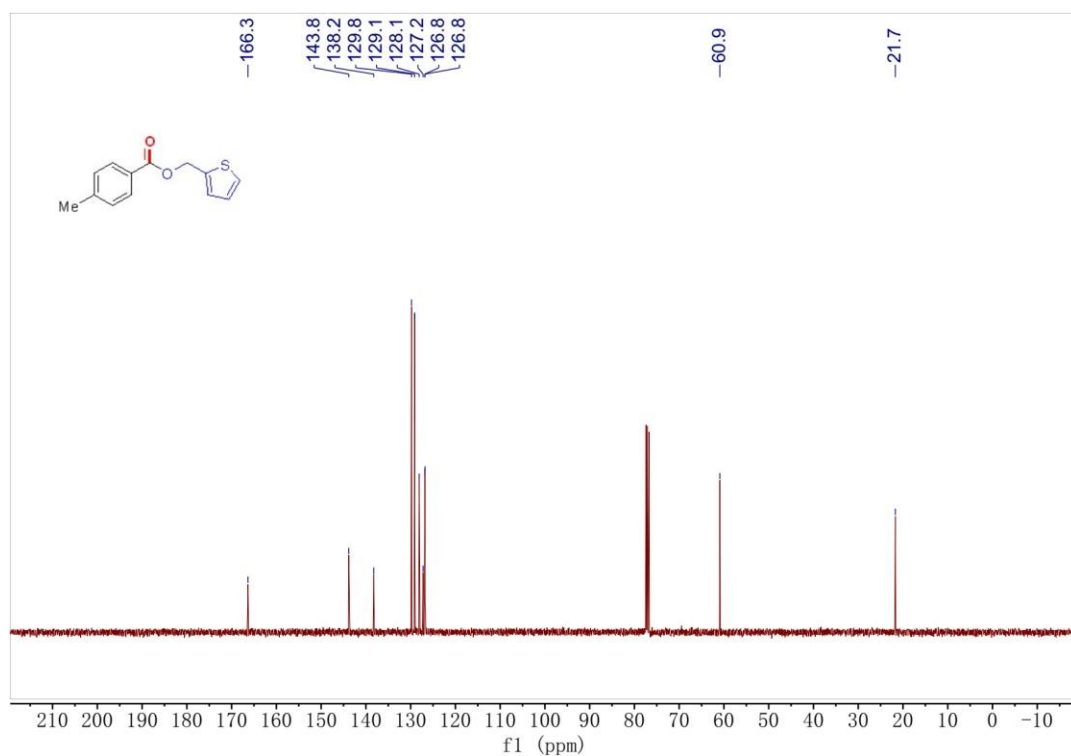
¹³C NMR (101 MHz, CDCl₃) 42



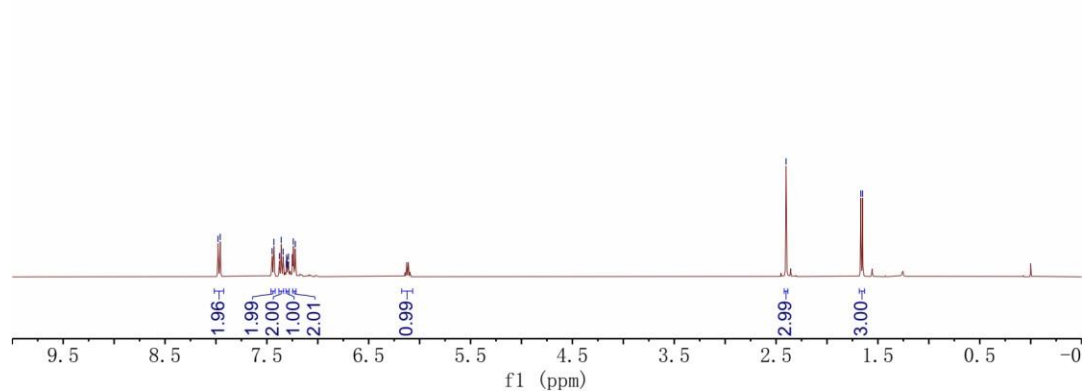
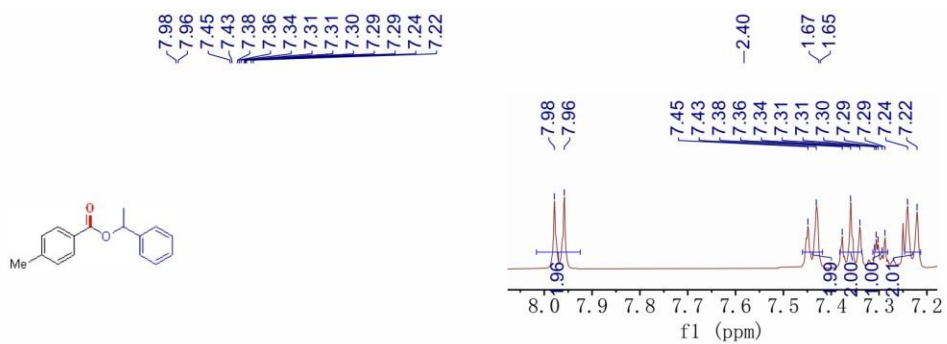
¹H NMR (400 MHz, CDCl₃) 481



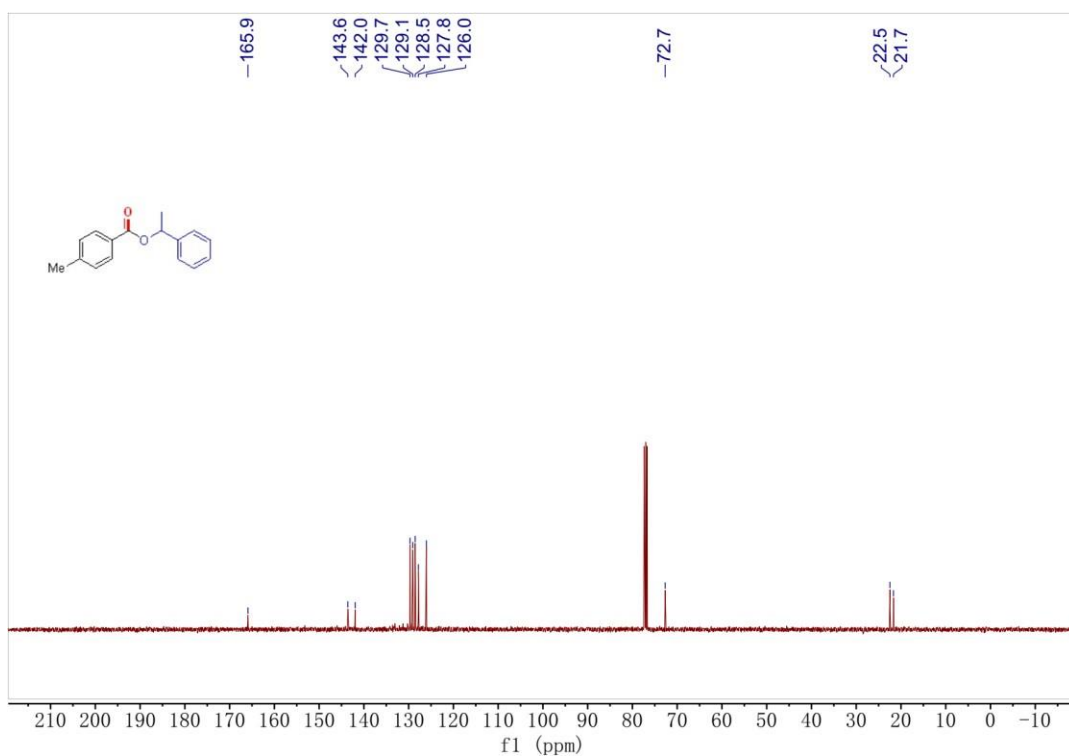
¹³C NMR (101 MHz, CDCl₃) 43



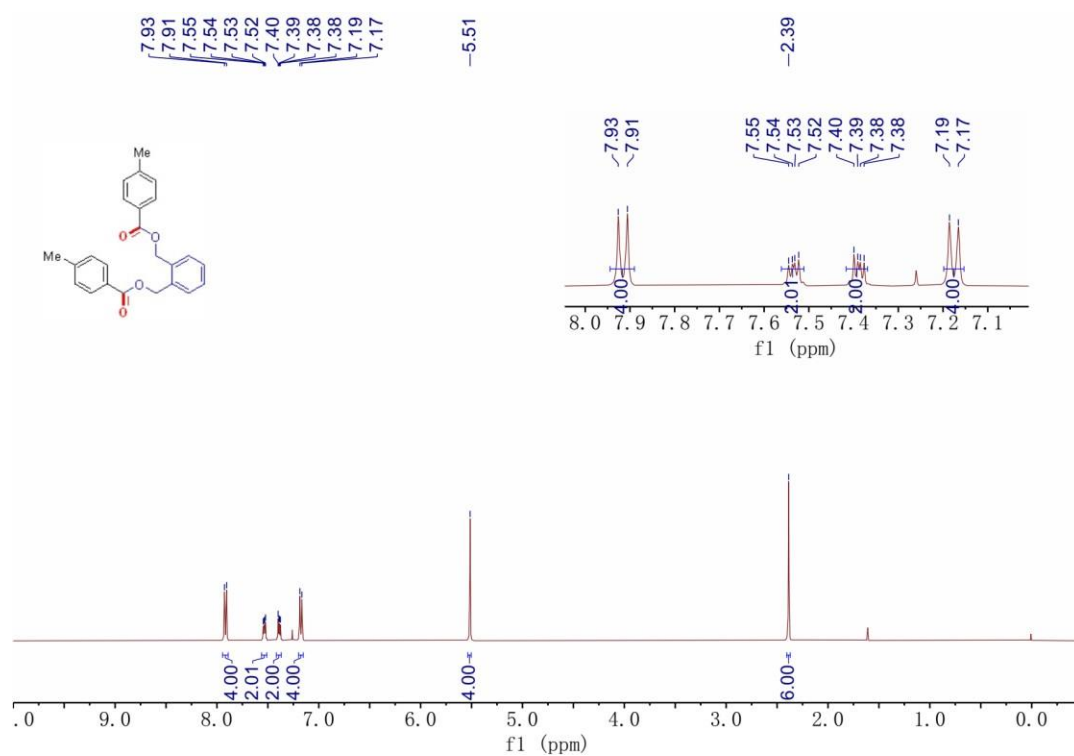
¹H NMR (400 MHz, CDCl₃) 482



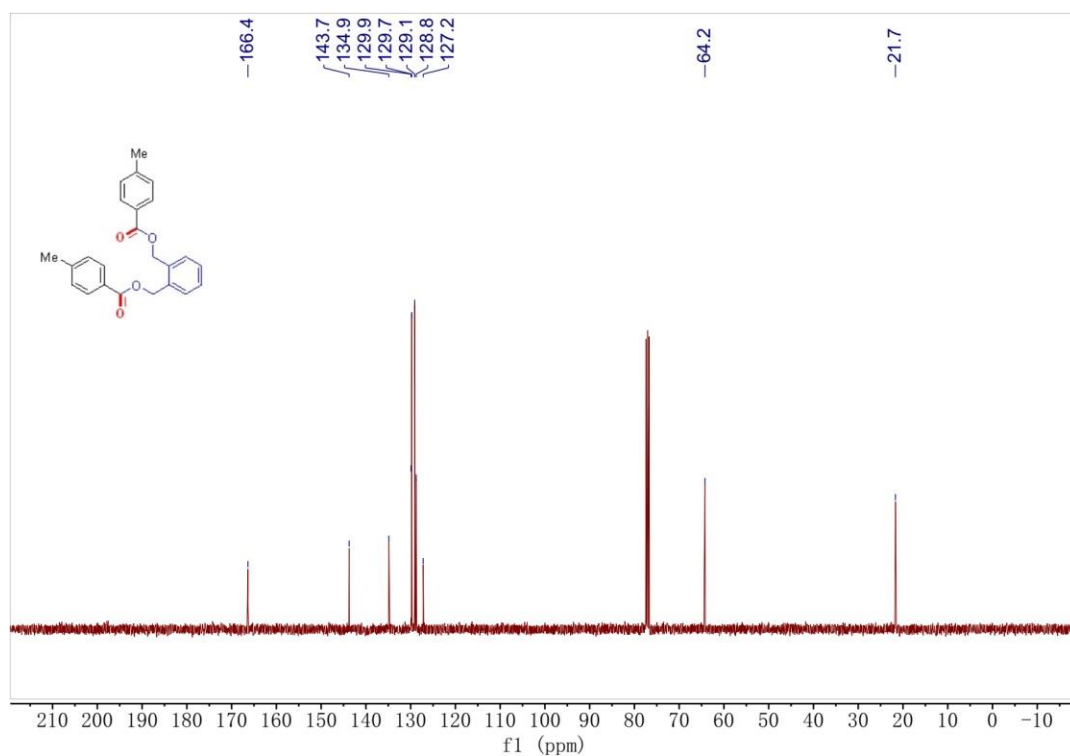
¹³C NMR (101 MHz, CDCl₃) 44



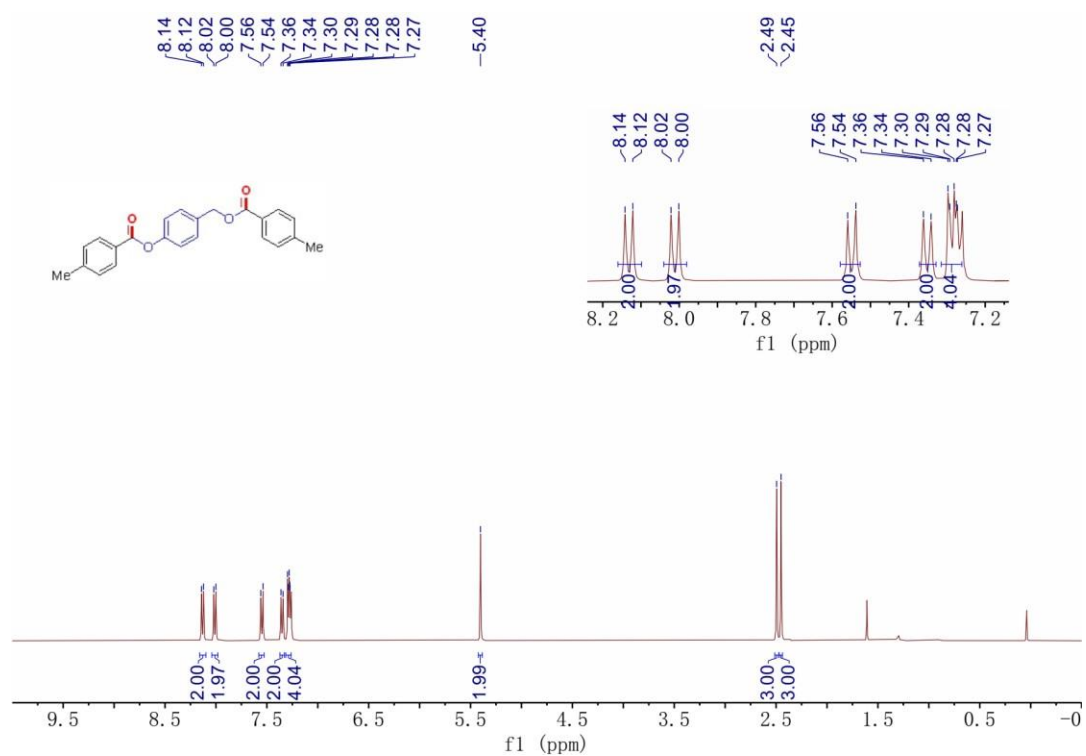
¹H NMR (400 MHz, CDCl₃) 483



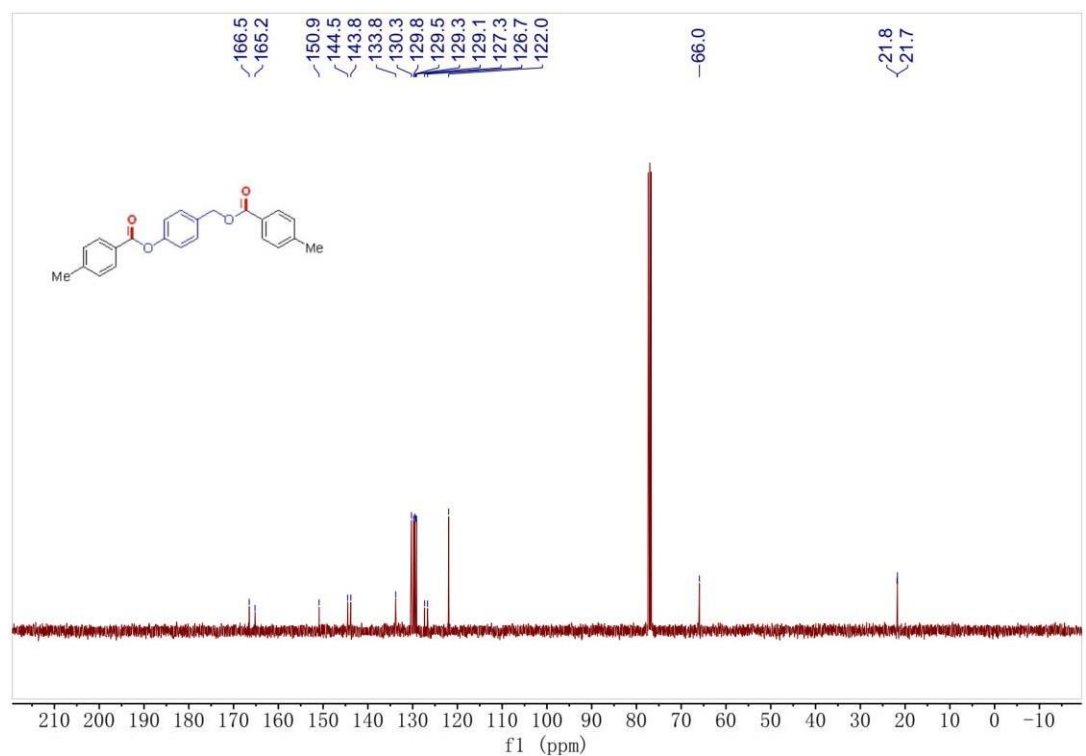
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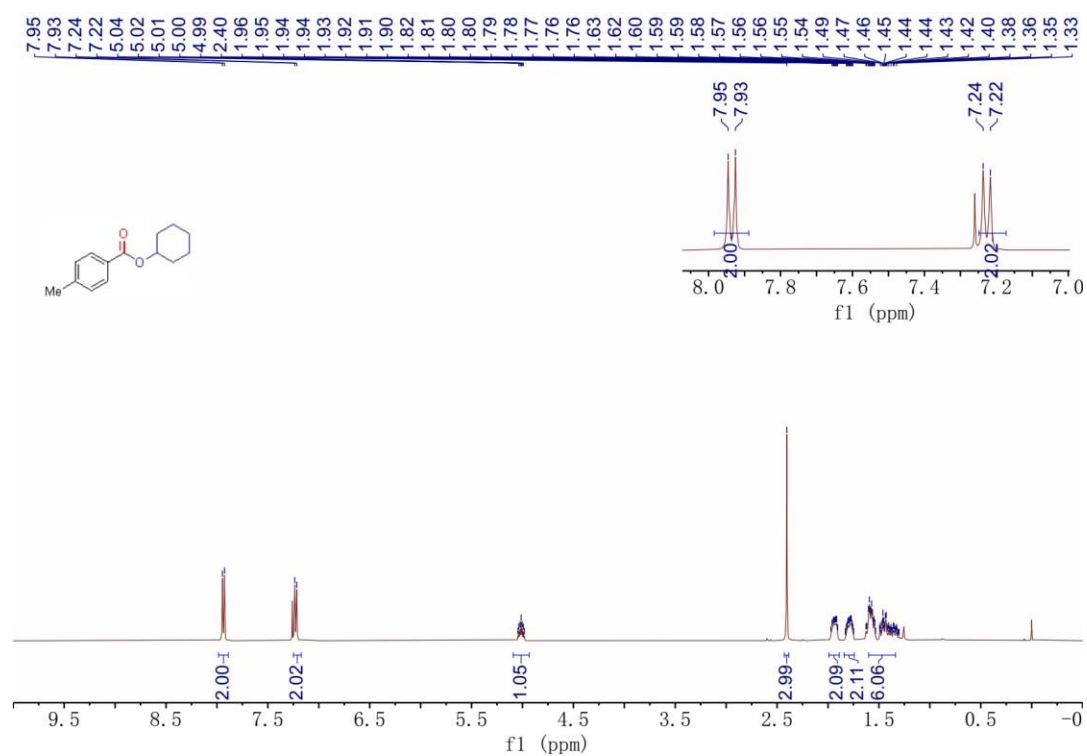
¹H NMR (400 MHz, CDCl₃) 484



