

–Supporting Information–

Transition-metal-free carbonylation of aryl halides with arylboronic acids by utilizing stoichiometric CHCl_3 as the carbon monoxide-precursor

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Table of Contents

Content	Page
1. General Information	S3
2. Products from General Procedure for transition-metal-free carbonylation of aryl halides with arylboronic acids by utilizing stoichiometric CHCl_3 as the CO source	S3
3. Effect of A Free-Radical Probe	S5
4. Comparison of catalyst systems	S5
5. Analytical Data of Products	S7
6. References	S18
7. Copies of NMR Spectra	S19

1. General Information

Reagent Information. All the aryl halides and the arylboronic acids were purchased from Alfa Aesar, Energy Chemical, Beijing InnoChem Science & Technology Co., Ltd., and Accela ChemBio Co., Ltd. and were used as received. Glycol and PEG-400 (bought from Acros, Energy Chemical, and Aladdin) was pre-dried (toluene azeotrope) and deoxygenated. The following reagents were used: Na₂CO₃ (99.5%, Alfa Aesar), Na₂CO₃ (99.997%, Alfa Aesar), CsOH·H₂O (99%, Energy Chemical), CsOH·H₂O (99.95%, Sigma Aldrich), NaI (99%, Alfa Aesar), NaI (99.99%, Acros) and PivOH (99%, Alfa Aesar).

Physical Methods. ¹H and ¹³C NMR spectra of solutions in CDCl₃ or CD₃COCD₃ were recorded on a Bruker Avance 400 instrument. Chemical shifts were expressed in parts per million (ppm) downfield from tetramethylsilane and refer to the solvent signals (CDCl₃: H 7.26 and C 77.0 ppm; CD₃COCD₃: H 2.05 and C 29.84 ppm). The signals of water were observed at about 1.57 ppm in CDCl₃ and 2.84 ppm in CD₃COCD₃, respectively. Abbreviations for signal couplings are: br, broad; s, singlet; d, doublet; t, triplet; m, multiplet; dd, doublet of doublets; dt, triplet of doublets; td, doublet of triplets; tt, triplet of triplets; tdd, doublet of doublet of triplets. Coupling constants, *J*, were reported in hertz unit (Hz). Infrared spectra of neat substances were recorded on a BRUKER TENSOR 27 FT-IR spectrometer. HRMS was performed on a Bruker's solarix 94 (ESI-FTICR-MS) mass spectrometer. ICP-AES analysis was measured on a Prodigy (LEEMAN LABS INC.) machine. GC-MS were determined with Agilent 7890-5975C. Column chromatography was performed using silica gel 300-400 mesh (Yantai Jiangyou Silica Gel Co., Ltd., China) as the solid support. *No special safety precautions were taken when the reaction was performed in a sealed borosilicate 3.3 glass tube of 3.5 mm wall thickness, 20 mm inside diameter and 3 cm length.*

2. General Procedure for transition-metal-free carbonylation of aryl halides with arylboronic acids by utilizing stoichiometric CHCl₃ as the CO source

General Procedure A: With no precautions to exclude air or moisture, a 10-ml screw-cap vial equipped with a magnetic stir bar was charged with aryl halide (0.25 mmol), arylboronic acid (0.375 mmol), NaI (0.25 mmol, 37.8 mg), Na₂CO₃ (0.5 mmol, 53.3 mg), CsOH·H₂O (1.25 mmol, 212.0 mg), CHCl₃ (0.75

mmol, 61 μ L), PivOH (0.1875 mmol, 21 μ L), and Glycol (2.0 mL). The vial was capped and heated at 120 °C in a heating block for the indicated time. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (3 \times 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel (petroleum ether: diethyl ether = 100 : 1 to 10 : 1).

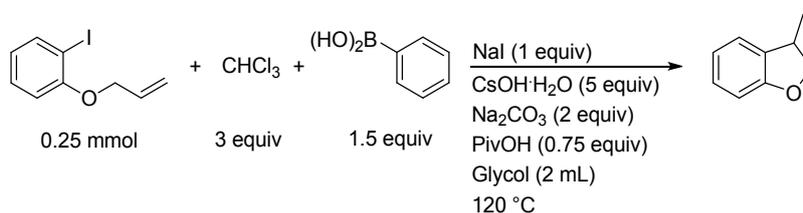
General Procedure B: With no precautions to exclude air or moisture, a 10-ml screw-cap vial equipped with a magnetic stir bar was charged with aryl halide (0.25 mmol), arylboronic acid (0.375 mmol), NaI (0.25 mmol, 37.8 mg), Na₂CO₃ (0.75 mmol, 80.0 mg), CsOH·H₂O (1.25 mmol, 212.0 mg), CHCl₃ (0.75 mmol, 61 μ L), PivOH (0.1875 mmol, 21 μ L), and Glycol (2.0 mL). The vial was capped and heated at 120 °C in a heating block for the indicated time. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (3 \times 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel (petroleum ether: diethyl ether = 100 : 1 to 10 : 1).

General Procedure C: With no precautions to exclude air or moisture, a 10-ml screw-cap vial equipped with a magnetic stir bar was charged with aryl halide (0.25 mmol), arylboronic acid (0.375 mmol), NaI (0.25 mmol, 37.8 mg), Na₂CO₃ (1.0 mmol, 106.6 mg), CsOH·H₂O (1.25 mmol, 212.0 mg), CHCl₃ (0.75 mmol, 61 μ L), PivOH (0.1875 mmol, 21 μ L), and Glycol (2.0 mL). The vial was capped and heated at 120 °C in a heating block for the indicated time. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (3 \times 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel (petroleum ether: diethyl ether = 100 : 1 to 10 : 1).

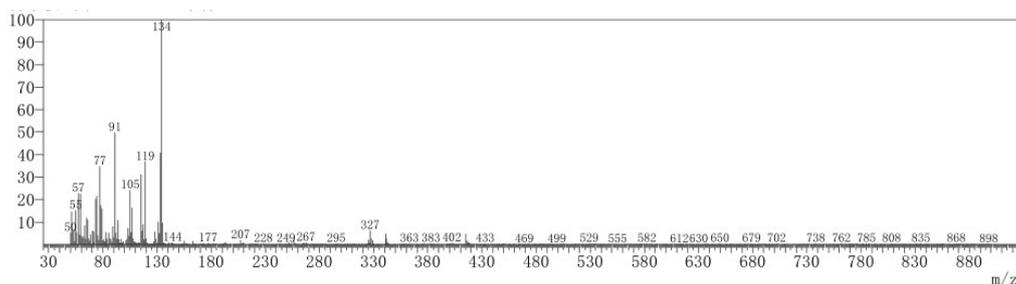
General Procedure D: With no precautions to exclude air or moisture, a 10-ml screw-cap vial equipped with a magnetic stir bar was charged with aryl halide (0.25 mmol), arylboronic acid (0.375 mmol), NaI (0.25 mmol, 37.8 mg), Na₂CO₃ (0.50 mmol, 53.