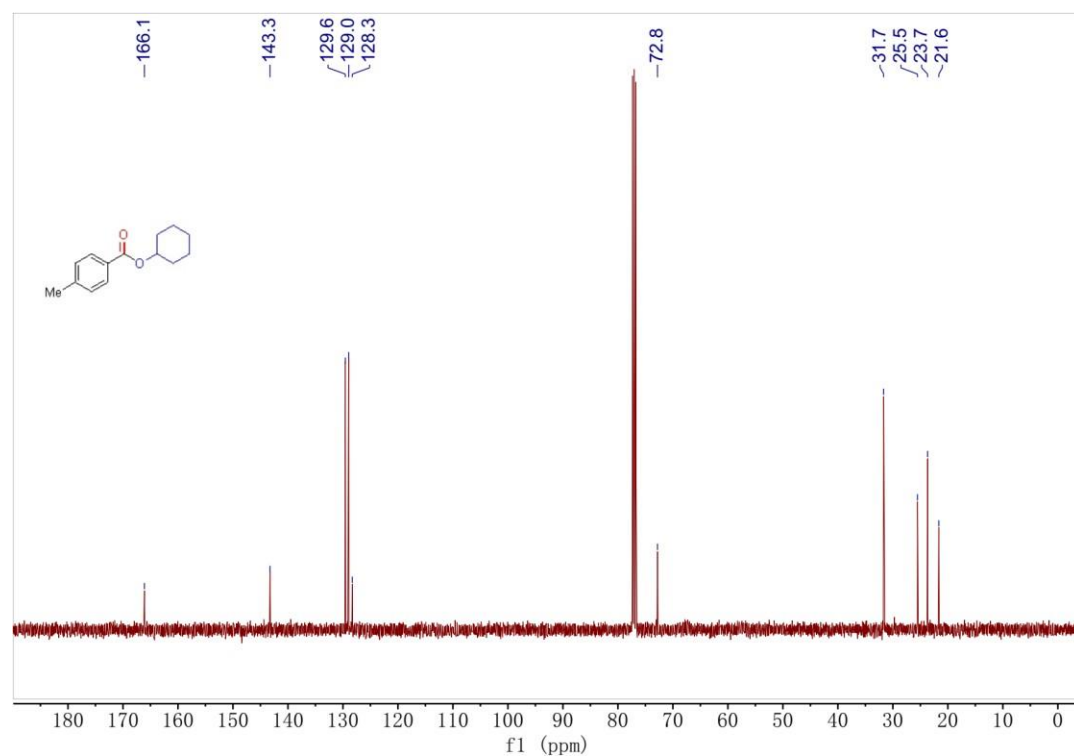
¹³C NMR (101 MHz, CDCl₃) 46



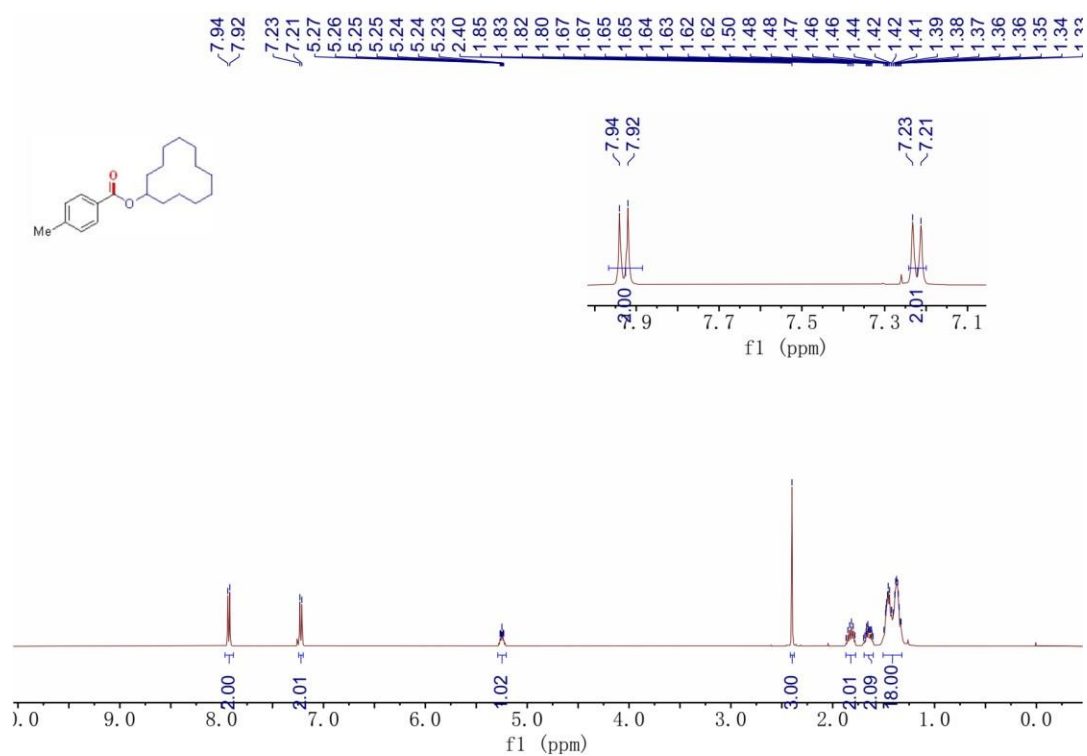
¹H NMR (400 MHz, CDCl₃) 485



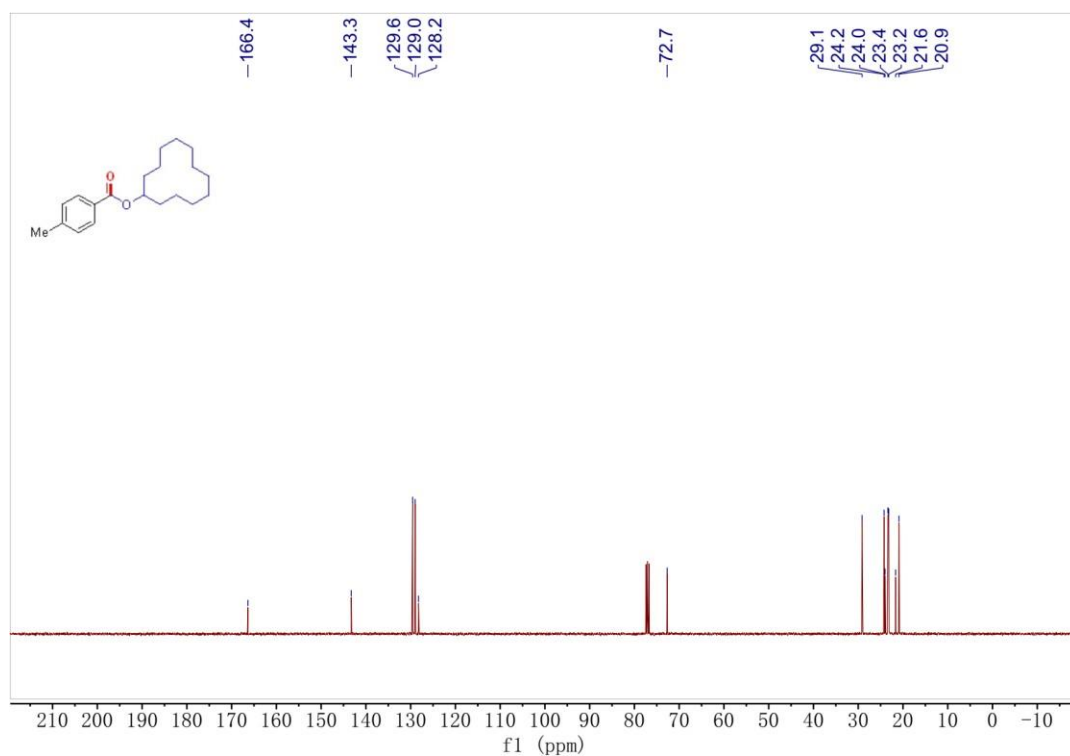
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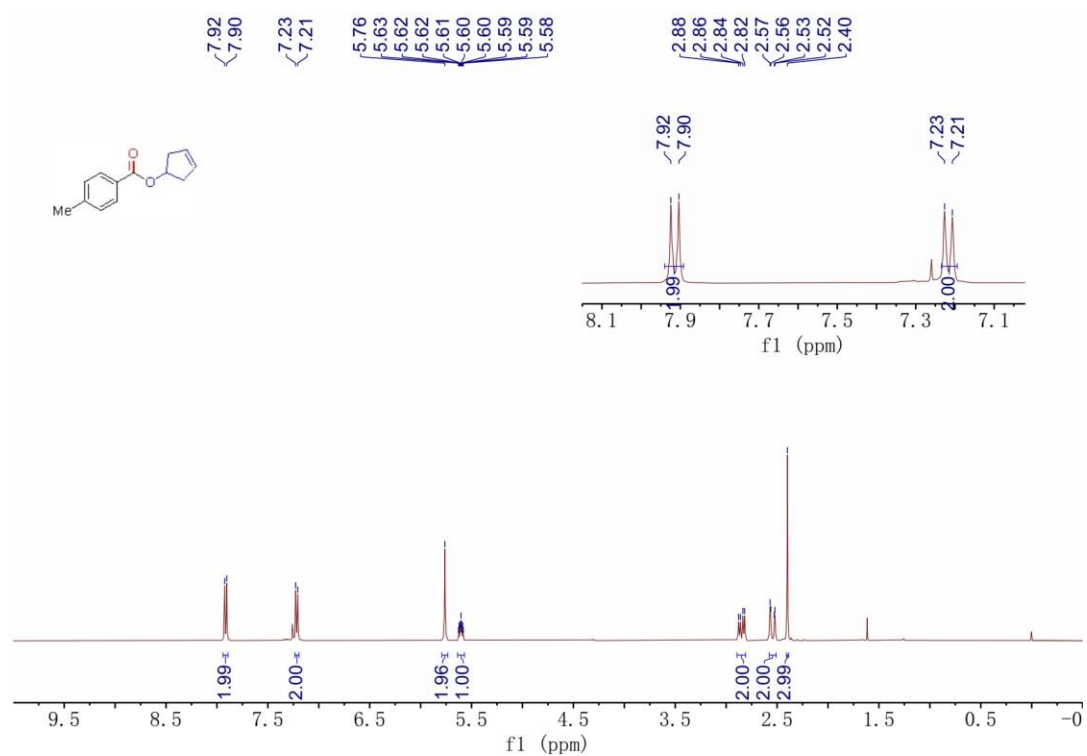
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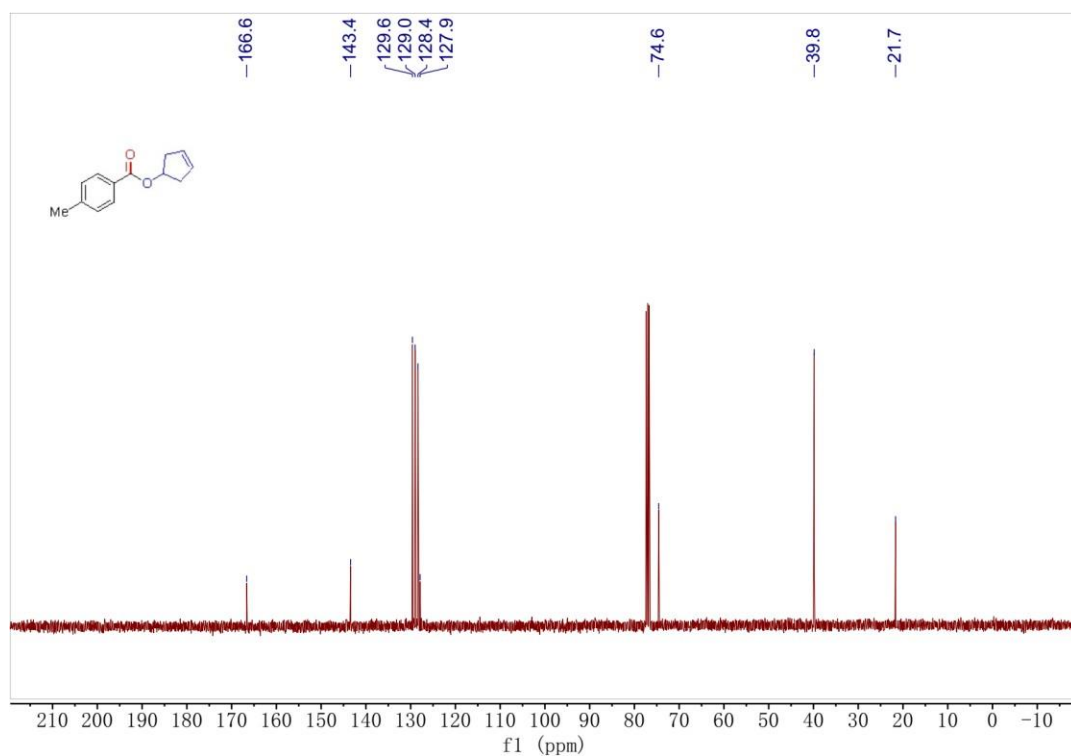
¹³C NMR (101 MHz, CDCl₃) 48



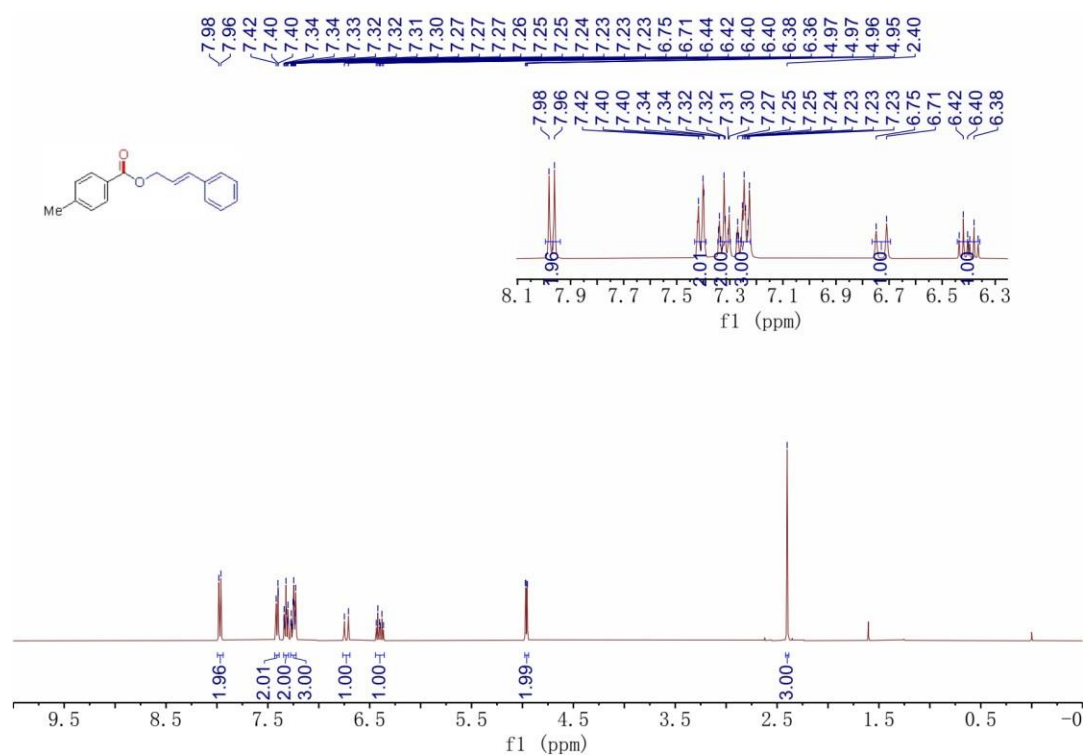
¹H NMR (400 MHz, CDCl₃) 487



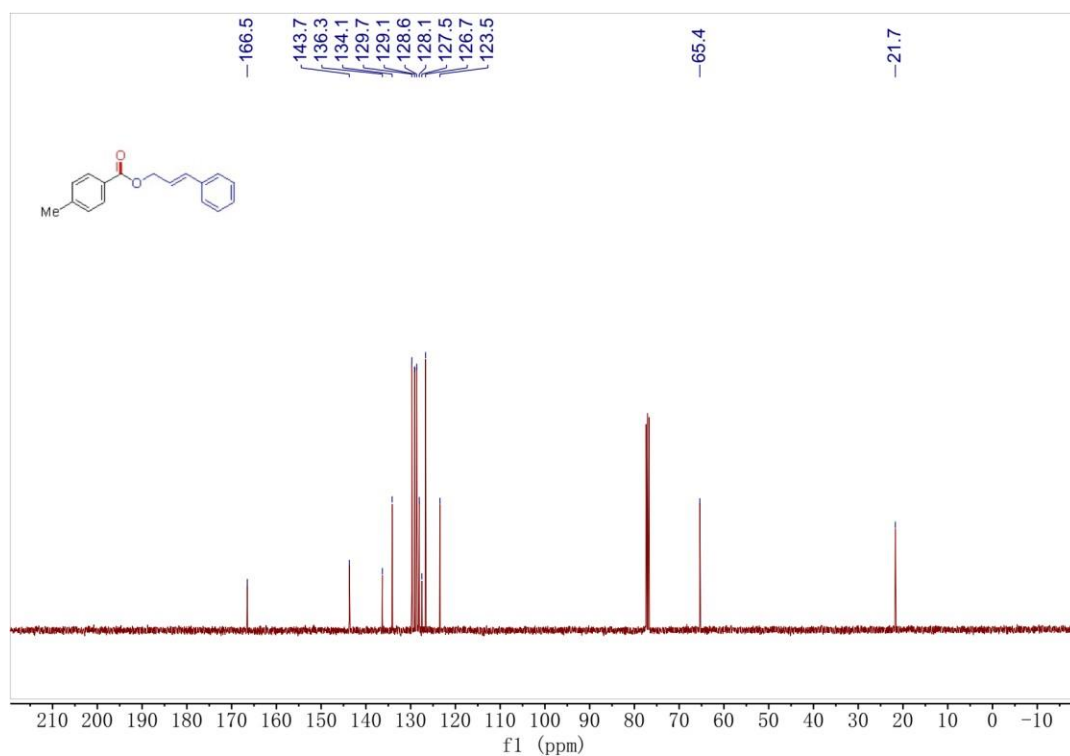
¹³C NMR (101 MHz, CDCl₃) 49



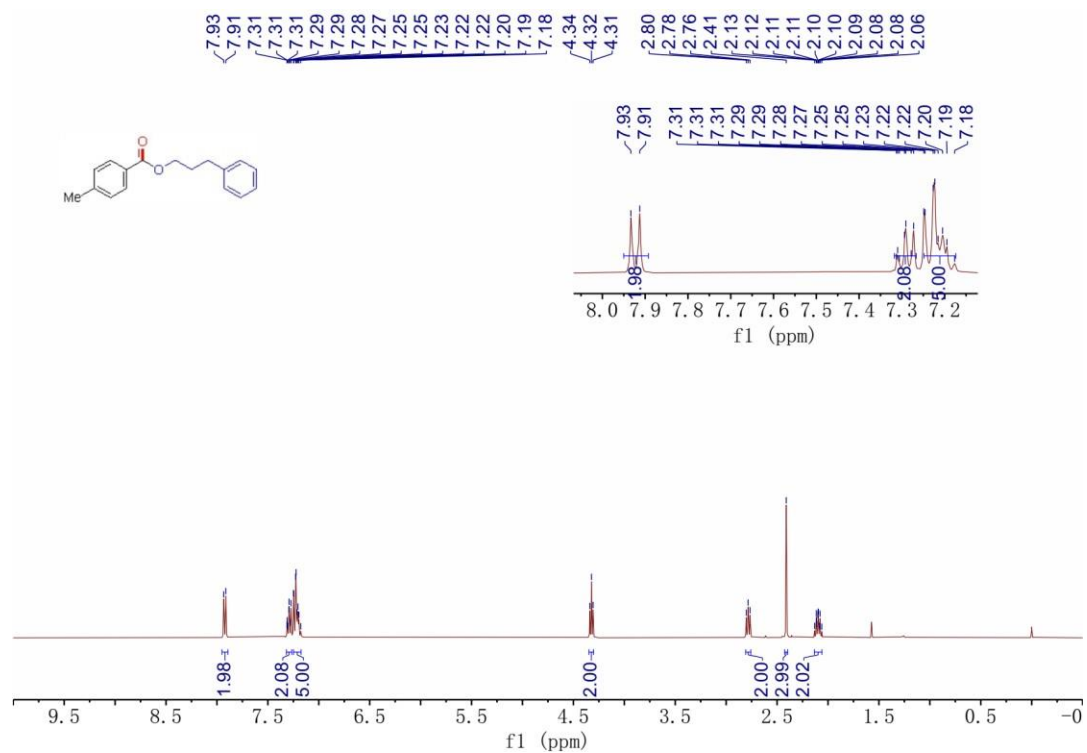
¹H NMR (400 MHz, CDCl₃) 50



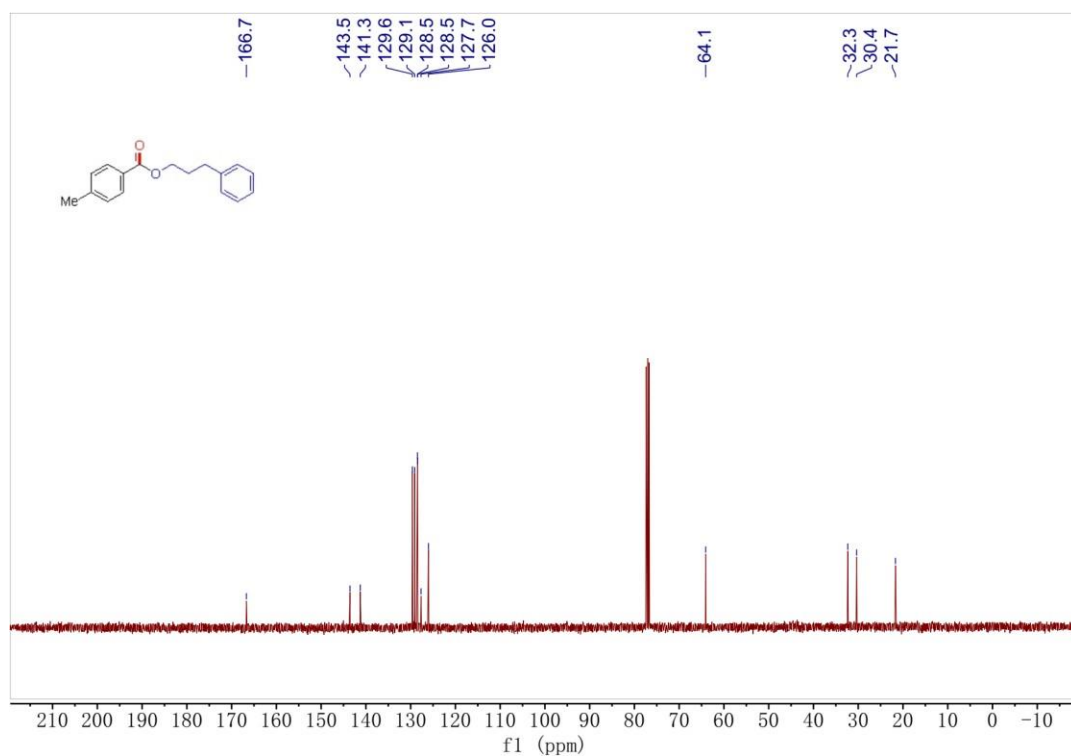
¹³C NMR (101 MHz, CDCl₃) 50



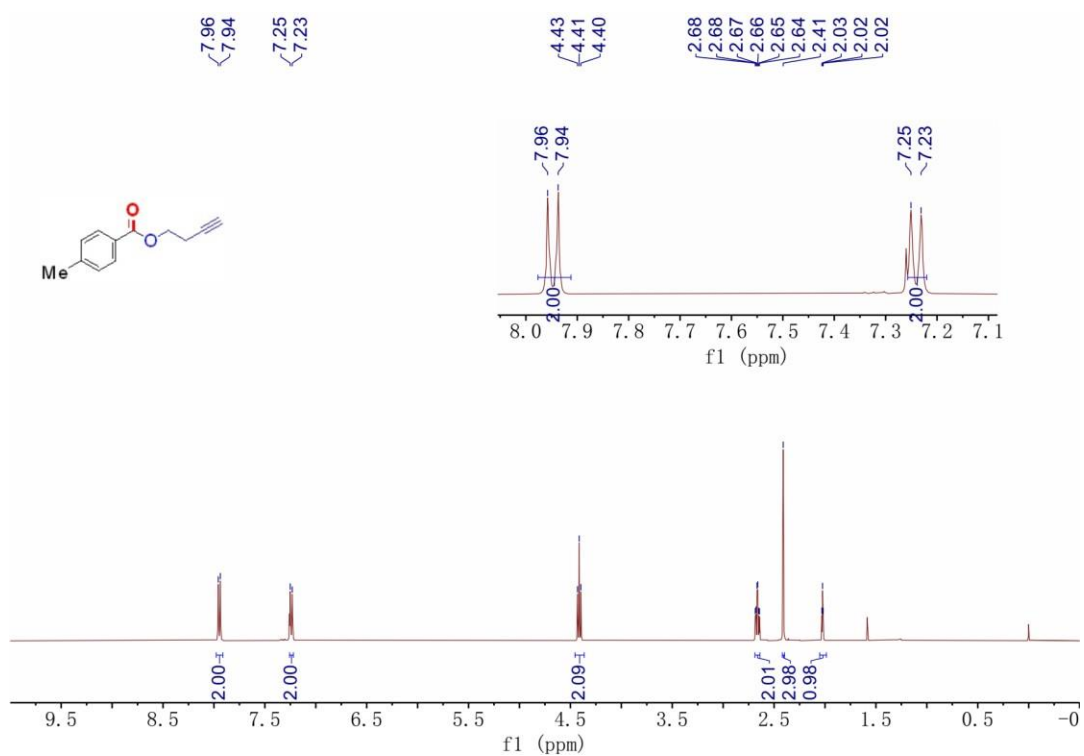
¹H NMR (400 MHz, CDCl₃) 589



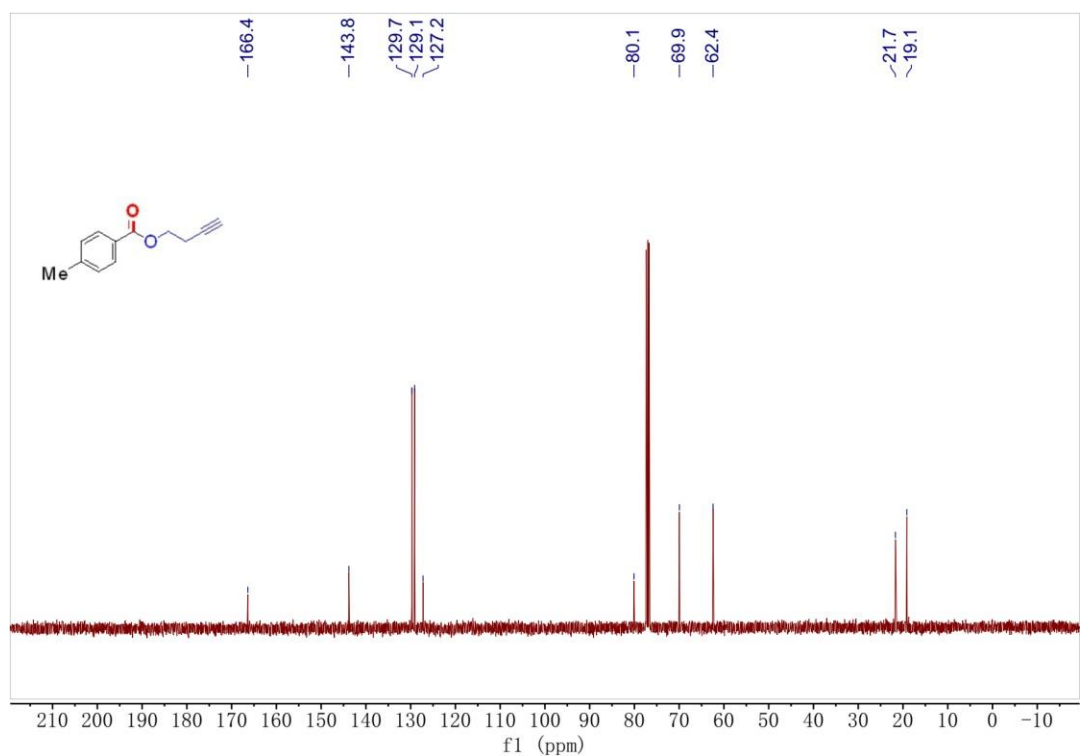
¹³C NMR (101 MHz, CDCl₃) 51



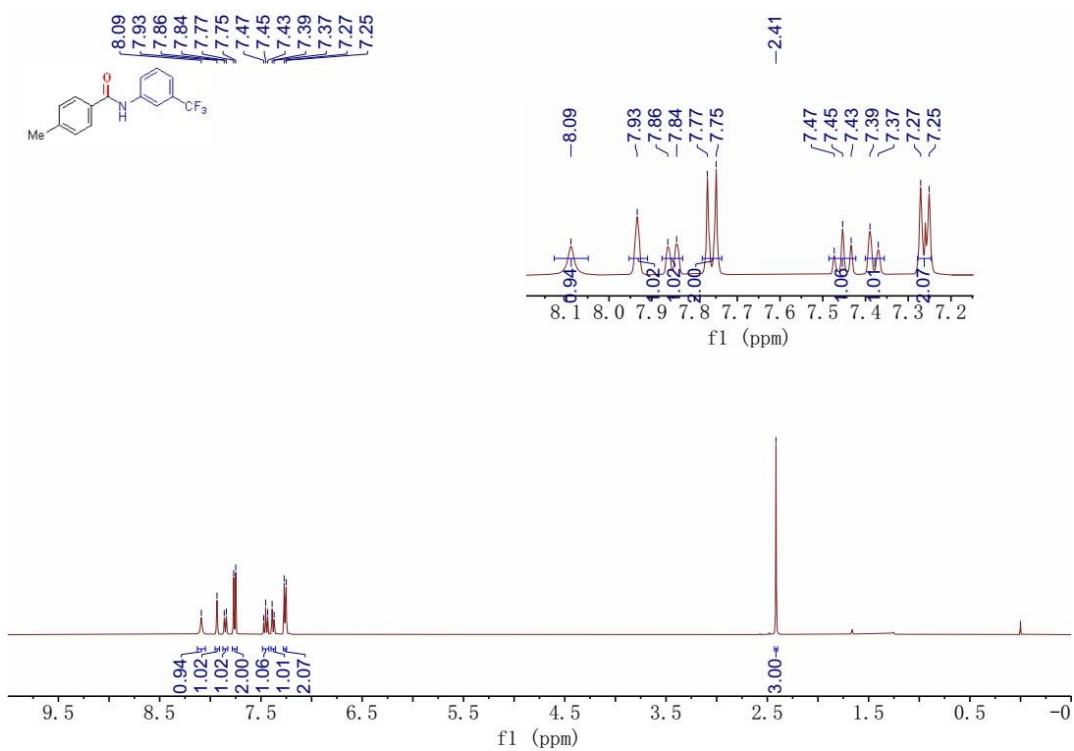
¹H NMR (400 MHz, CDCl₃) 590



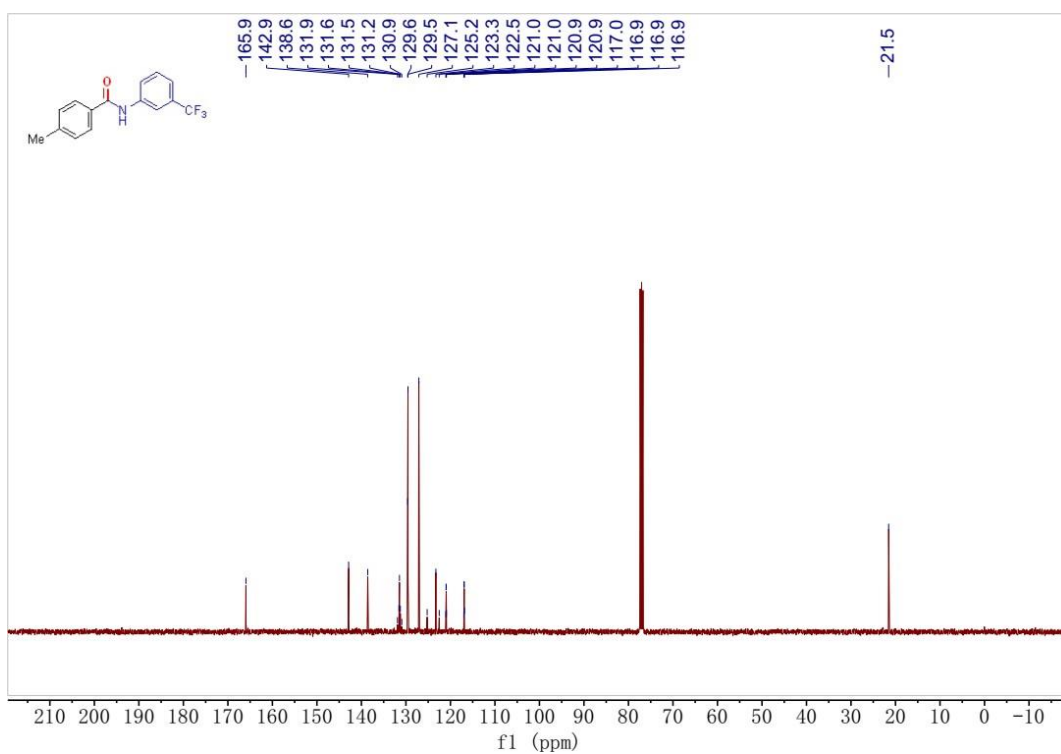
¹³C NMR (101 MHz, CDCl₃) 52



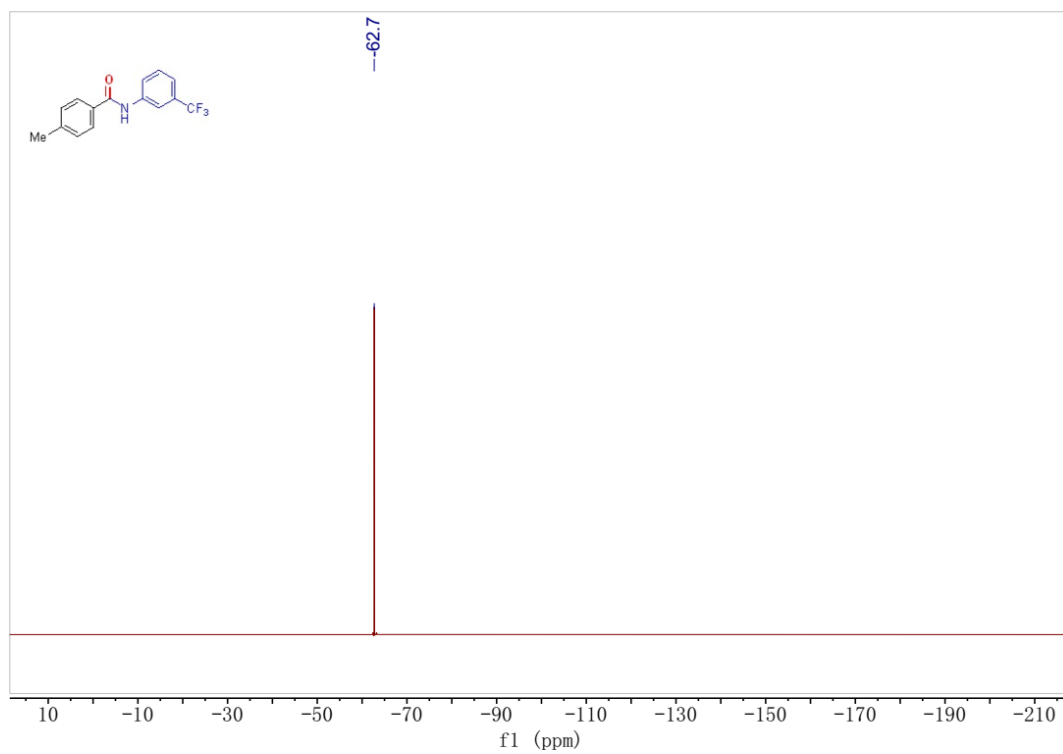
¹H NMR (400 MHz, CDCl₃) 591



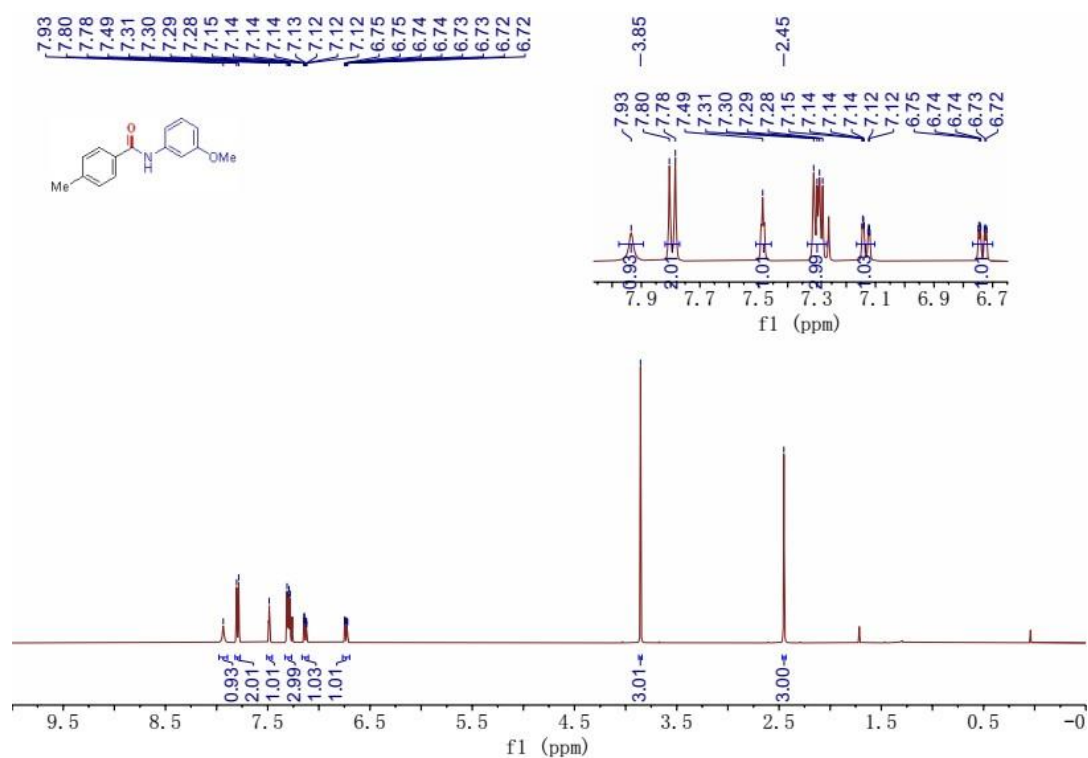
¹³C NMR (101 MHz, CDCl₃) 53



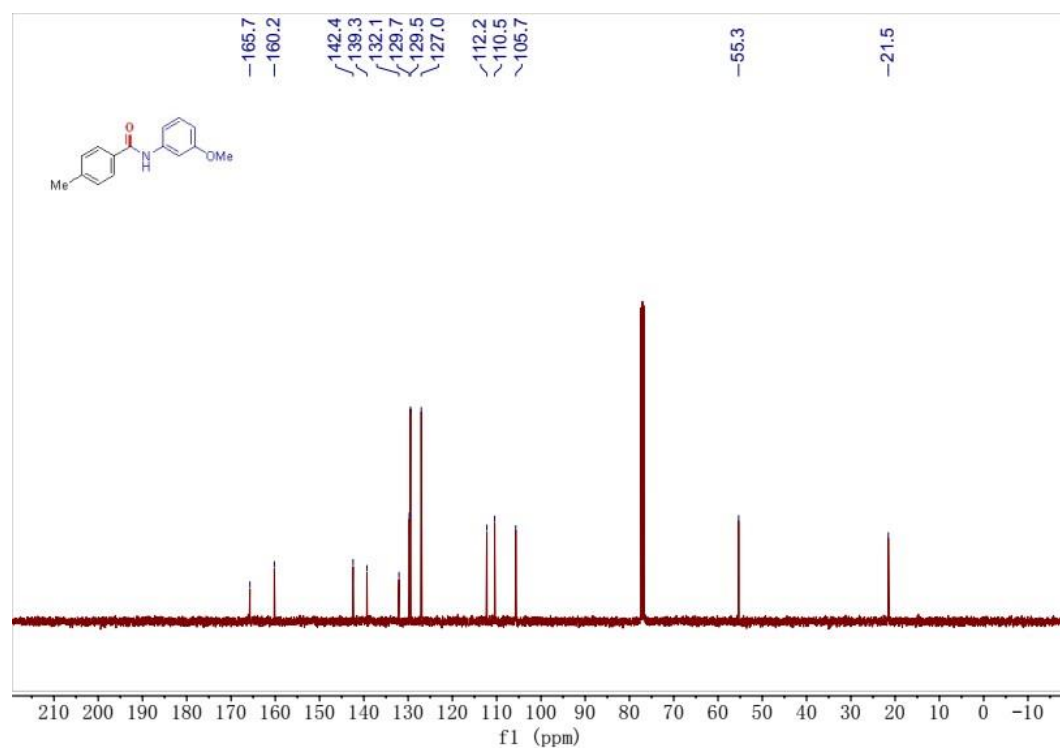
¹⁹F NMR (376 MHz, CDCl₃) 53



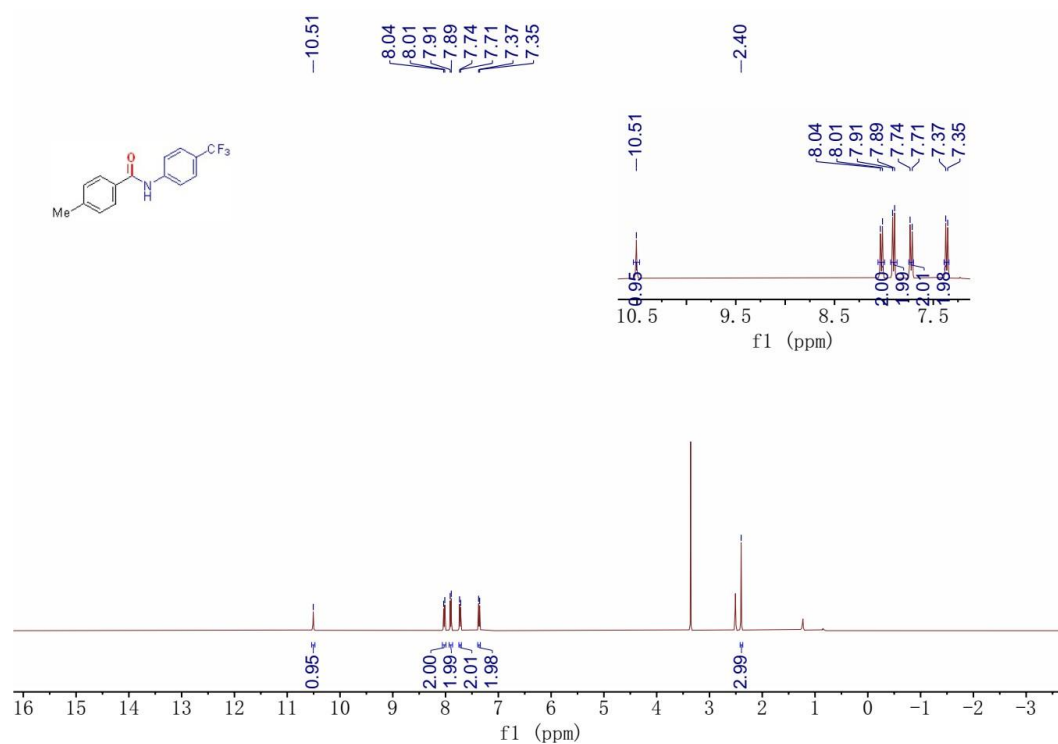
¹H NMR (400 MHz, CDCl₃) 54



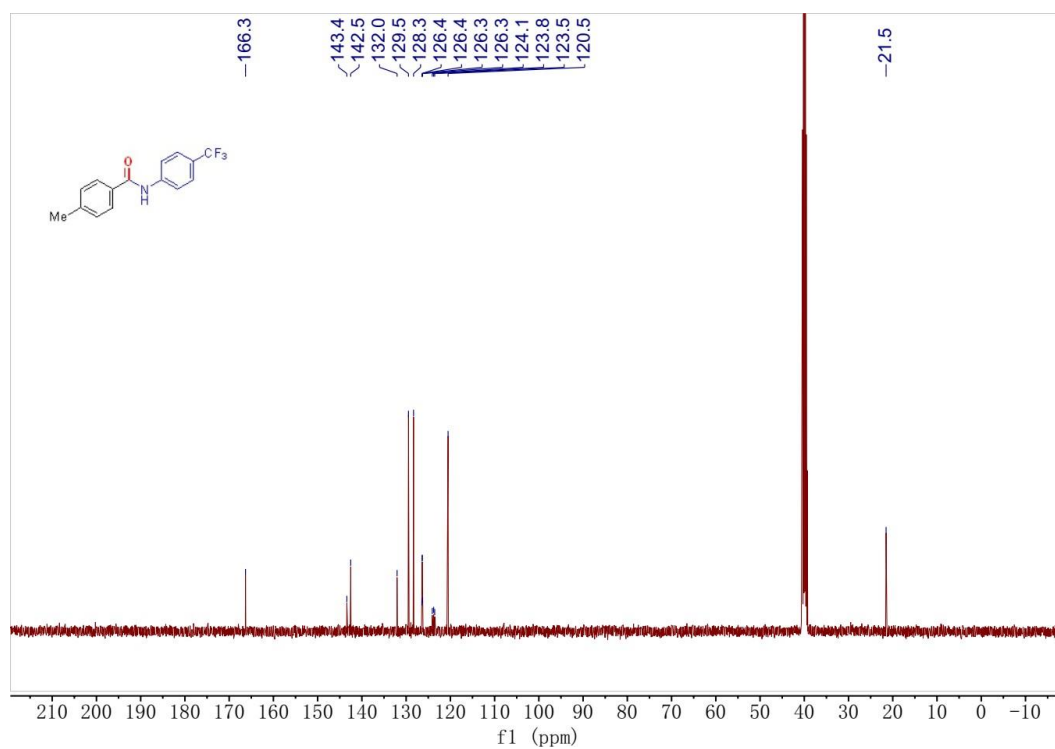
¹³C NMR (101 MHz, CDCl₃) 54



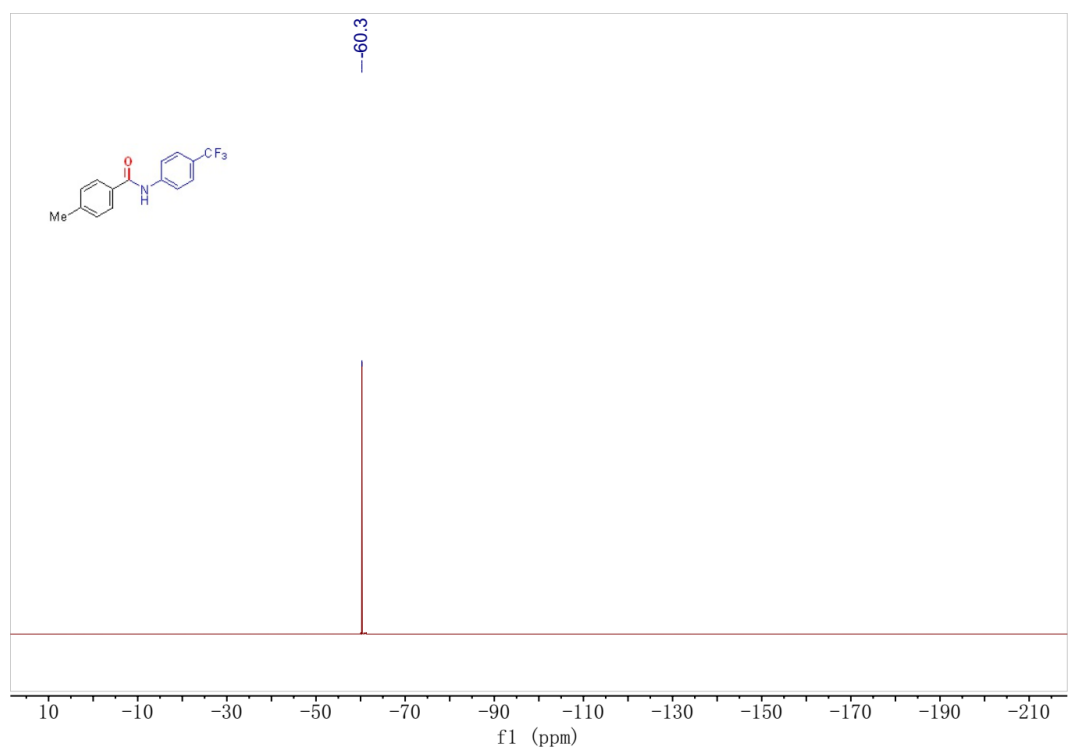
¹H NMR (400 MHz, DMSO-*d*₆) 55



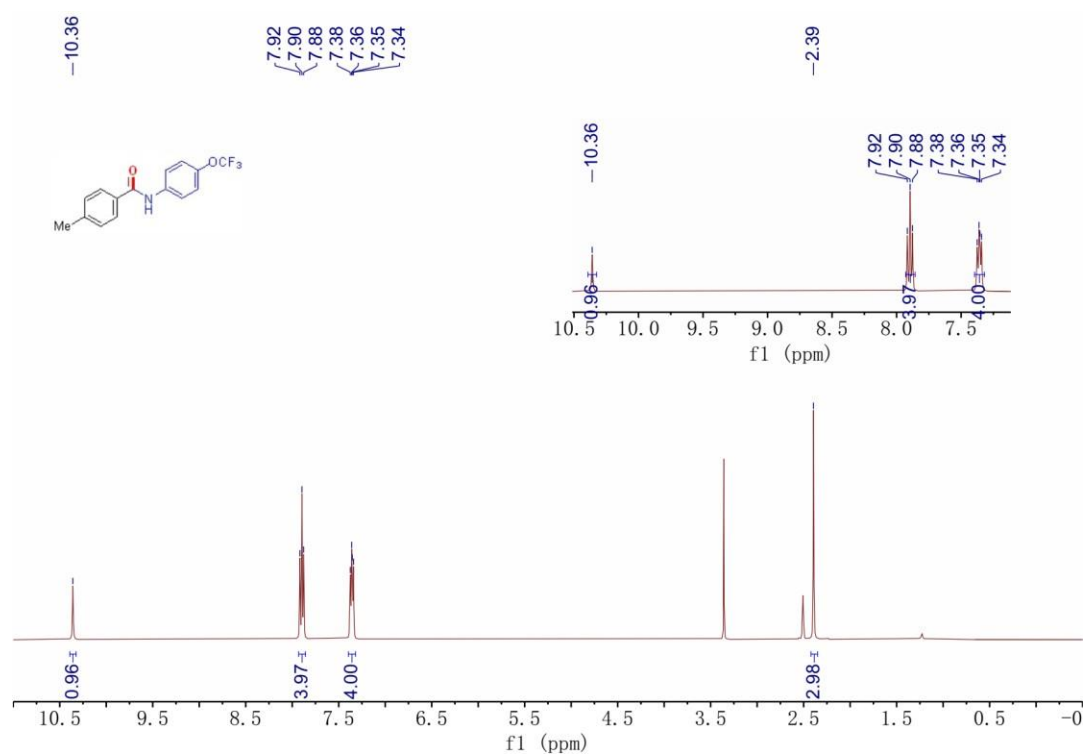
¹³C NMR (101 MHz, DMSO) 55



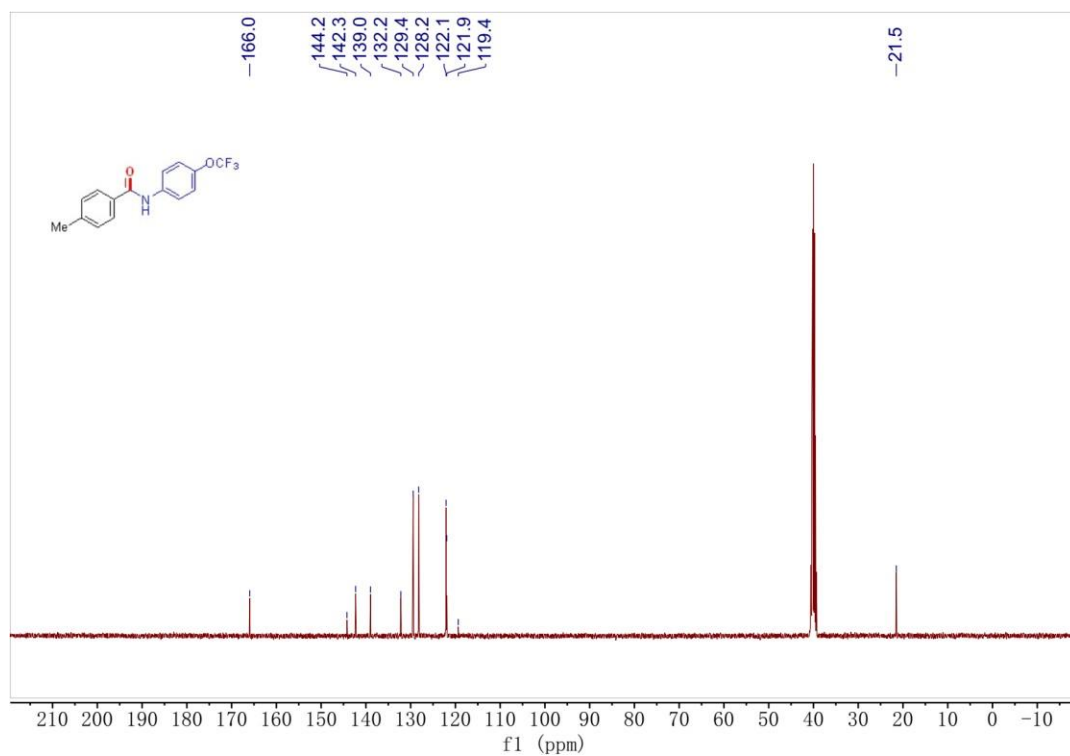
¹⁹F NMR (376 MHz, DMSO-*d*₆) 55



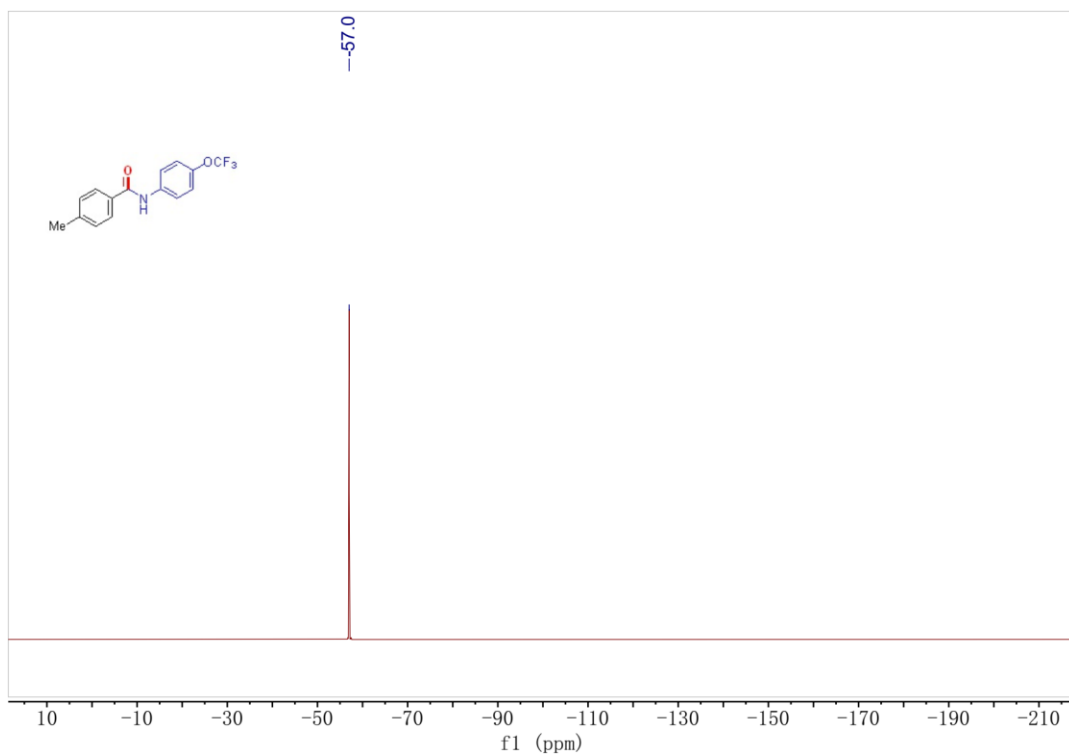
¹H NMR (400 MHz, DMSO-*d*₆) 56



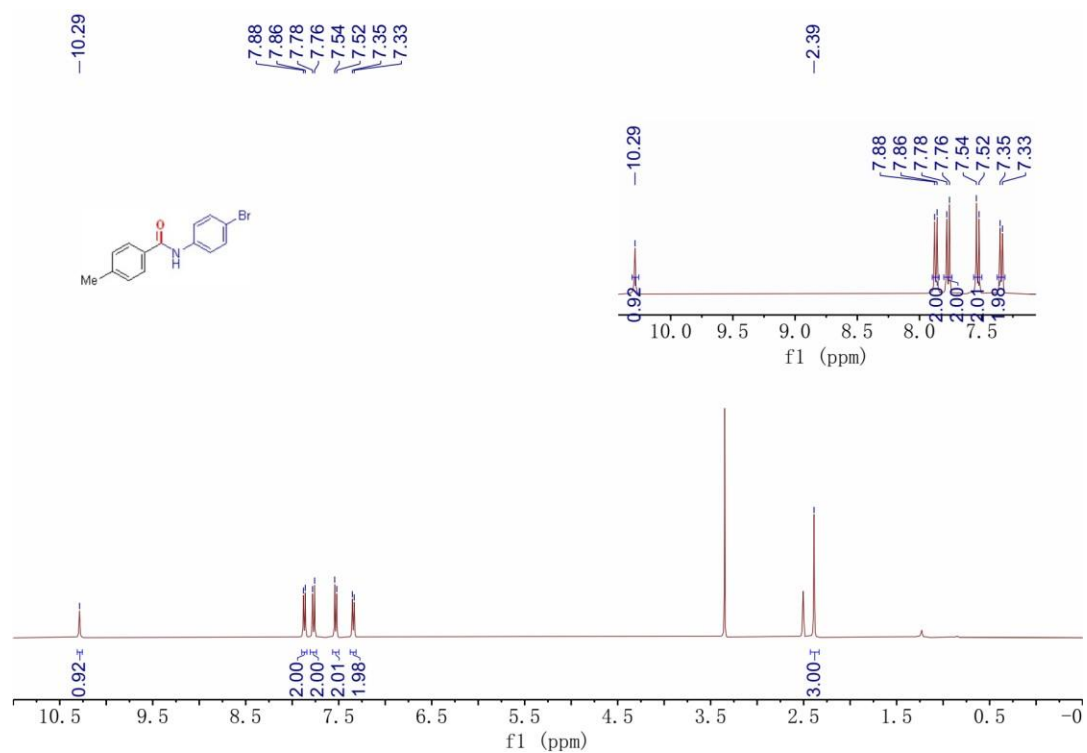
¹³C NMR (101 MHz, DMSO) 56



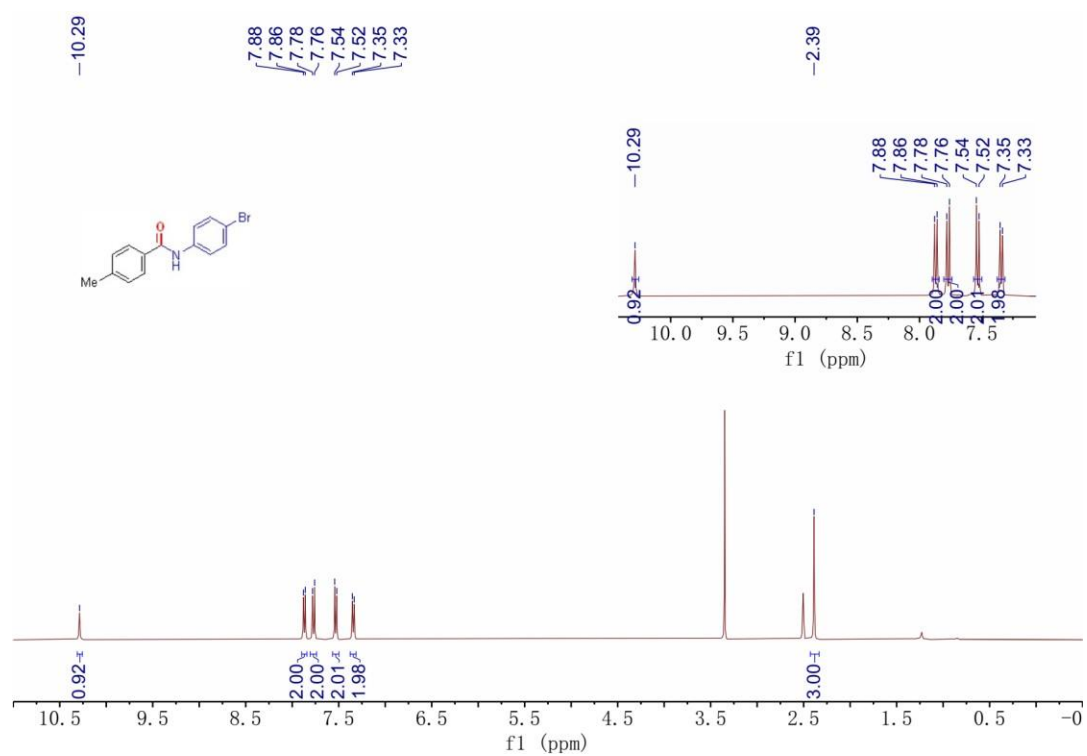
¹⁹F NMR (376 MHz, DMSO-*d*₆) 56



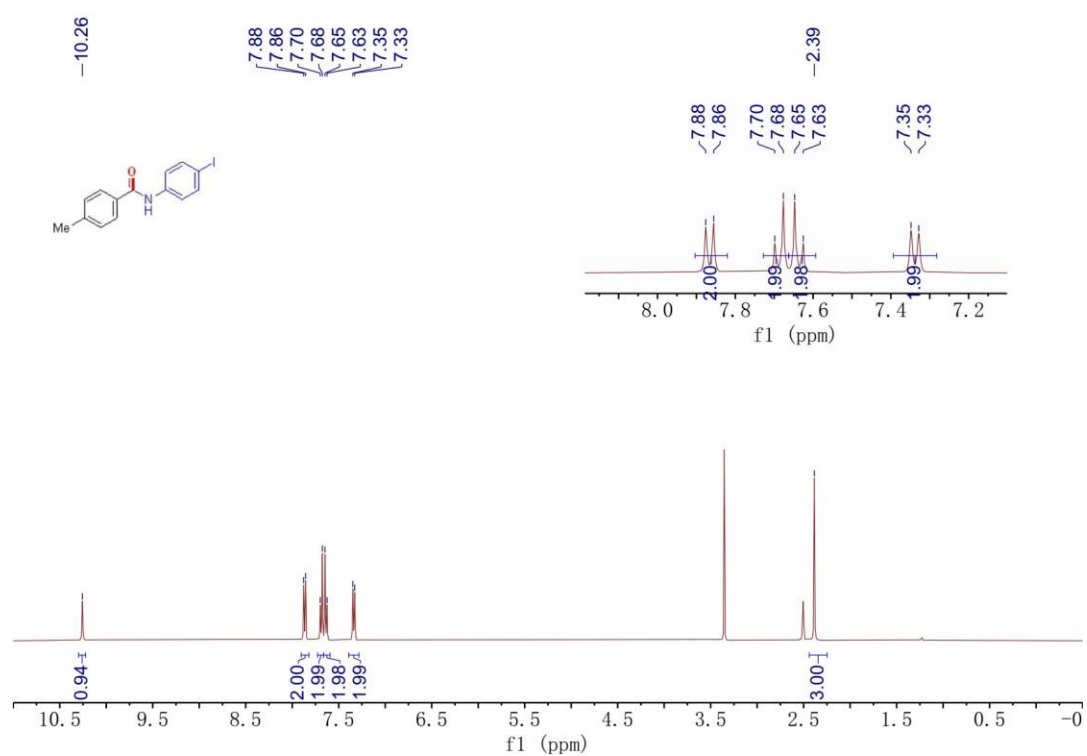
¹H NMR (400 MHz, DMSO-*d*₆) 598



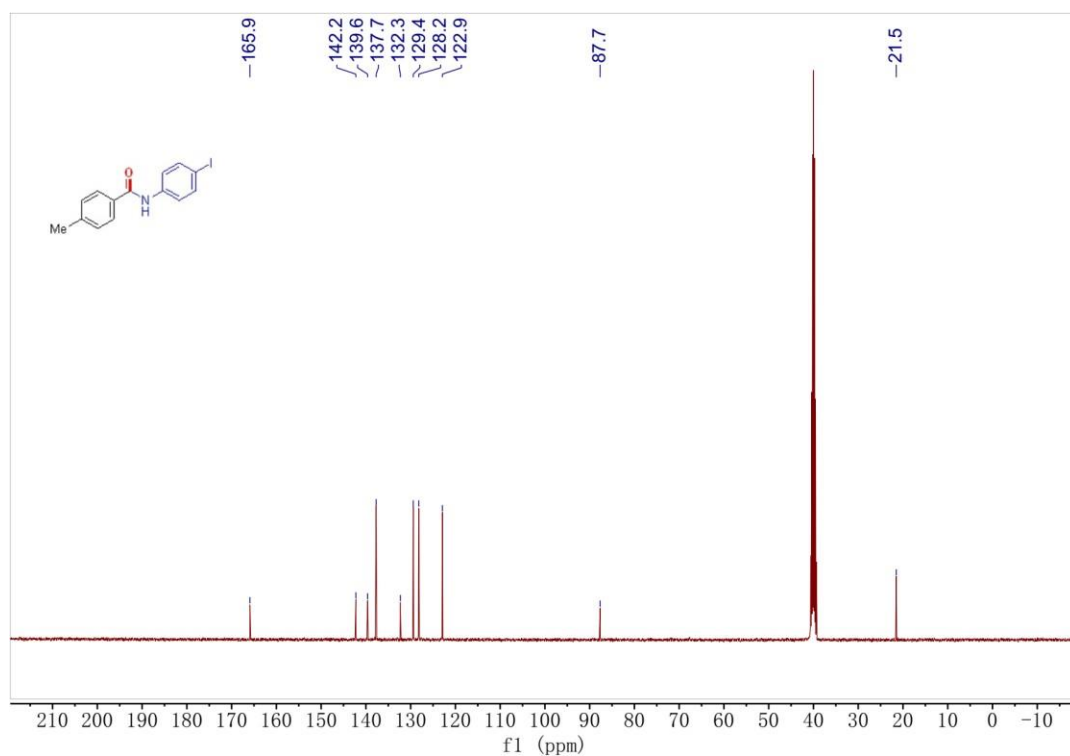
¹³C NMR (101 MHz, DMSO) 57



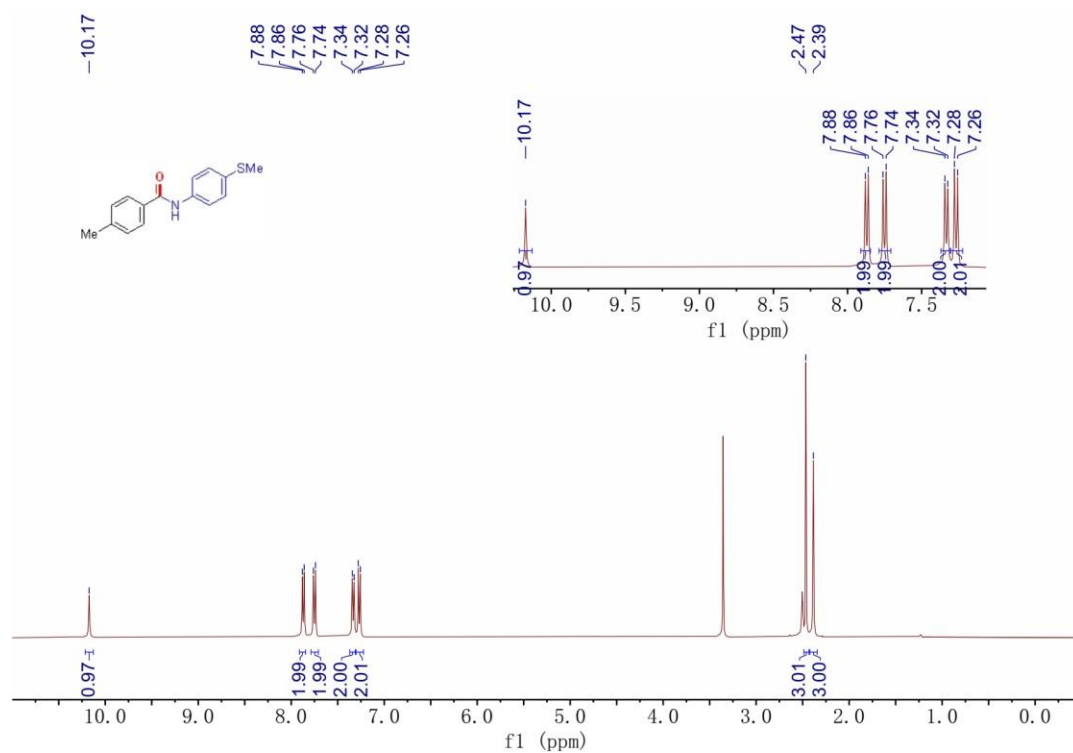
¹H NMR (400 MHz, DMSO-*d*₆) 599



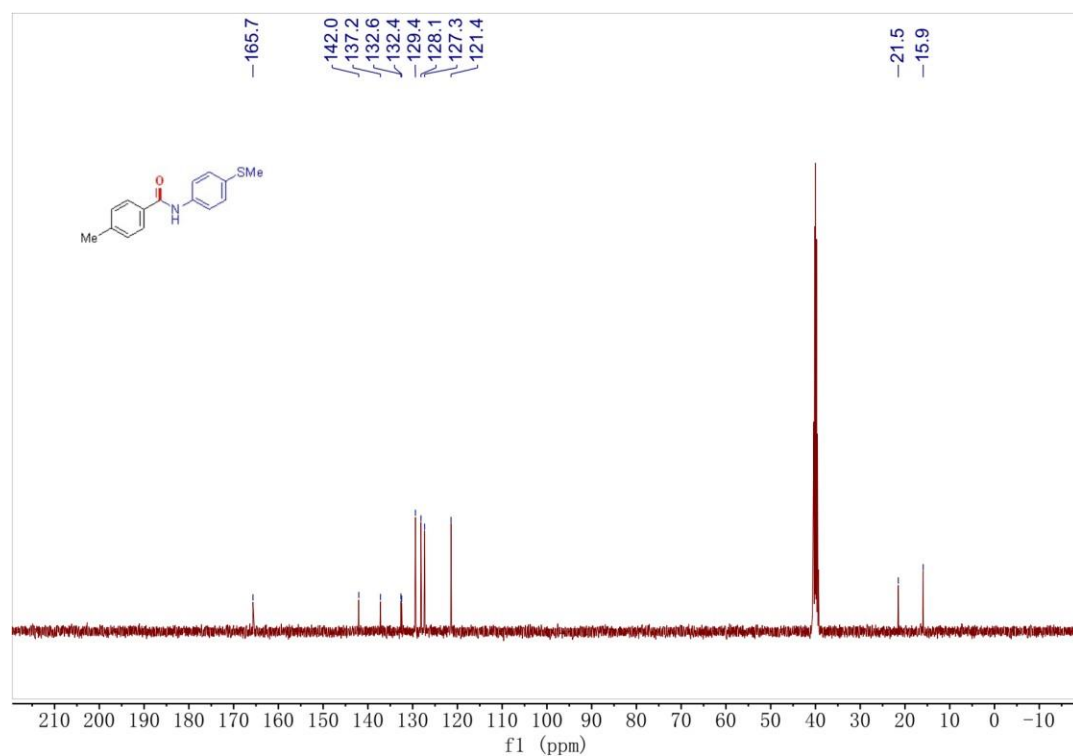
¹³C NMR (101 MHz, DMSO) 58



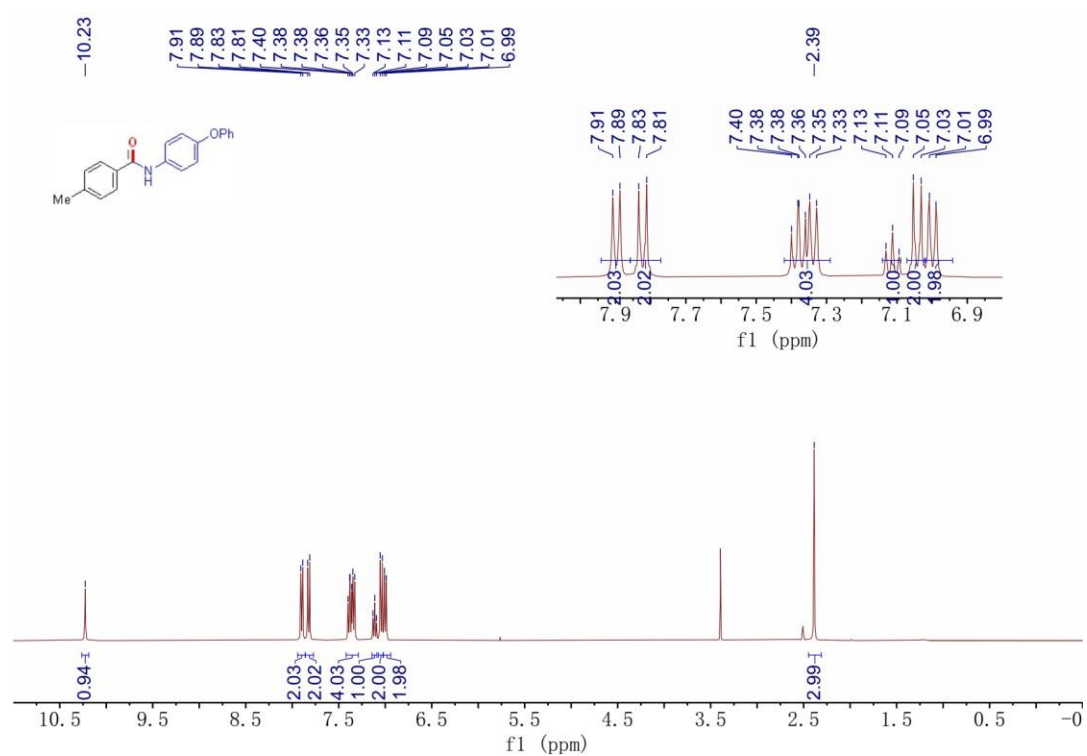
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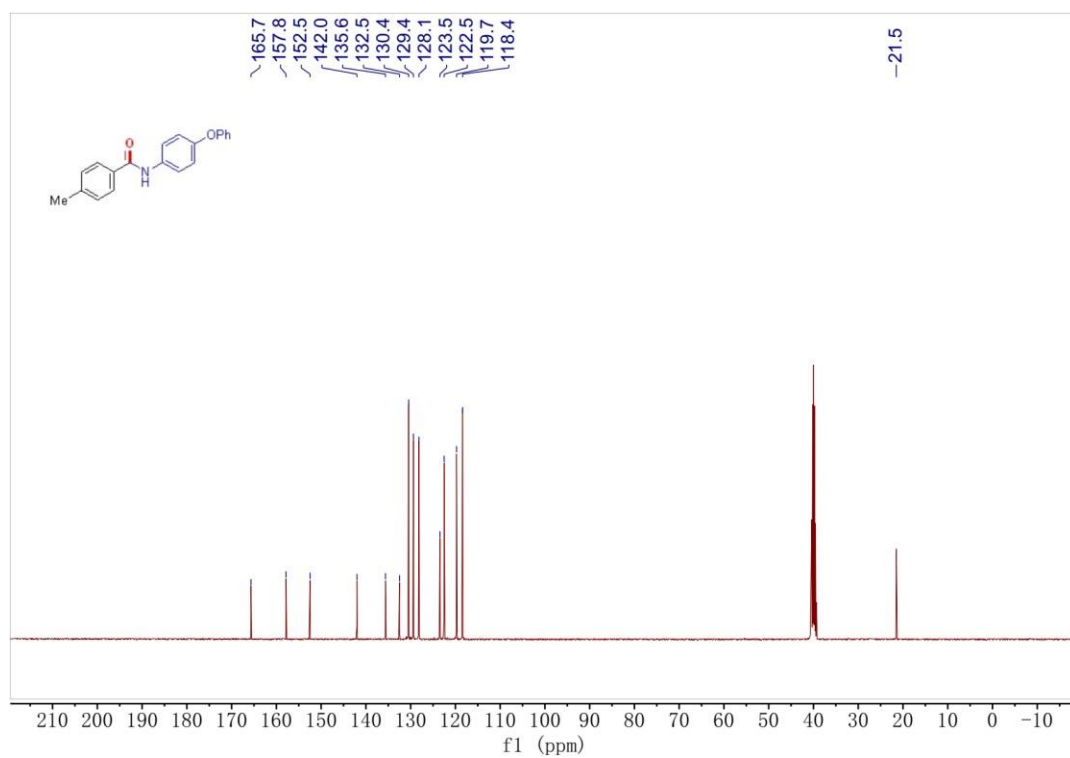
¹³C NMR (101 MHz, DMSO) 59



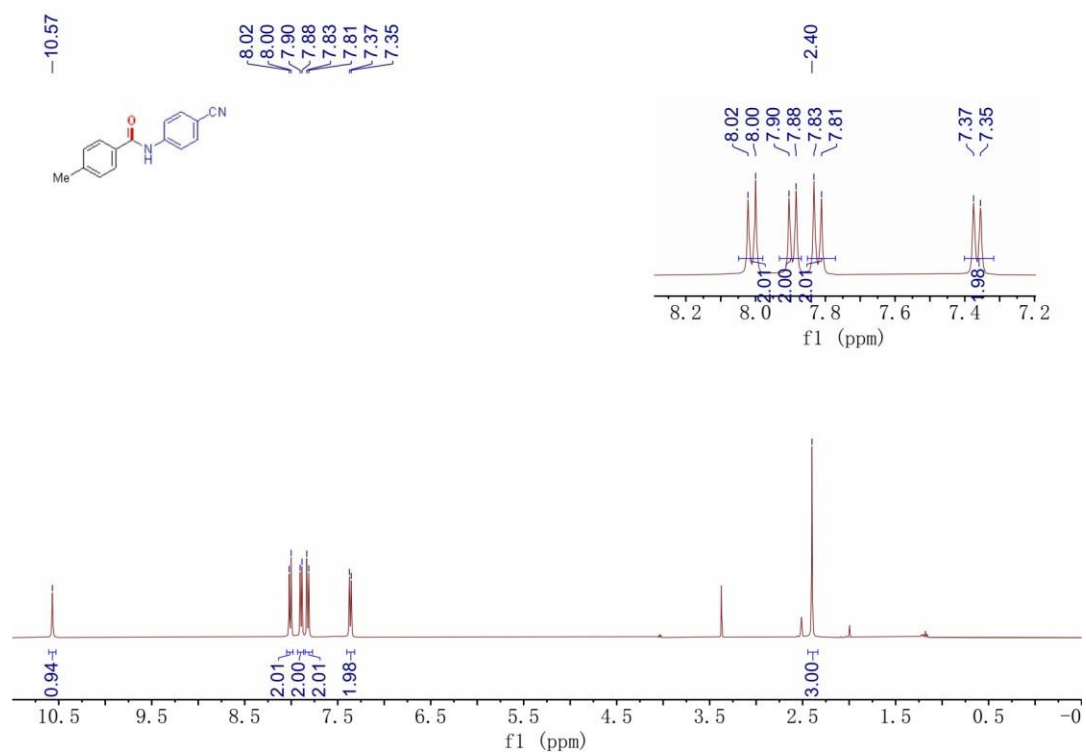
¹H NMR (400 MHz, DMSO-*d*₆) 60



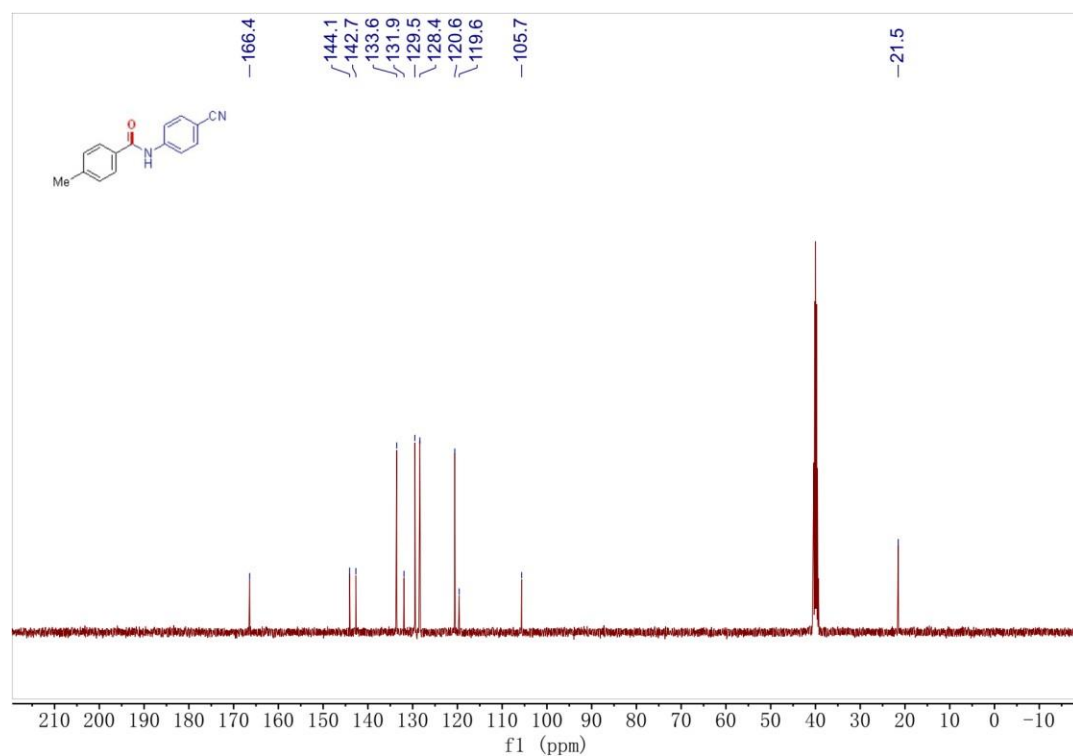
¹³C NMR (101 MHz, DMSO) 60



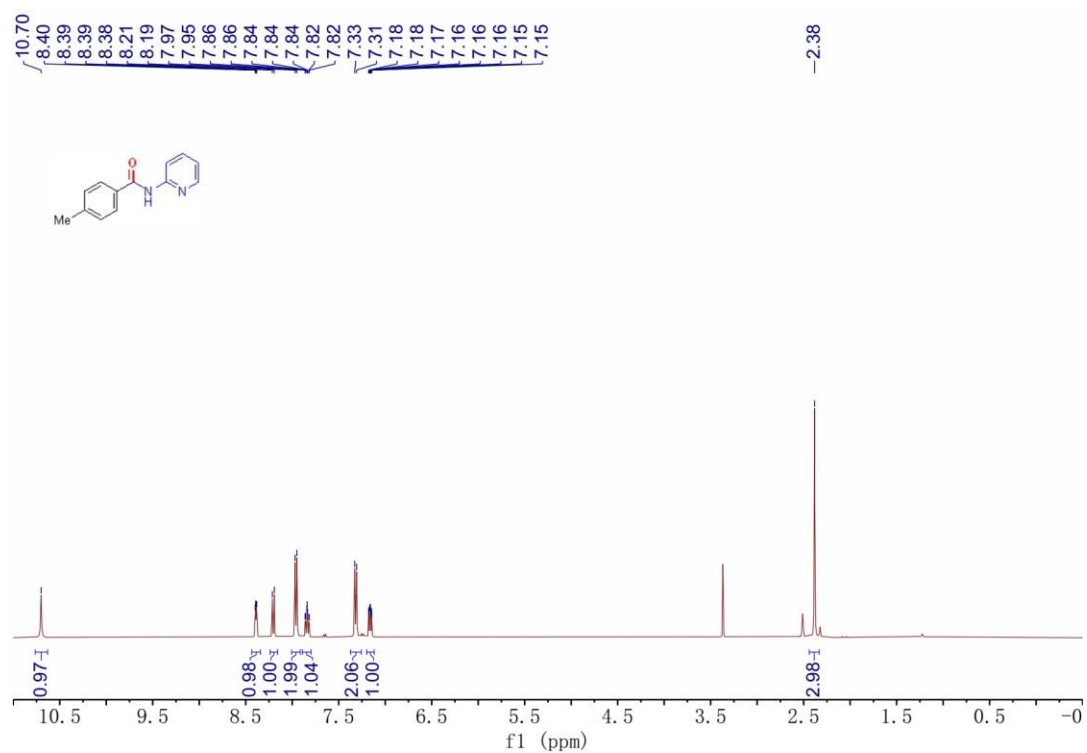
¹H NMR (400 MHz, DMSO-d₆) 6



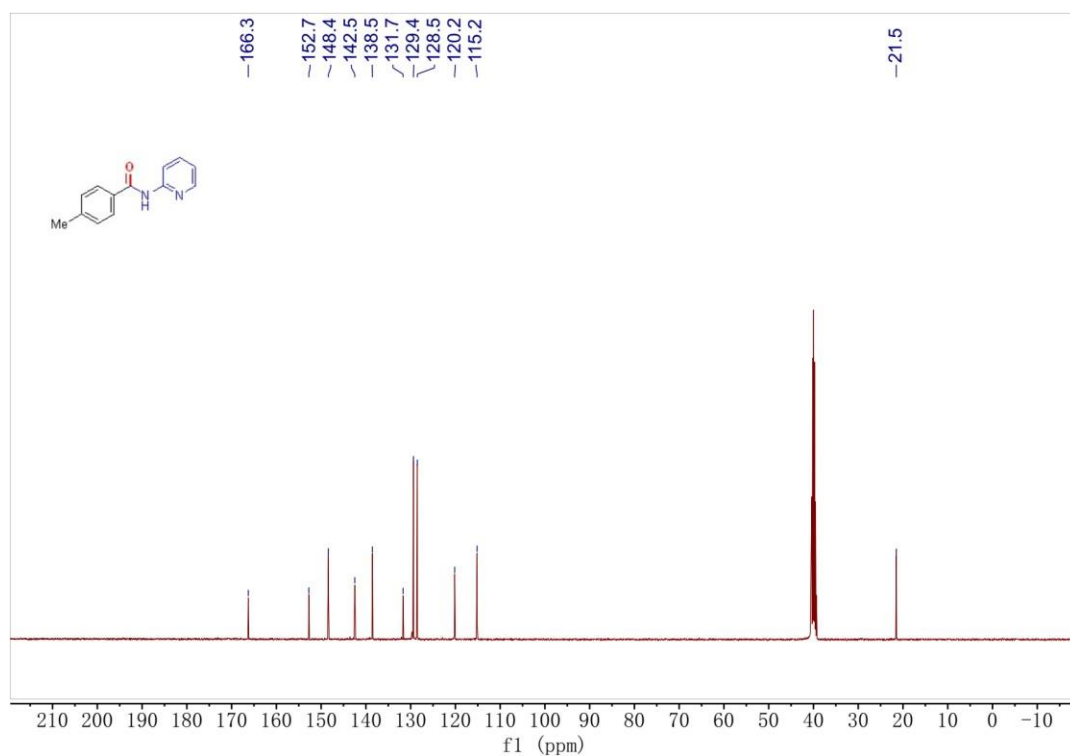
¹³C NMR (101 MHz, DMSO) 61



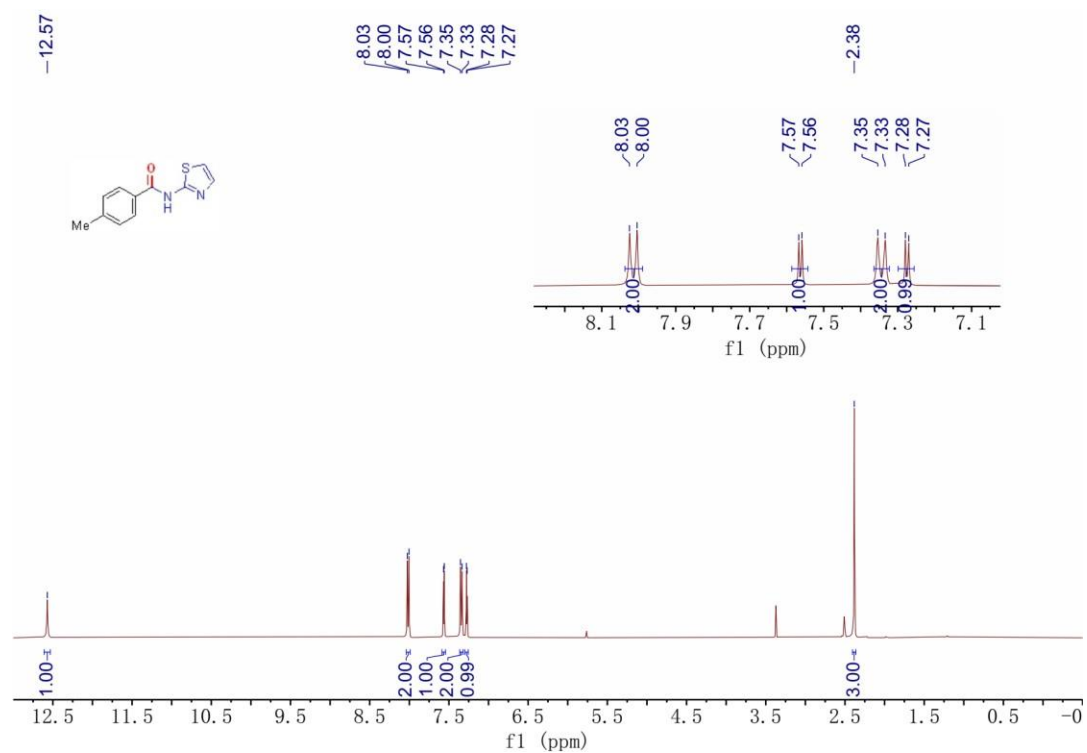
¹H NMR (400 MHz, DMSO-*d*₆) 6



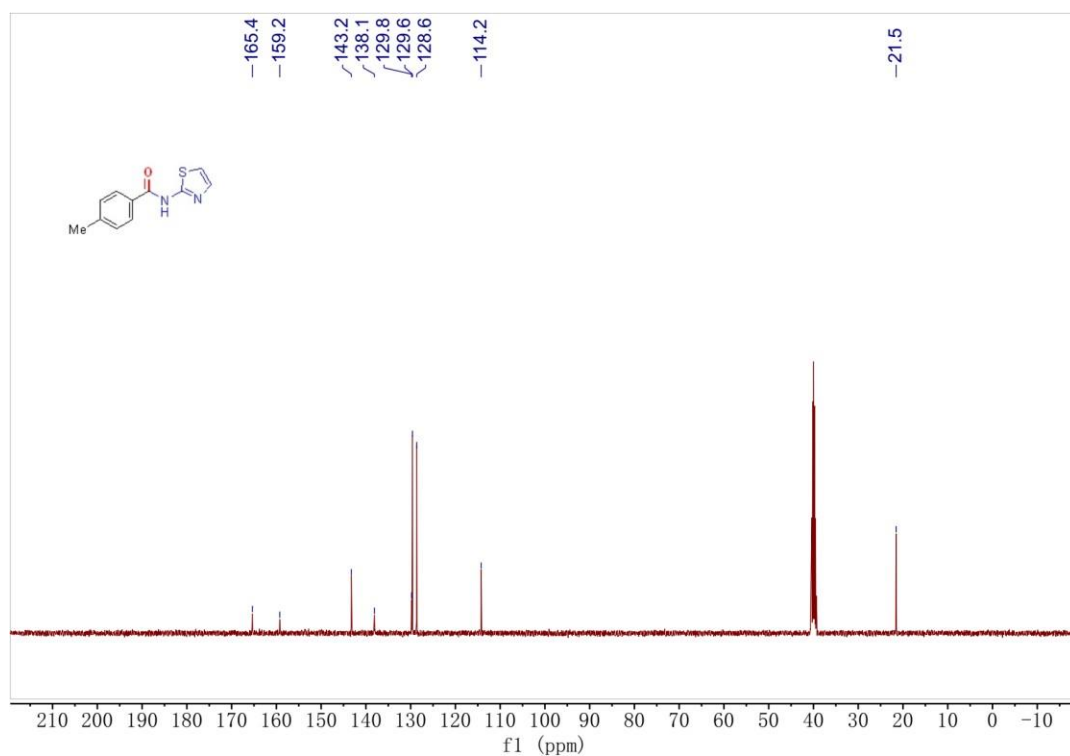
¹³C NMR (101 MHz, DMSO) 62



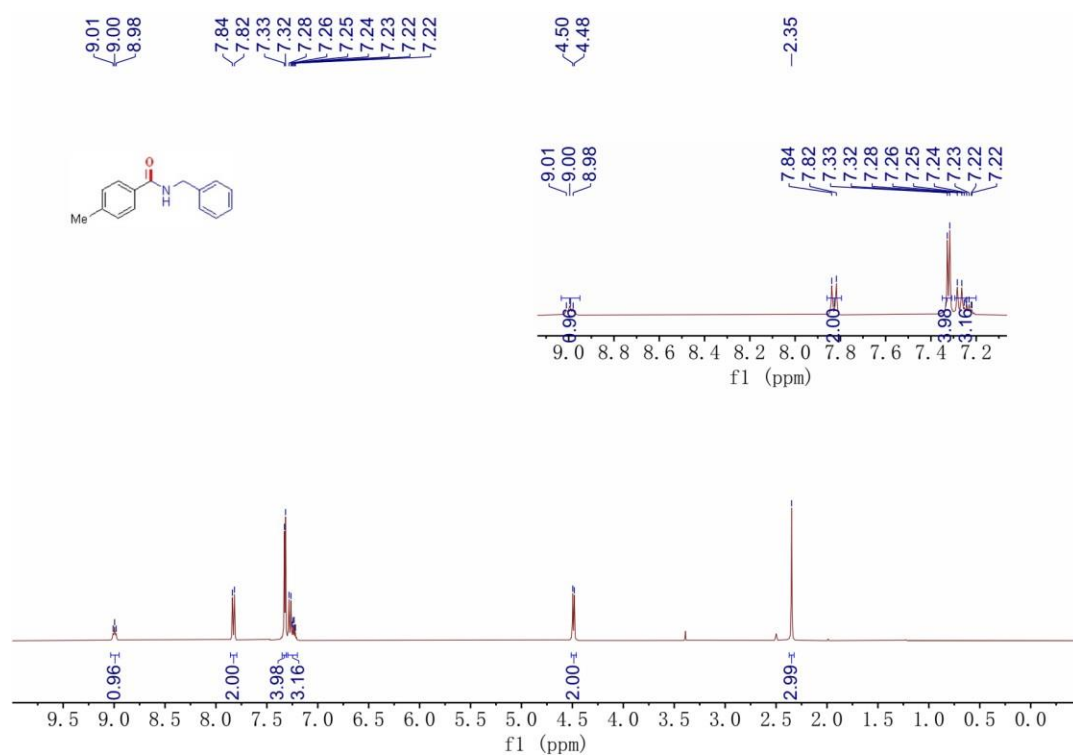
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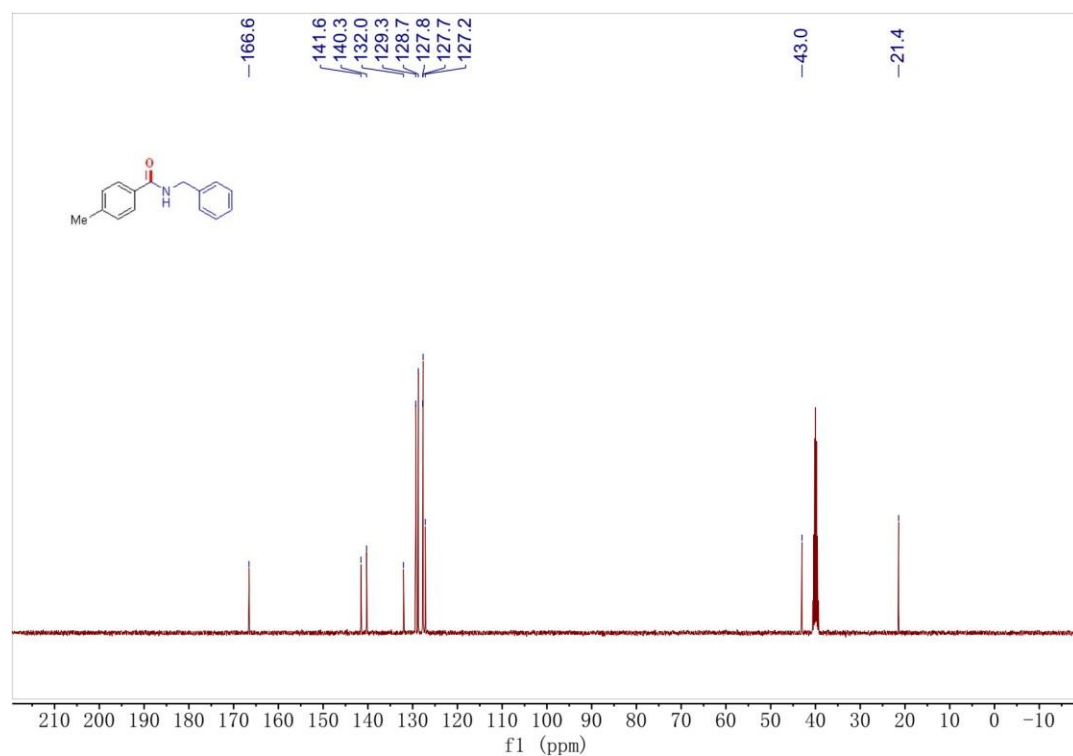
¹³C NMR (101 MHz, DMSO) 63



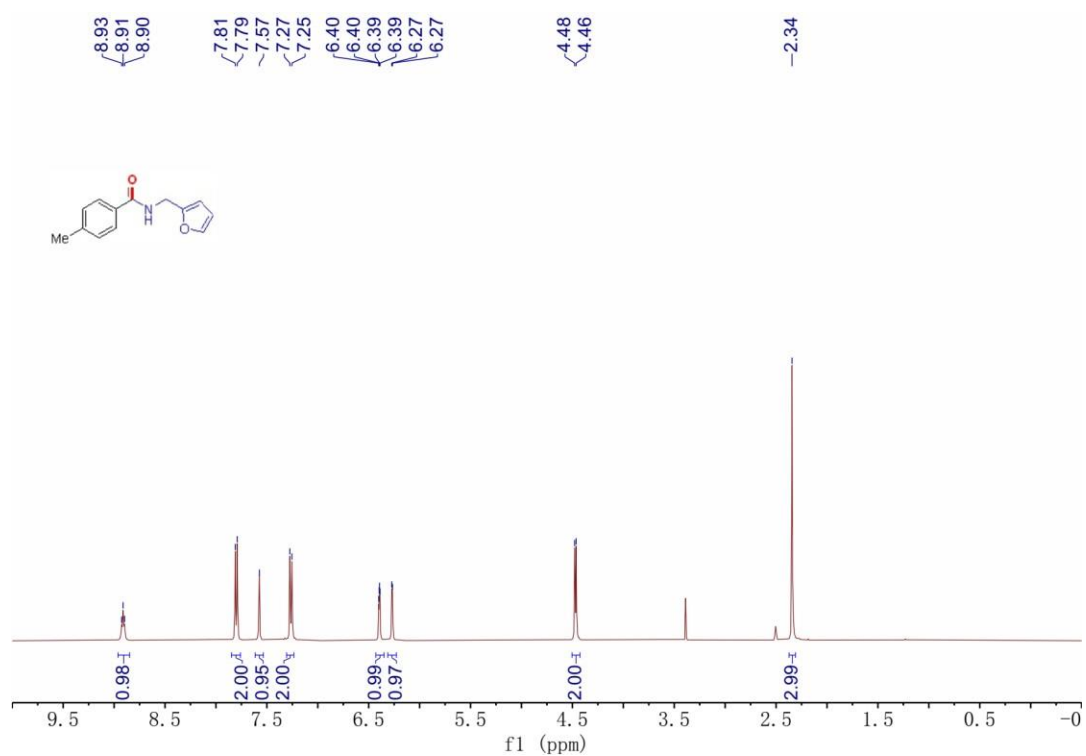
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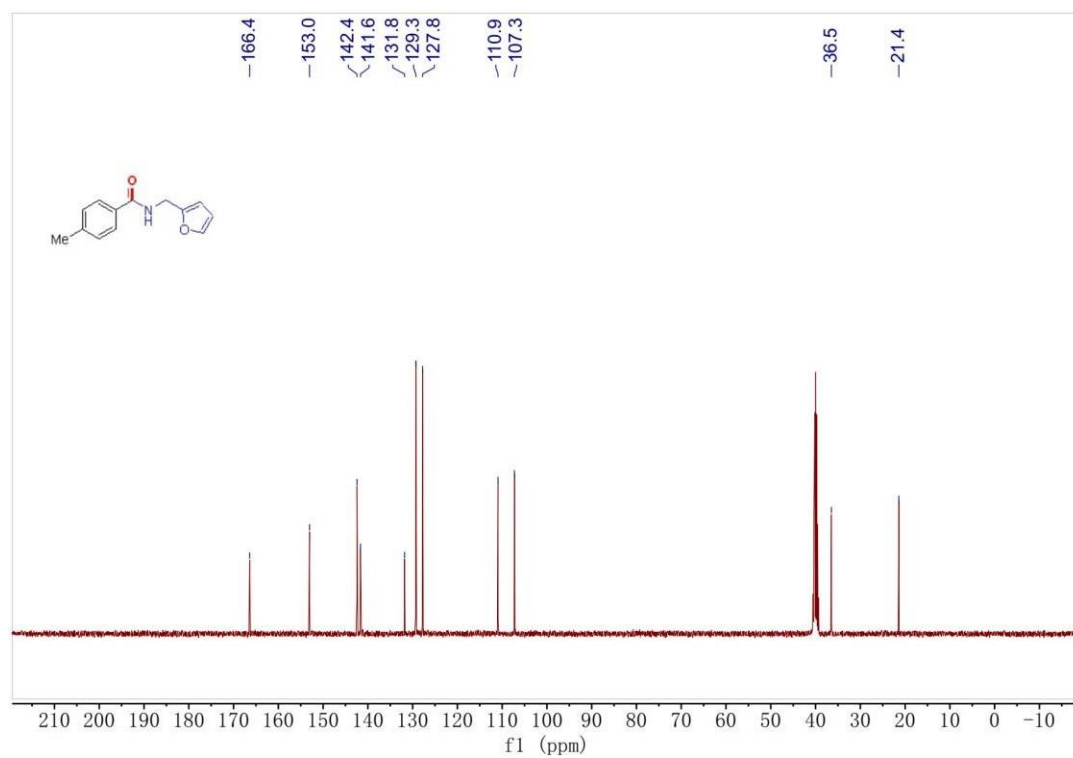
¹³C NMR (101 MHz, DMSO) 64



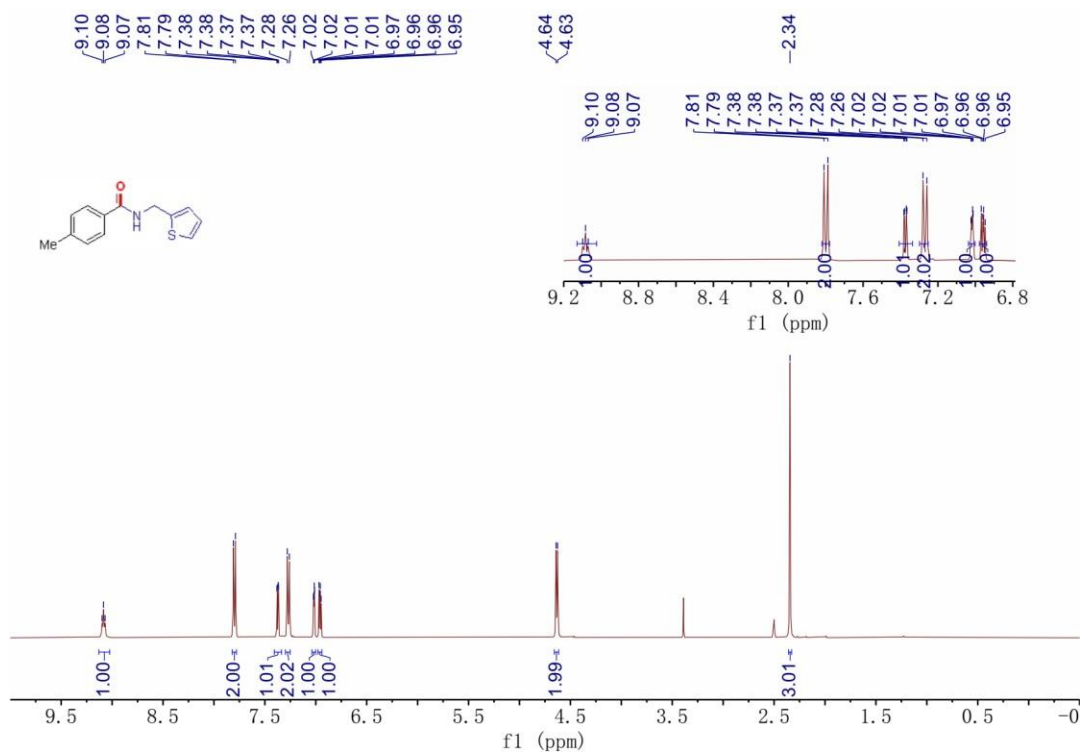
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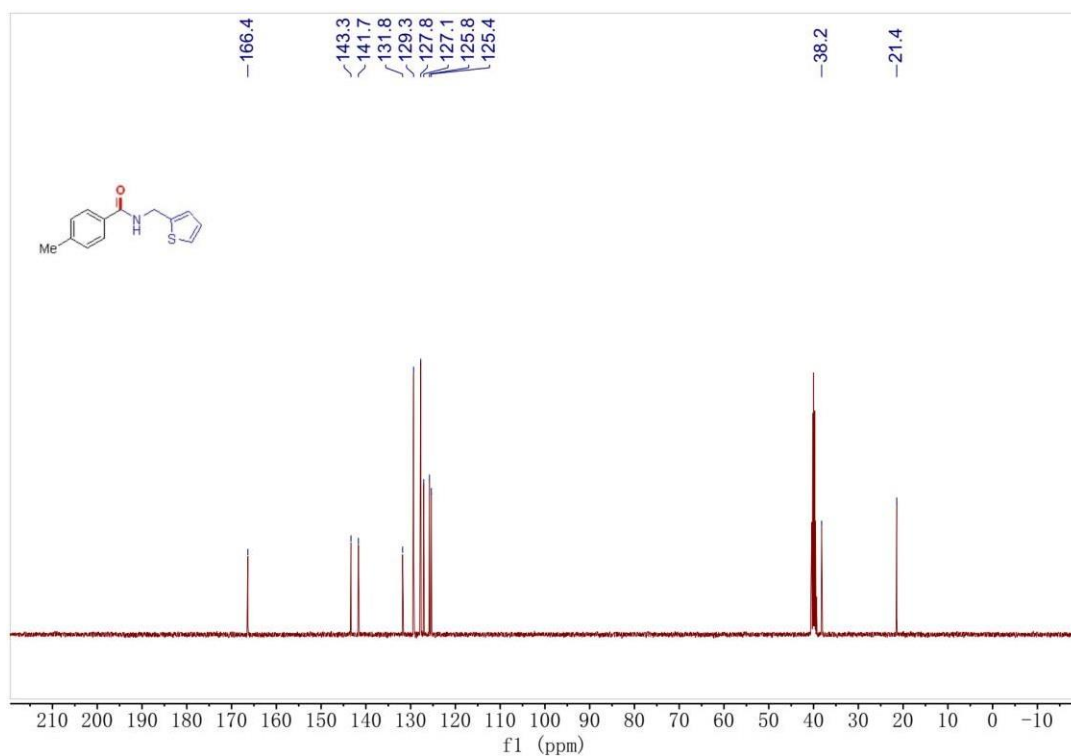
¹³C NMR (101 MHz, DMSO) 65



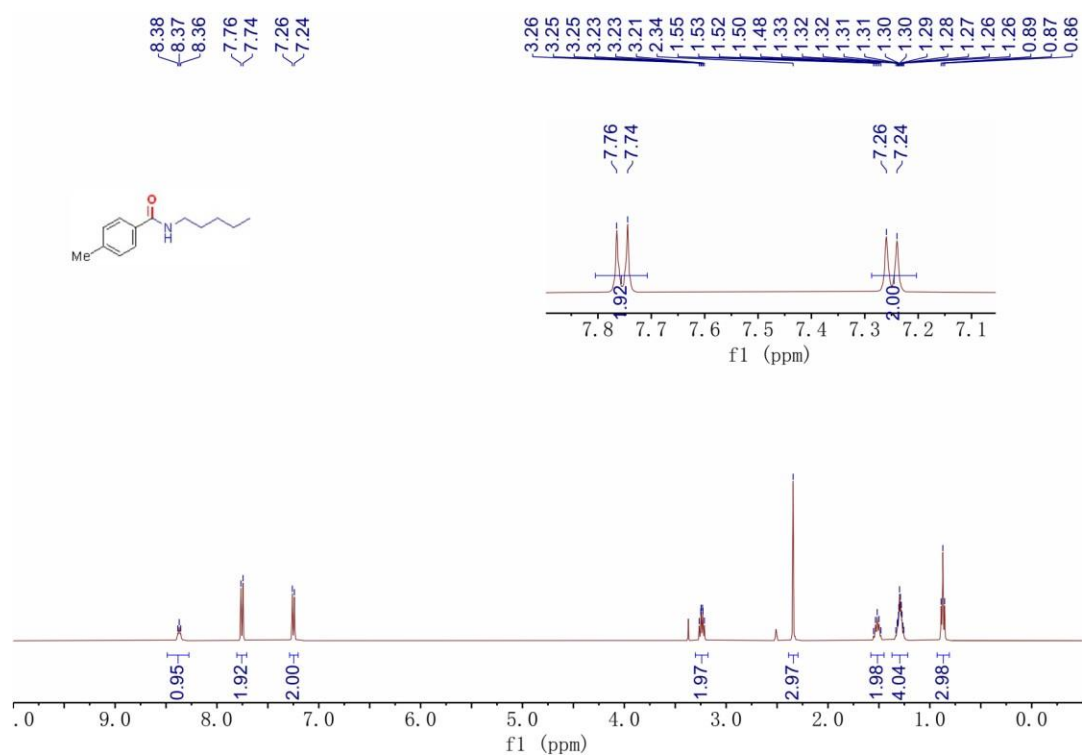
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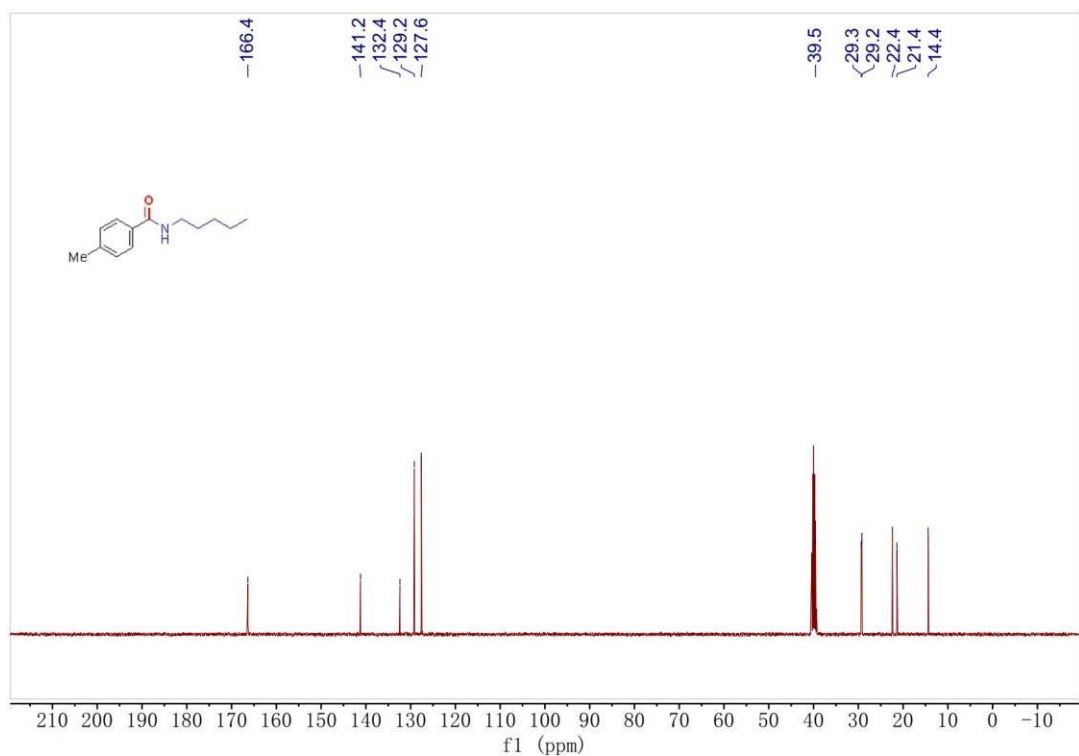
¹³C NMR (101 MHz, DMSO) 66



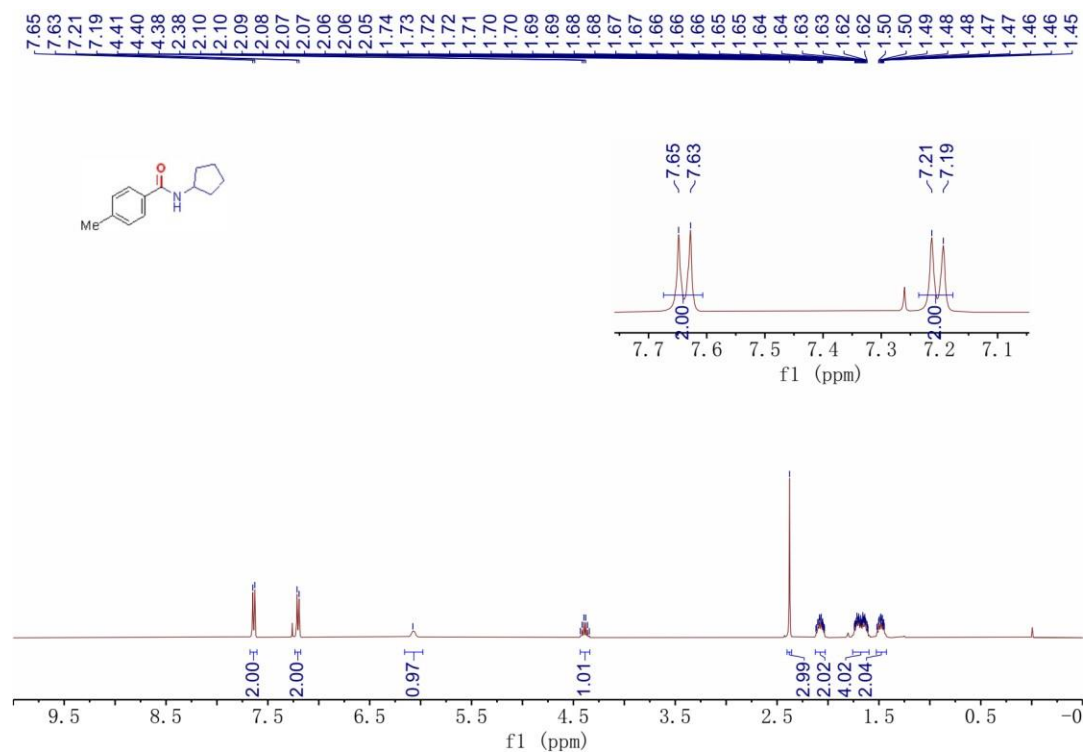
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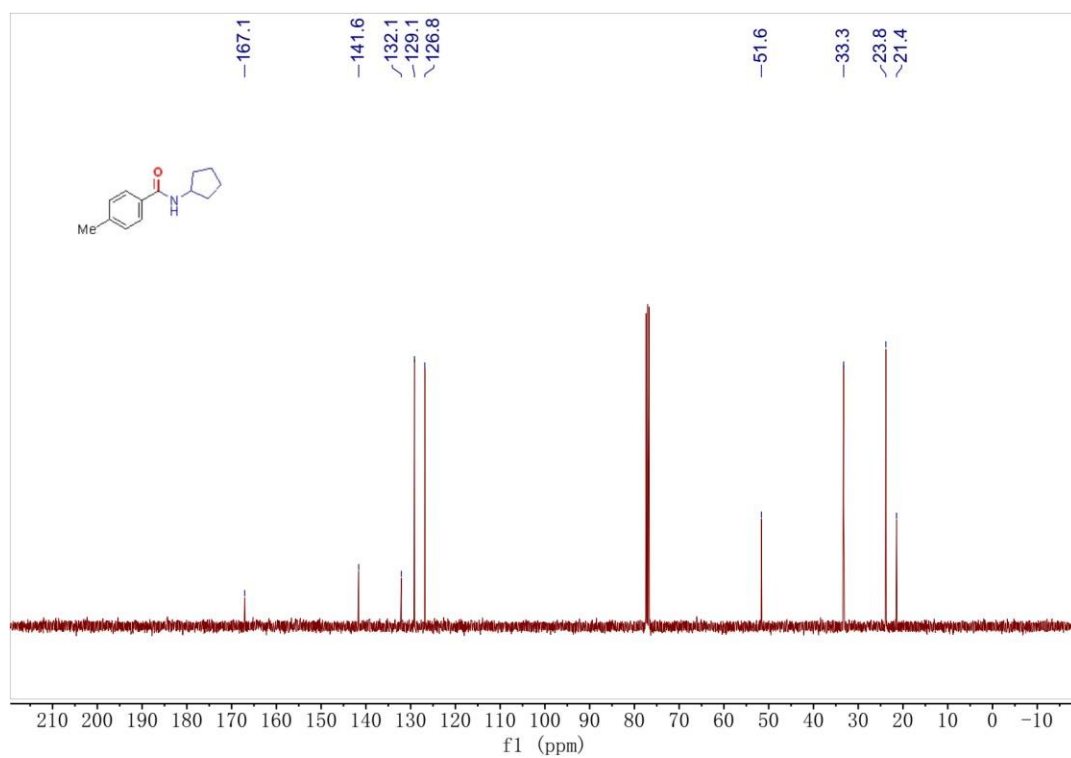
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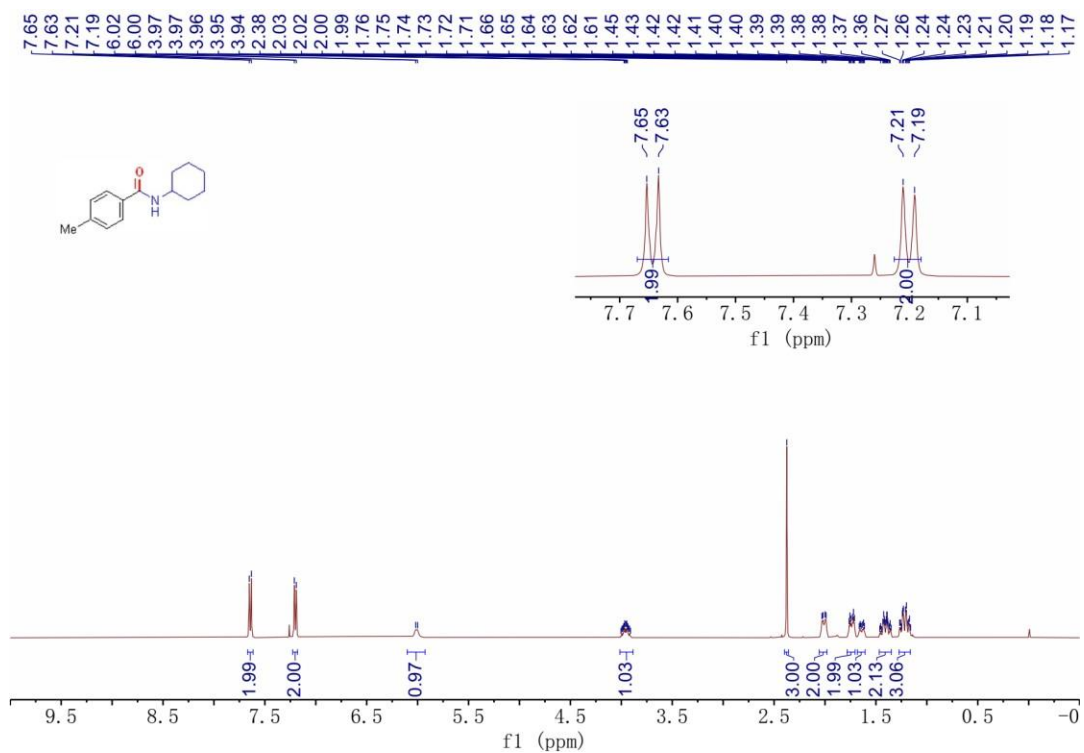
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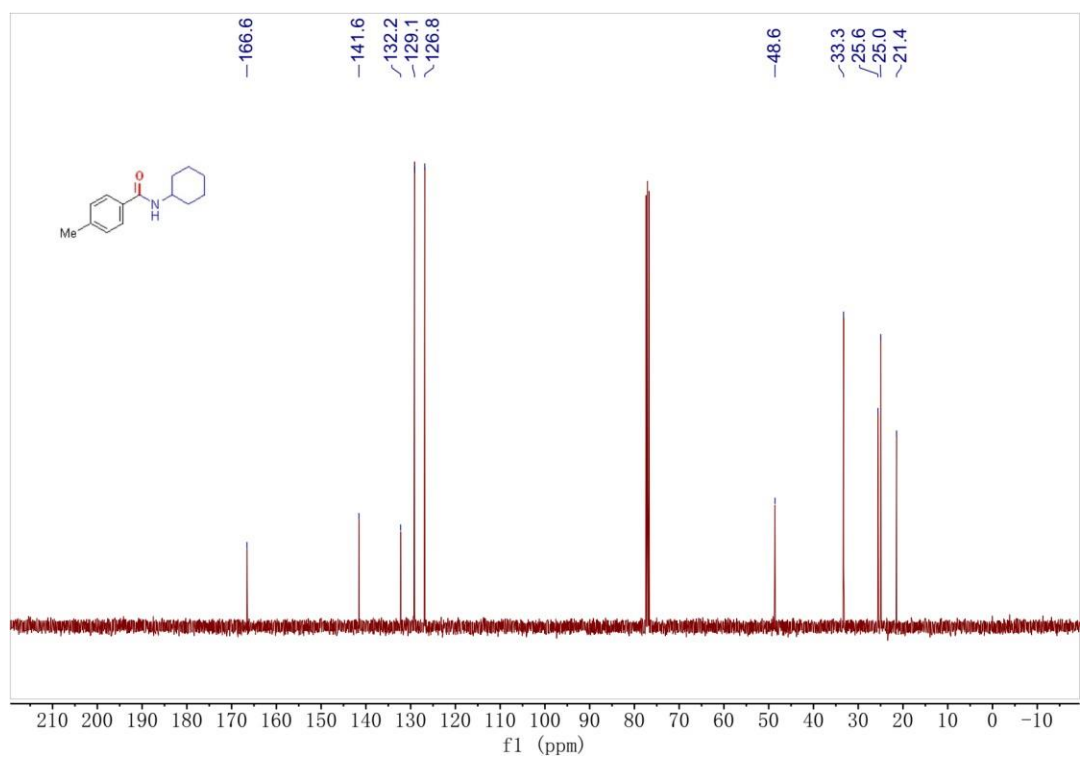
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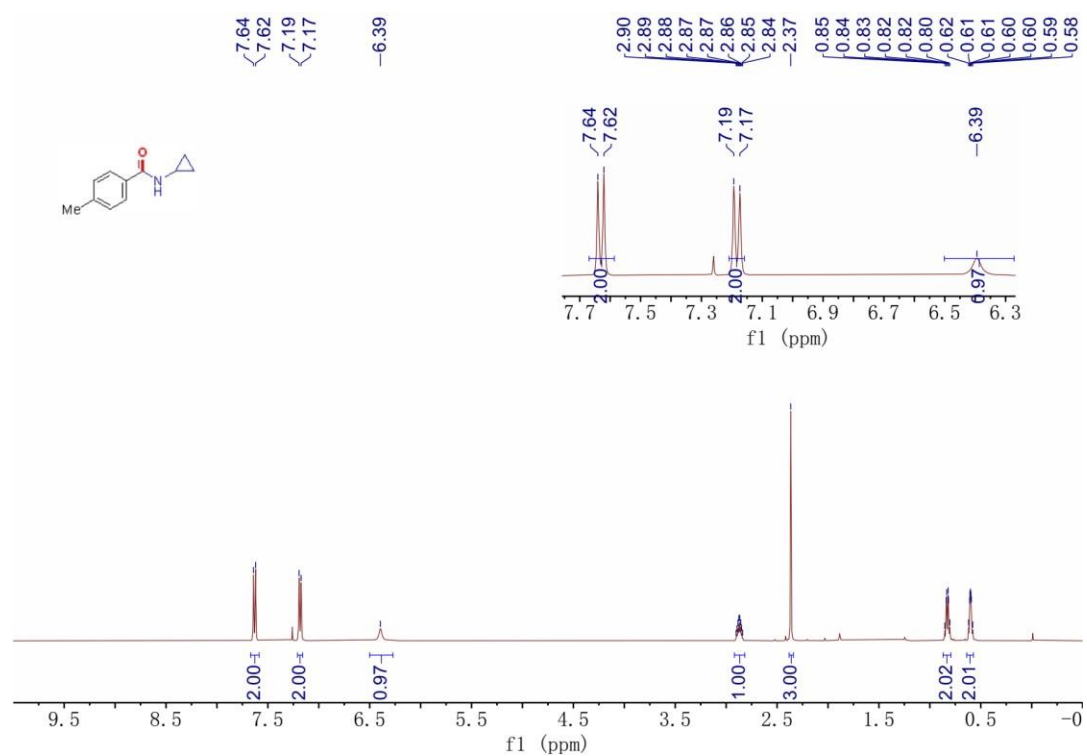
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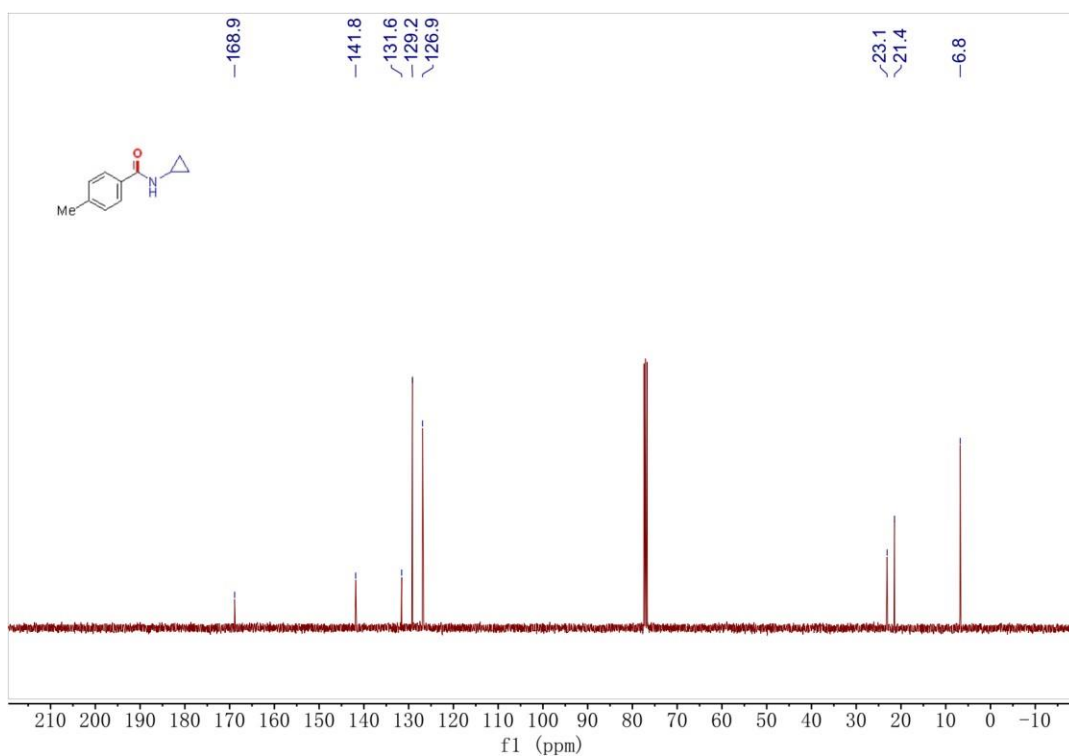
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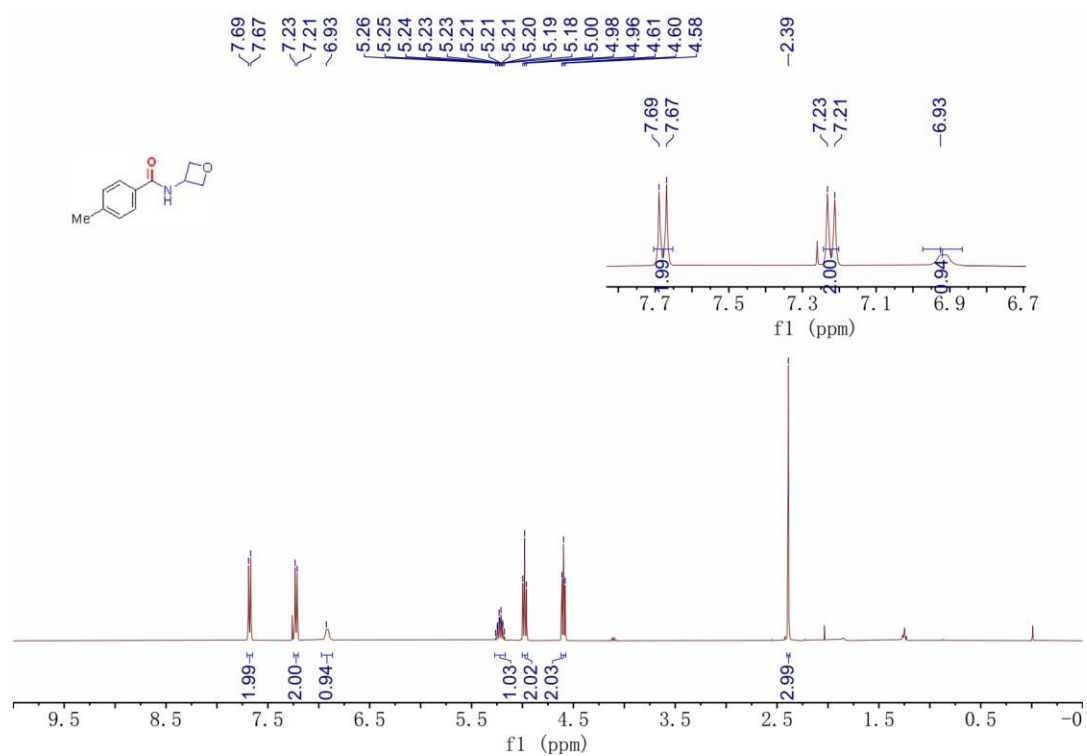
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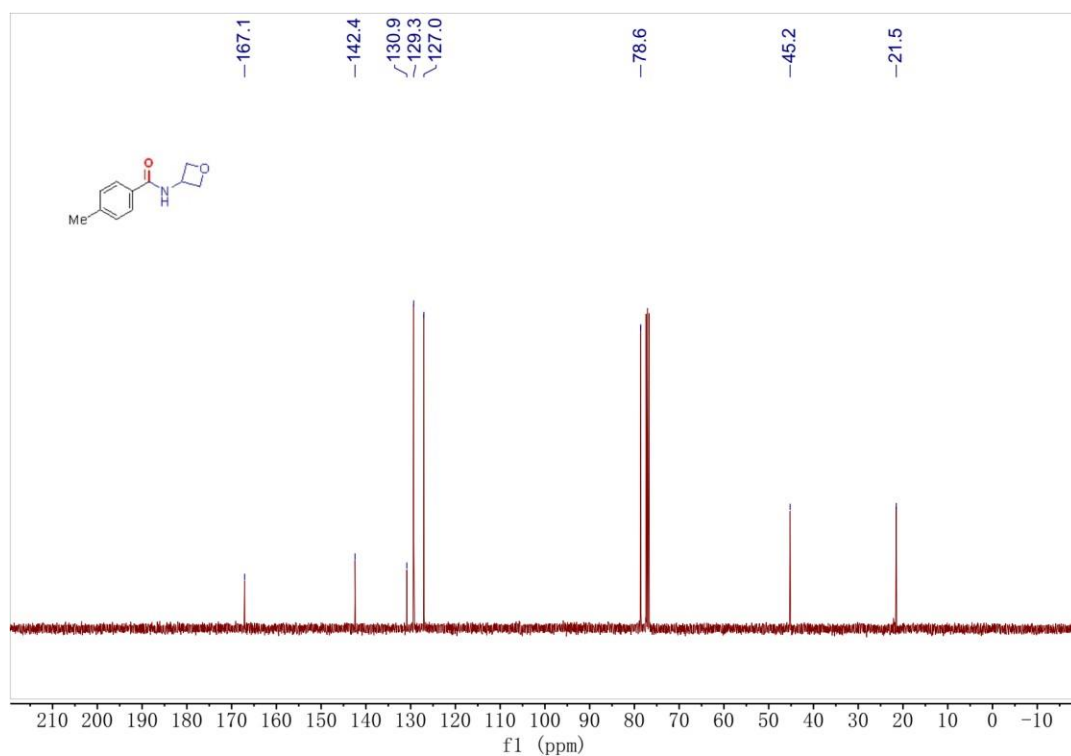
¹³C NMR (101 MHz, CDCl₃) 70



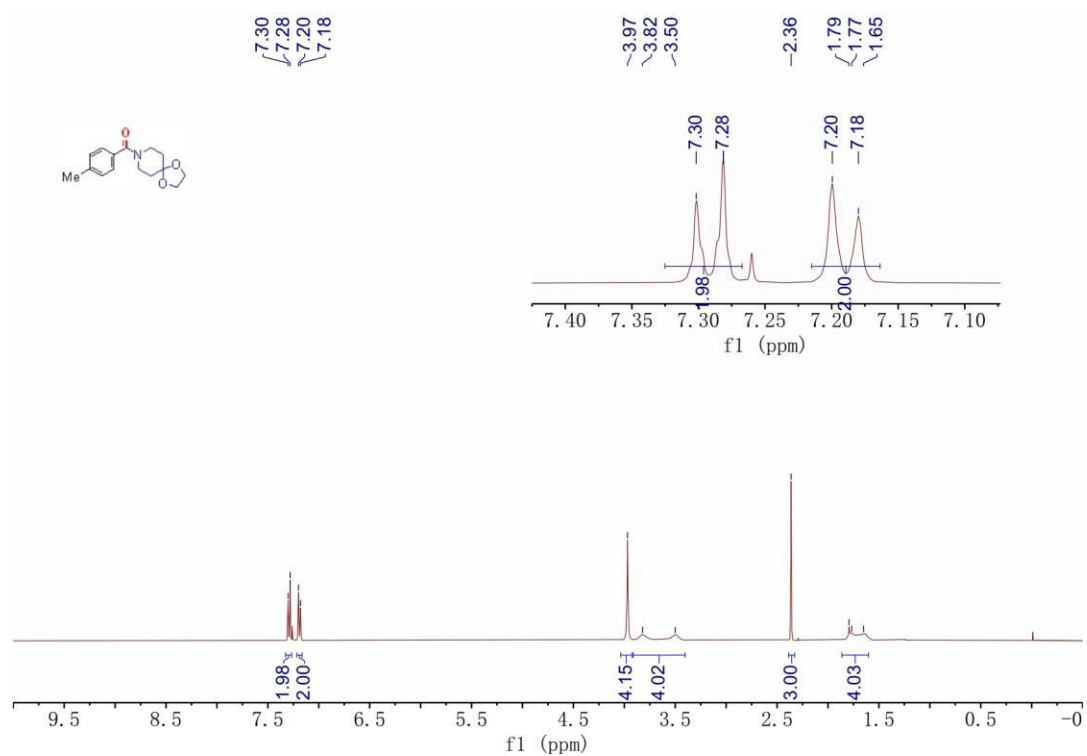
¹H NMR (400 MHz, CDCl₃) 7



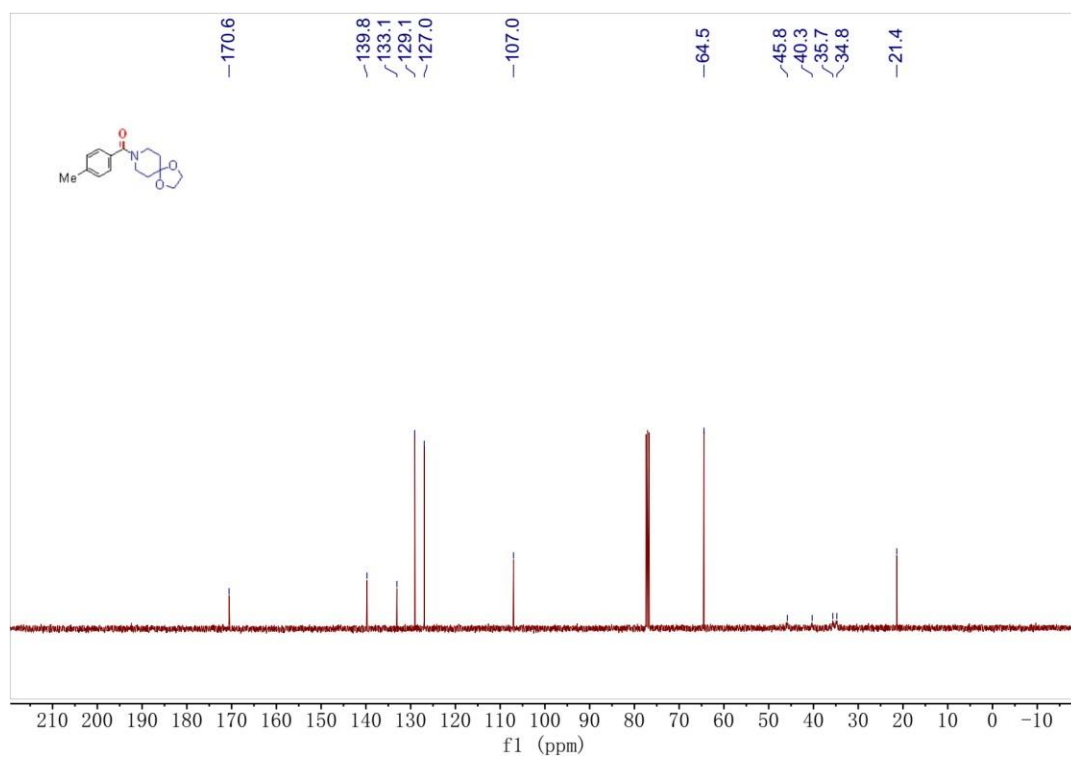
¹³C NMR (101 MHz, CDCl₃) 71



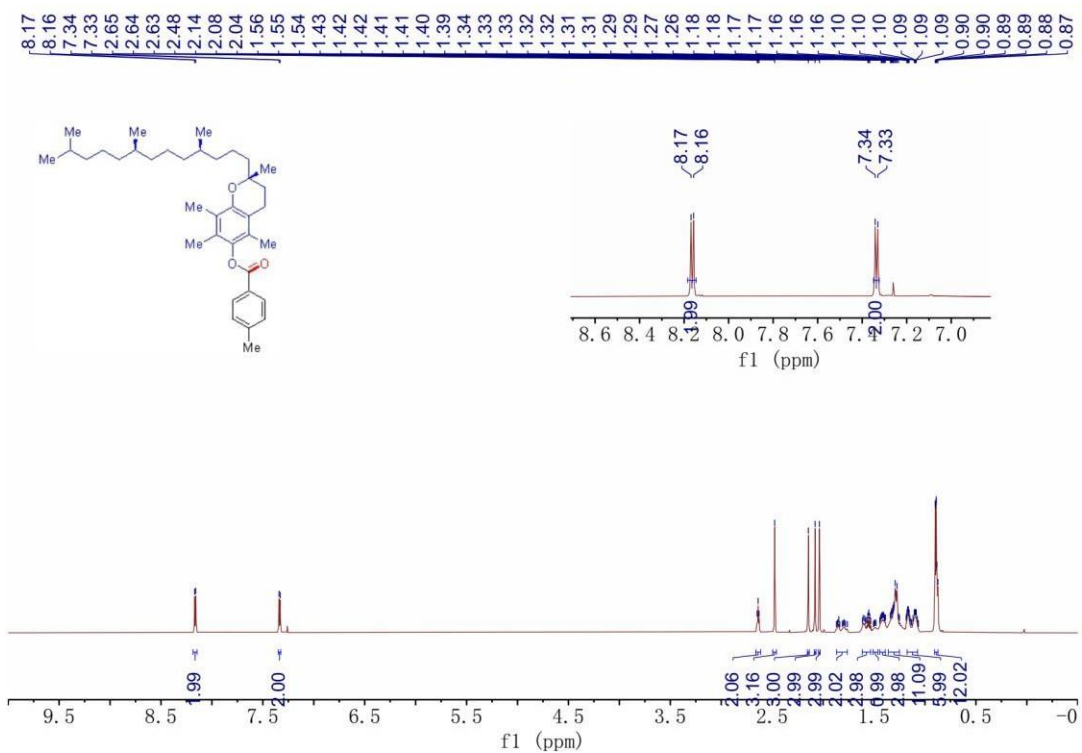
¹H NMR (400 MHz, CDCl₃) 7



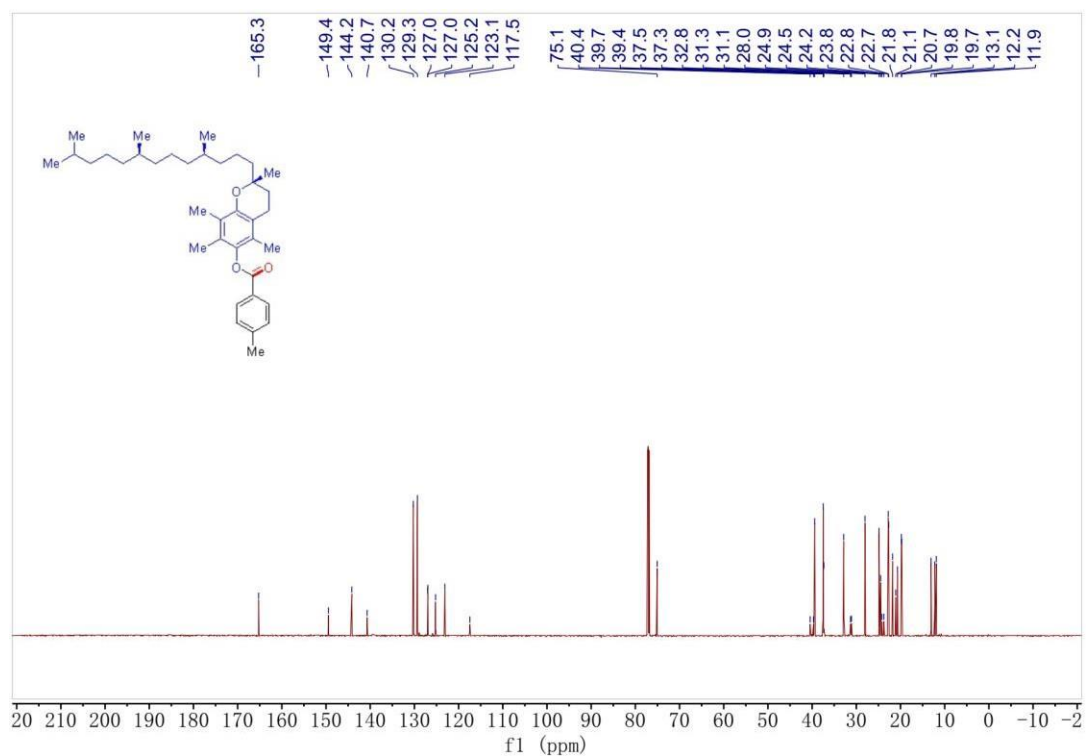
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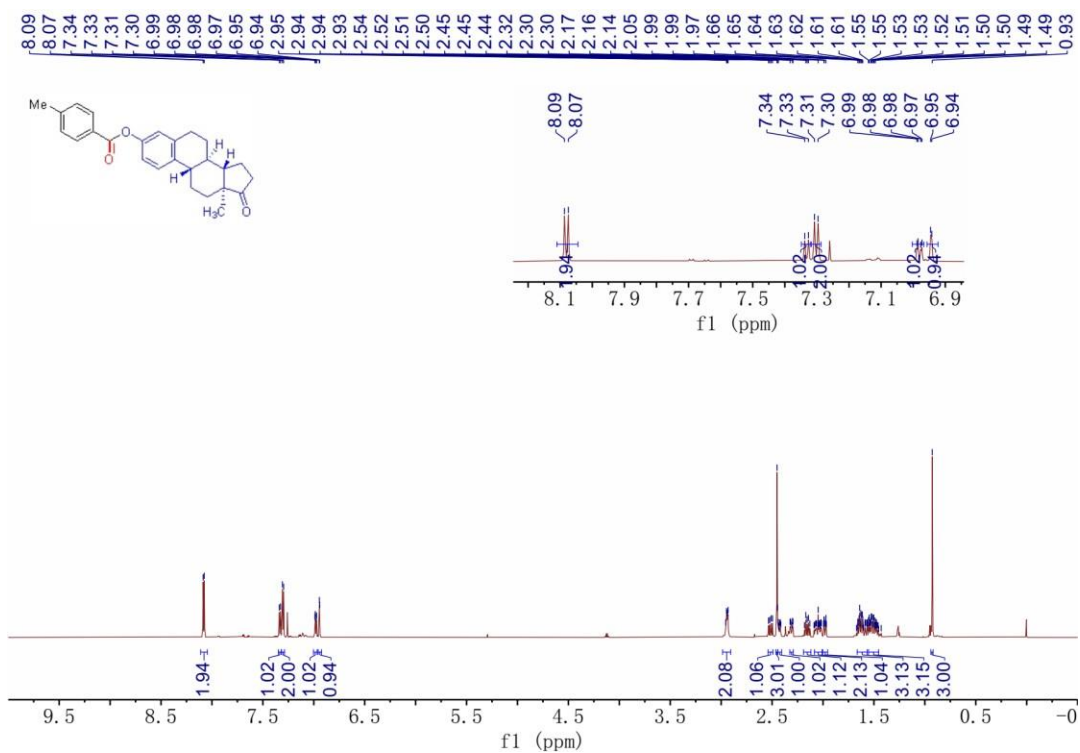
¹H NMR (700 MHz, CDCl₃) 7



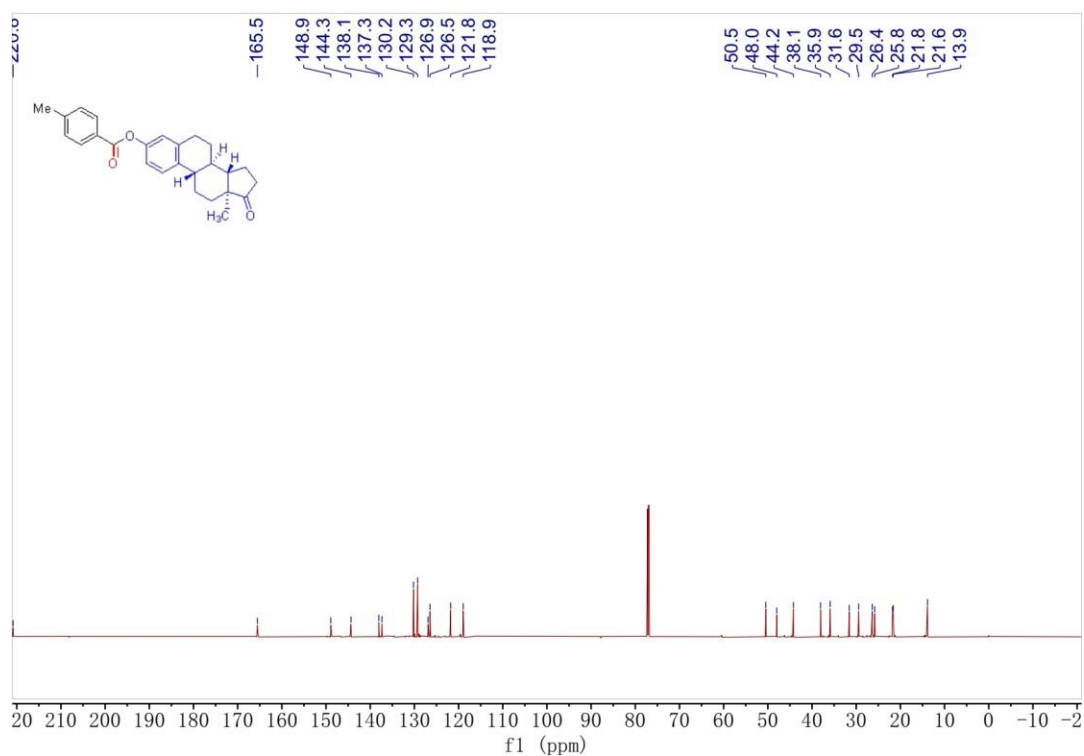
¹³C NMR (176 MHz, CDCl₃) 73



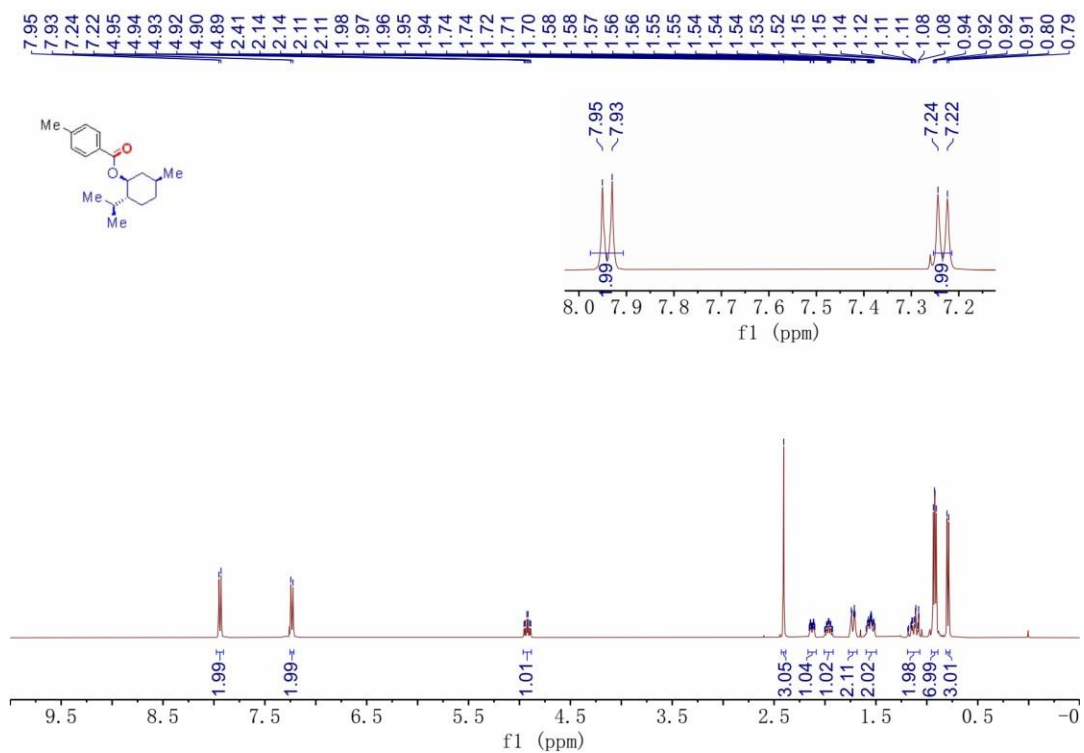
¹H NMR (700 MHz, CDCl₃) 7



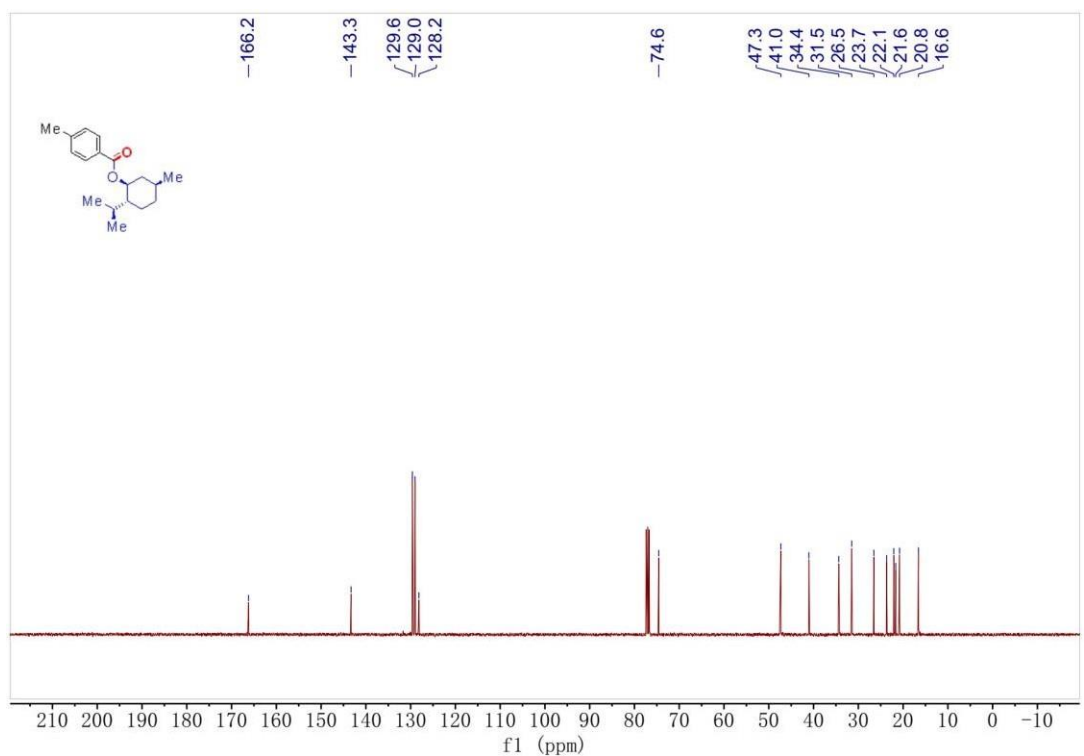
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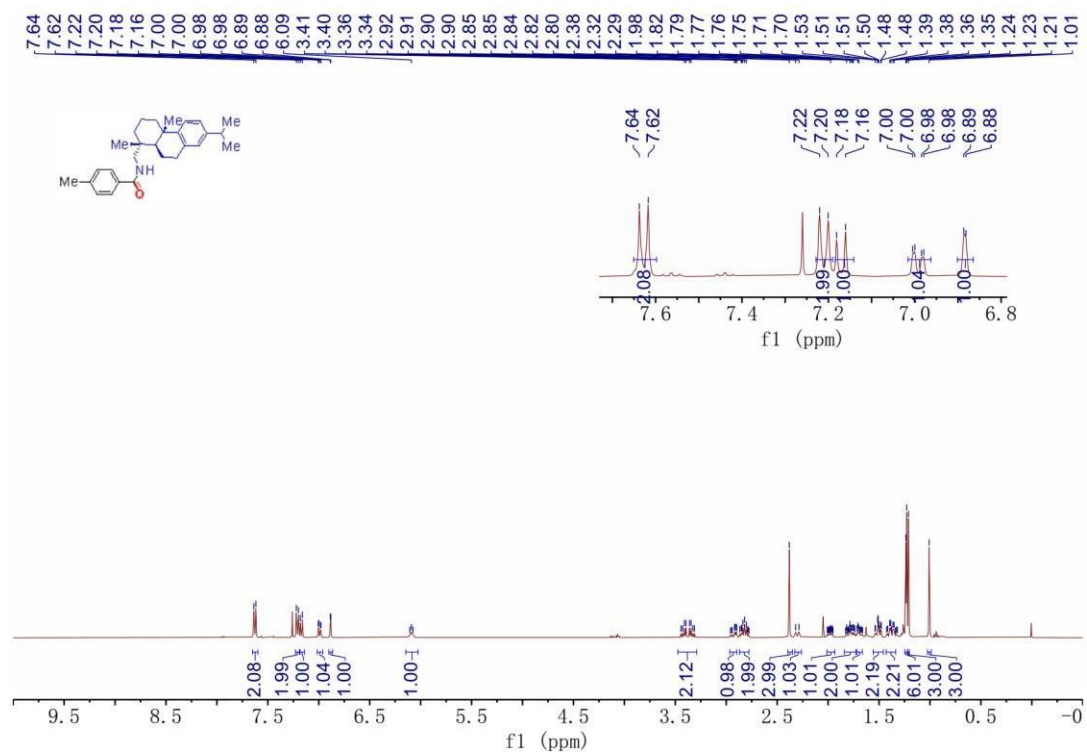
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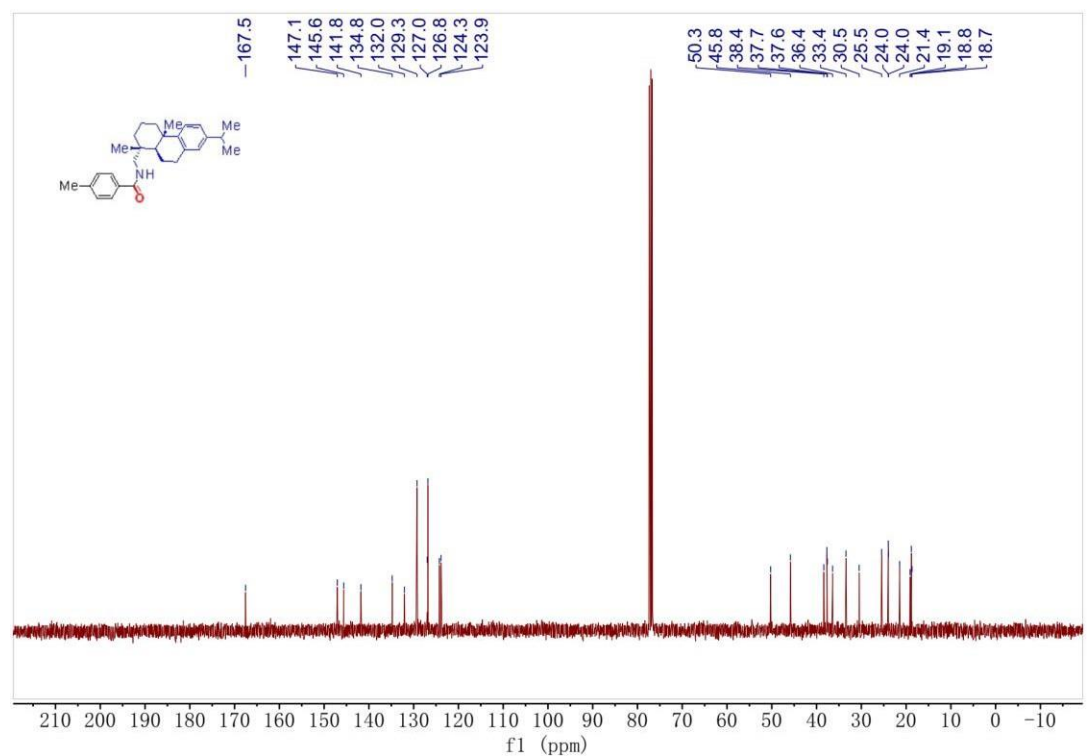
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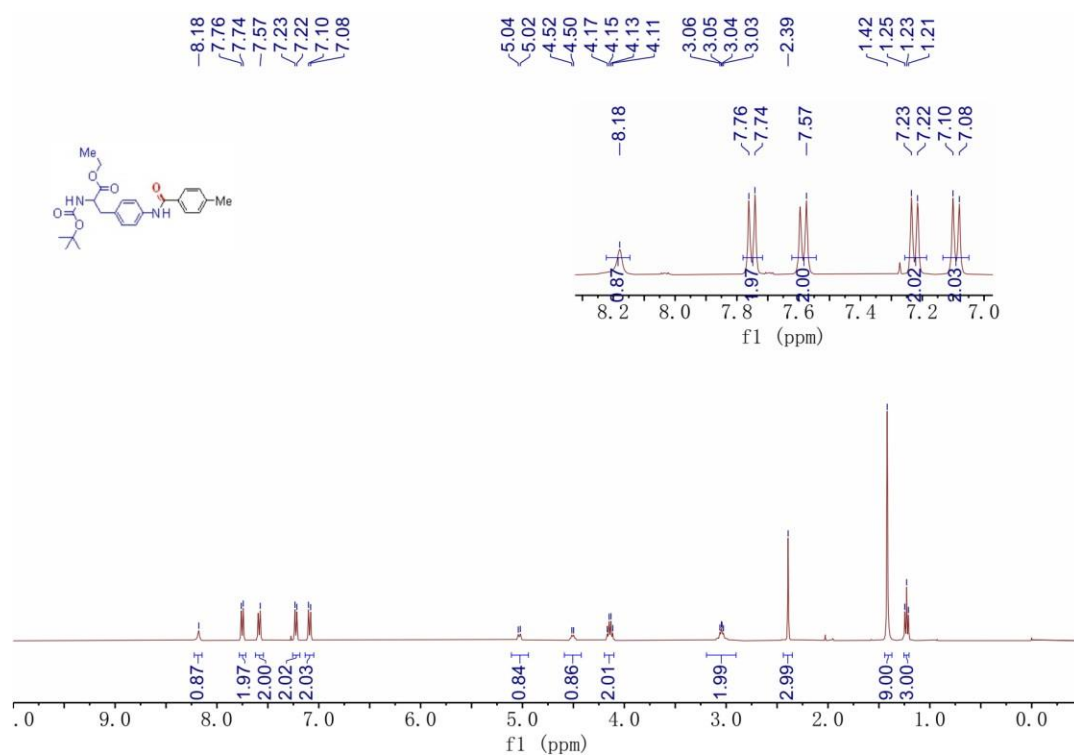
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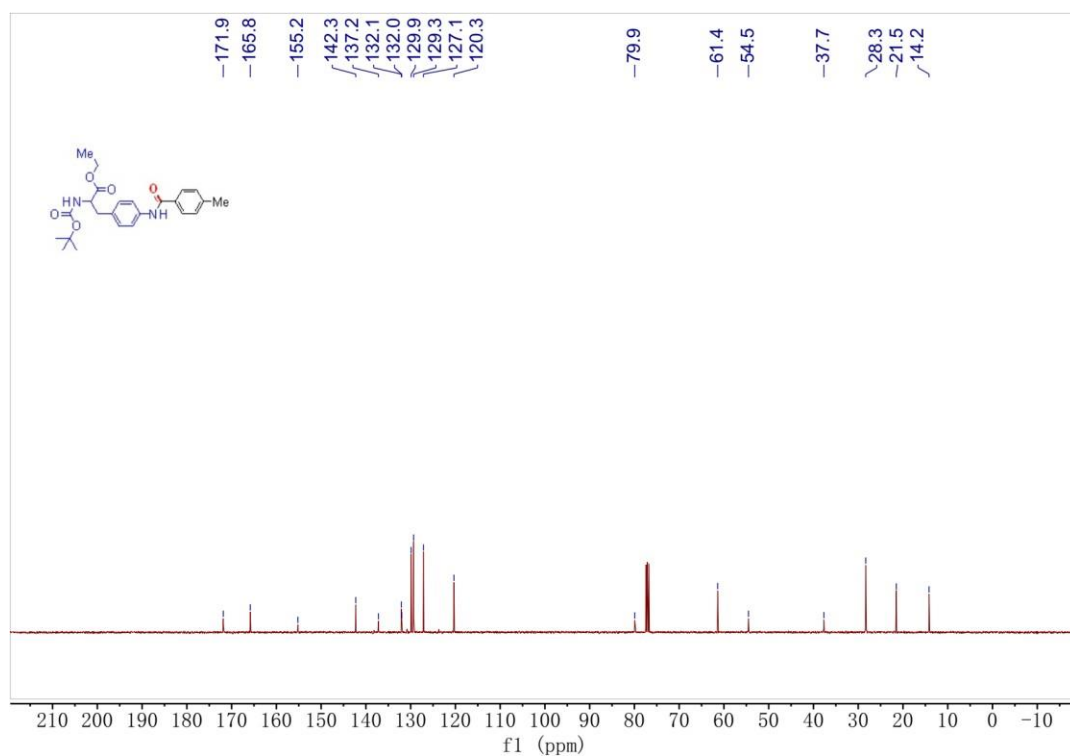
¹³C NMR (101 MHz, CDCl₃) 76



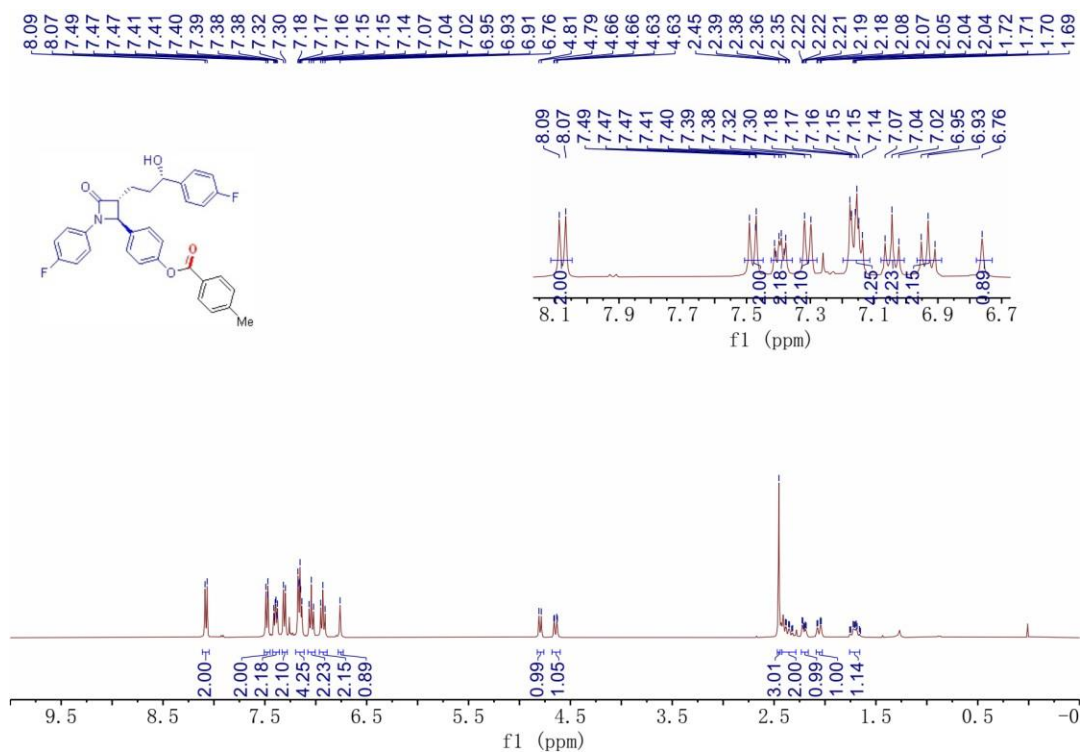
¹H NMR (400 MHz, CDCl₃) 7



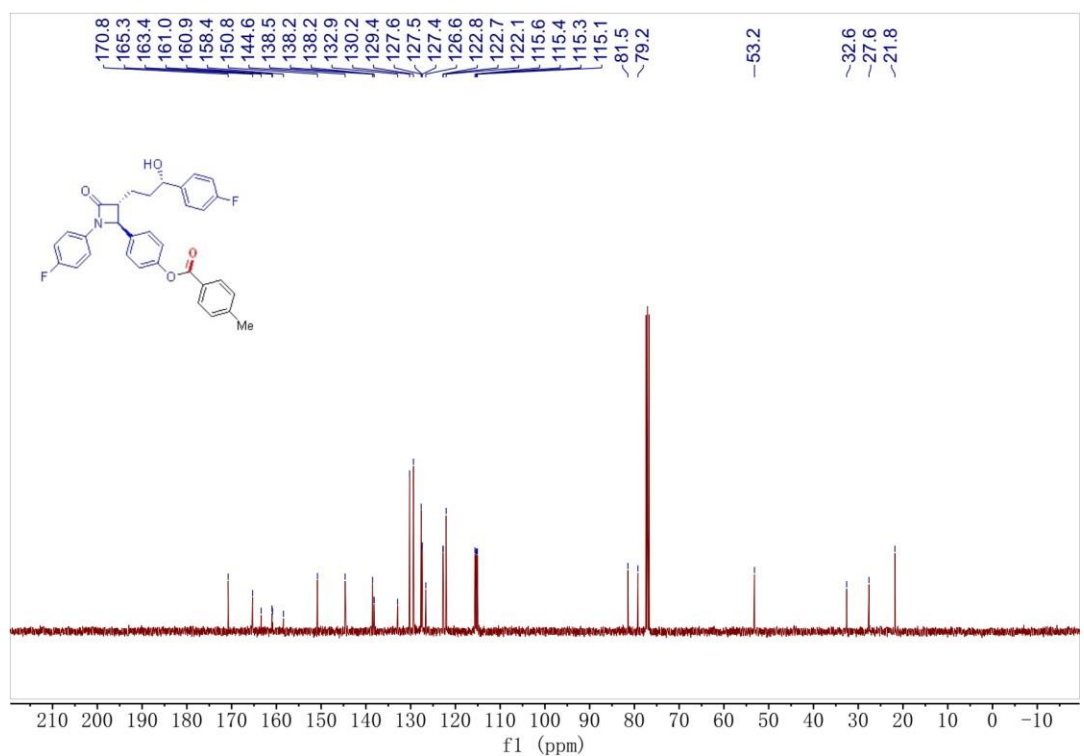
¹³C NMR (101 MHz, CDCl₃) 77



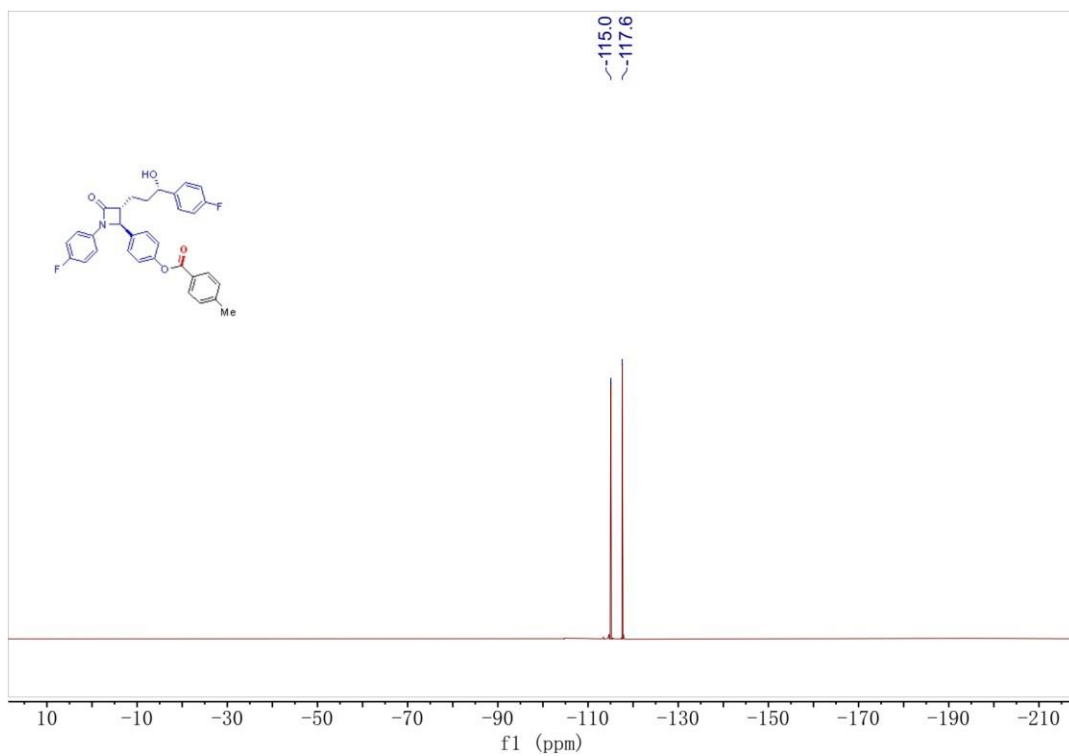
¹H NMR (400 MHz, CDCl₃) 7



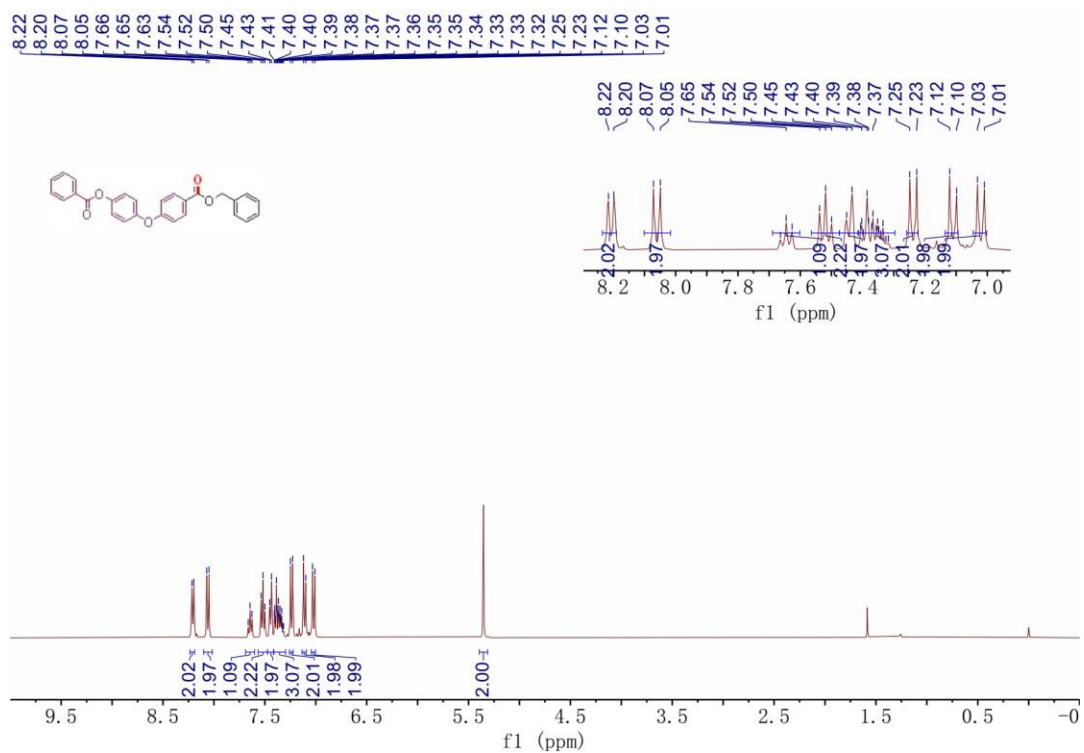
¹³C NMR (101 MHz, CDCl₃) 78



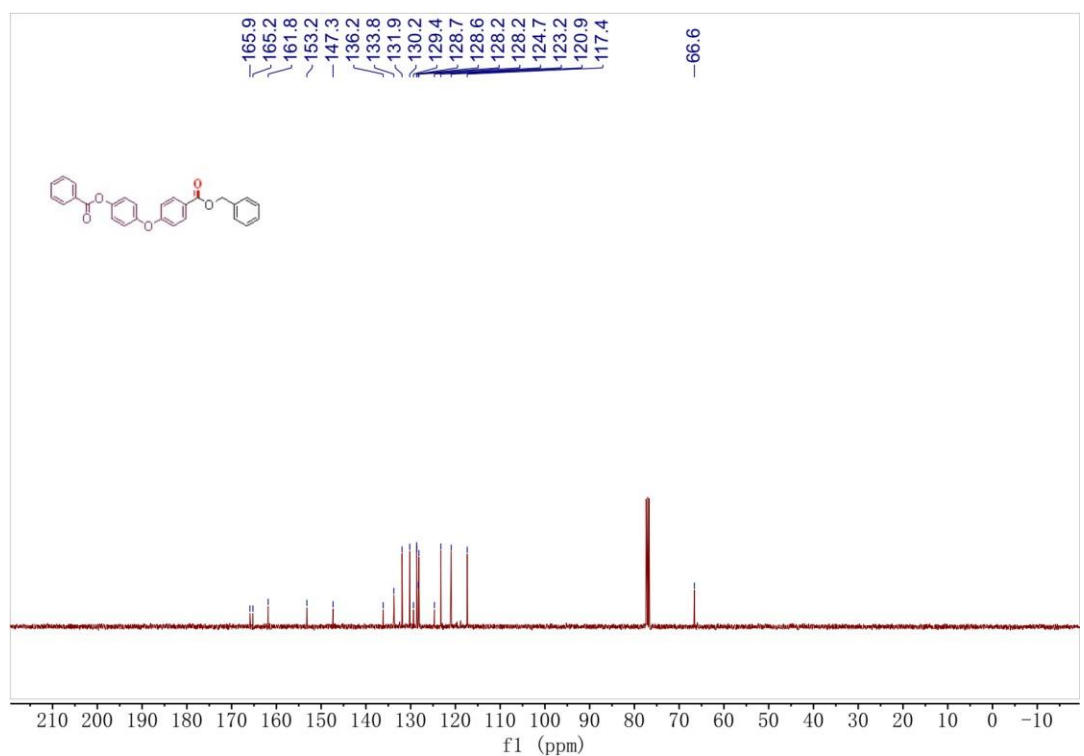
¹⁹F NMR (376 MHz, CDCl₃) 78



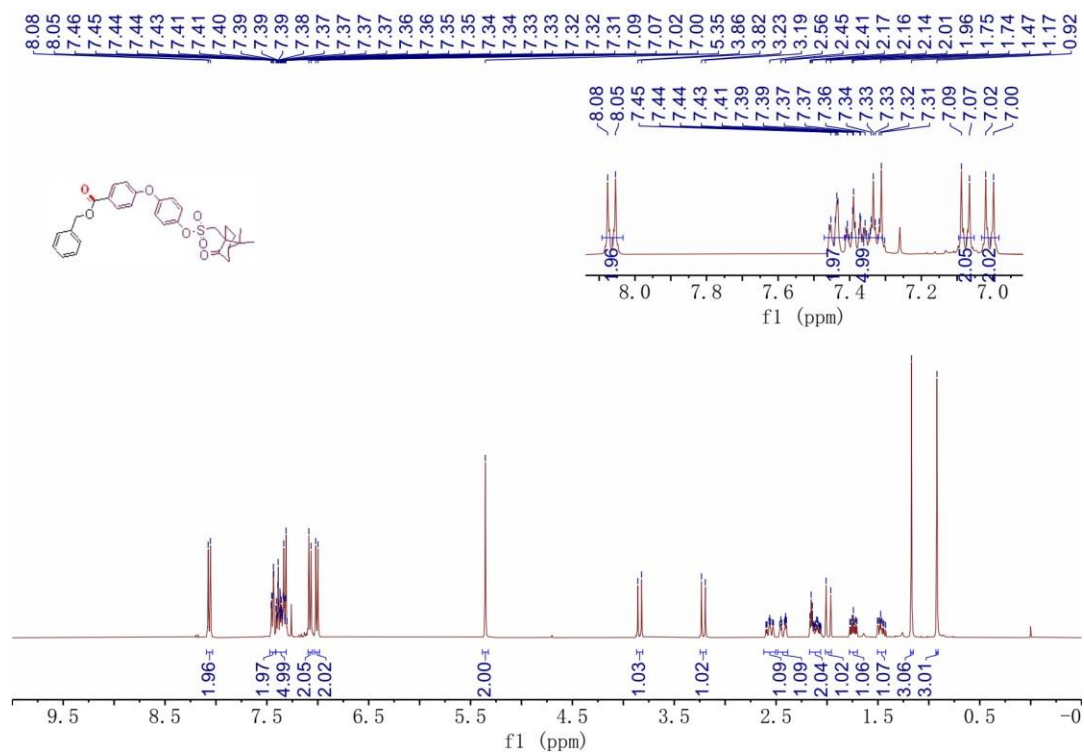
¹H NMR (400 MHz, CDCl₃) 79



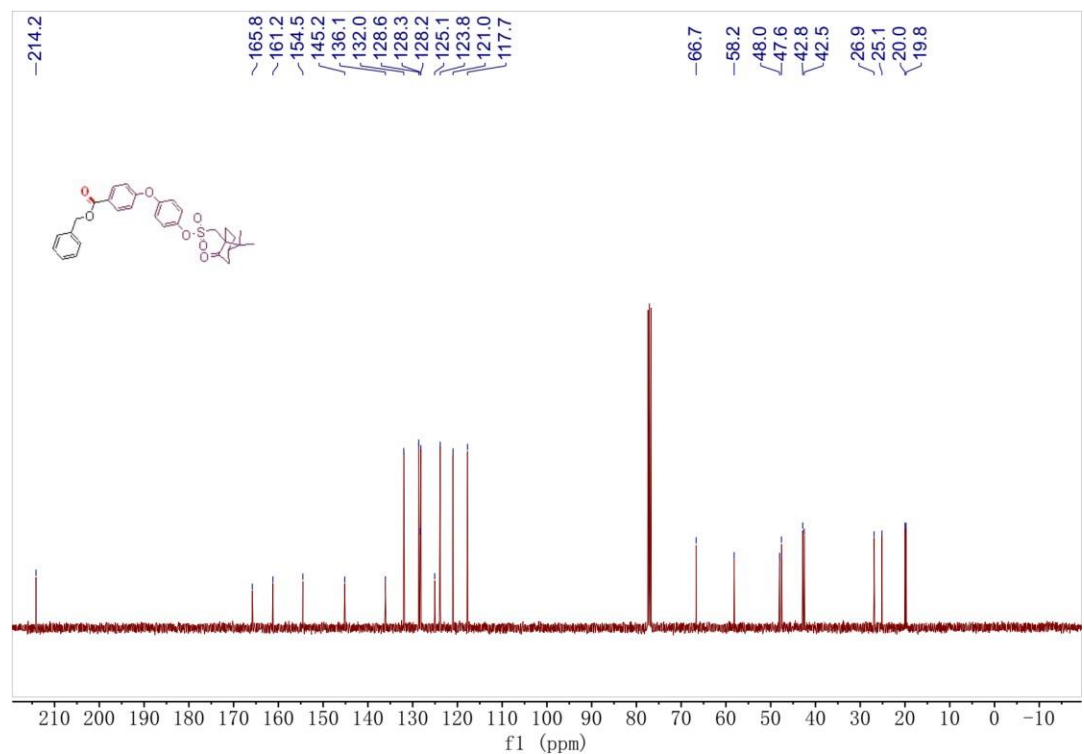
¹³C NMR (101 MHz, CDCl₃) 79



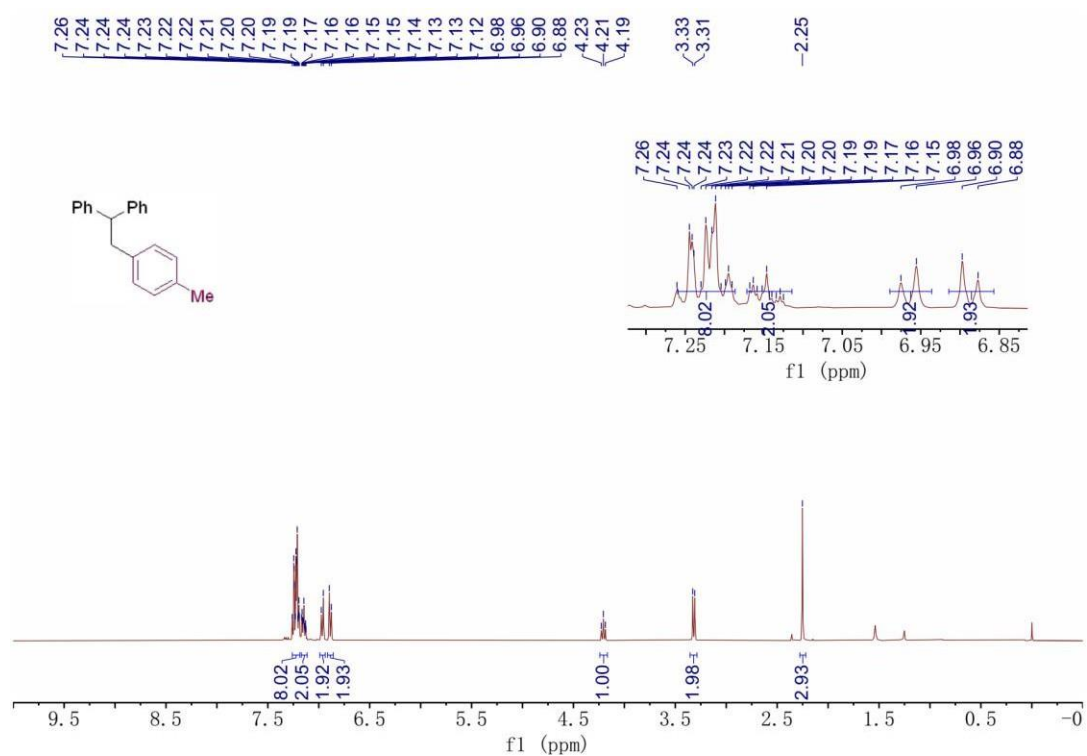
¹H NMR (400 MHz, CDCl₃) 80



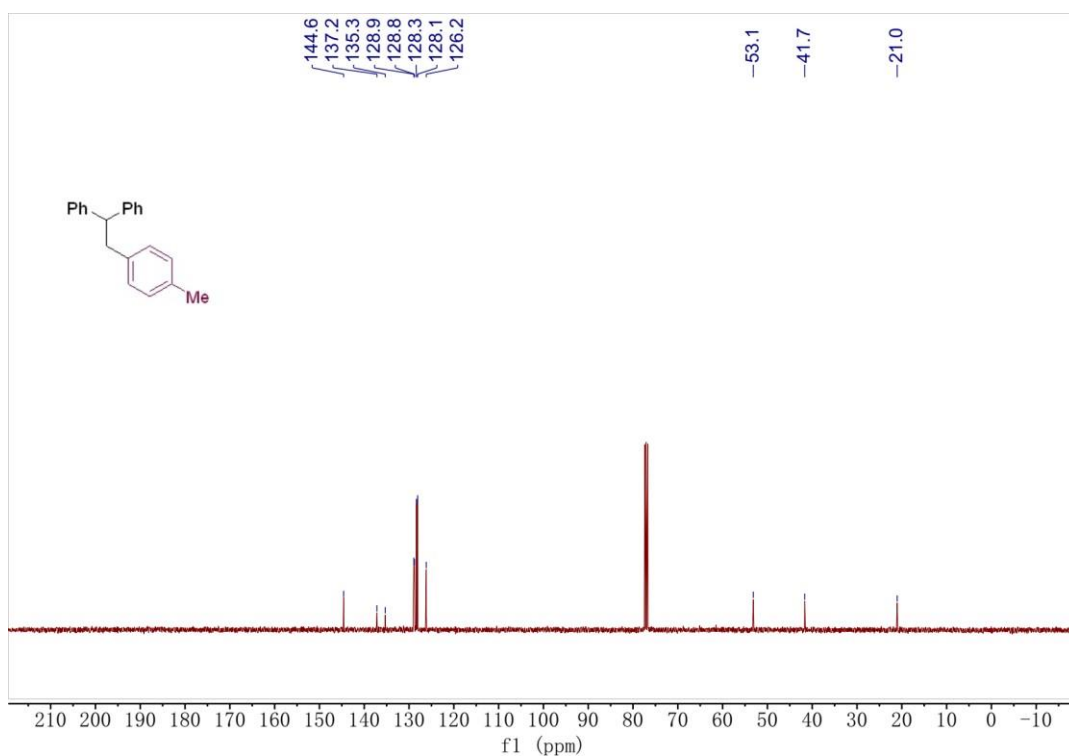
¹³C NMR (101 MHz, CDCl₃) 80



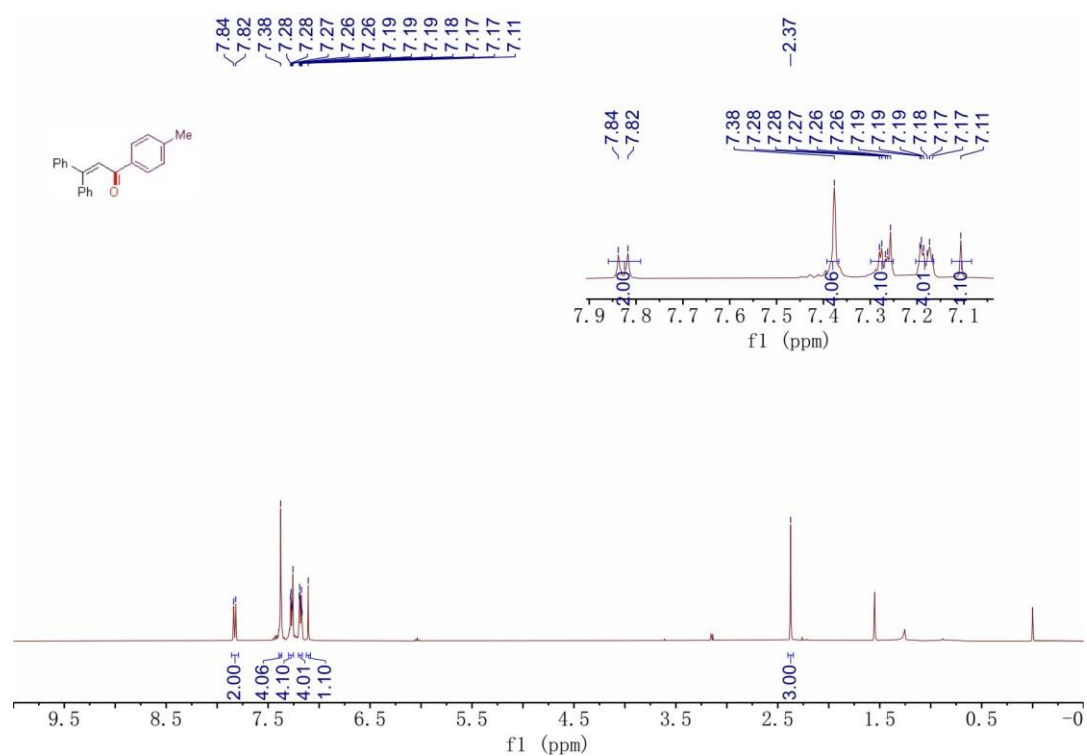
¹H NMR (400 MHz, CDCl₃) 8



¹³C NMR (101 MHz, CDCl₃) 82



¹H NMR (400 MHz, CDCl₃) 8



¹³C NMR (101 MHz, CDCl₃) 83

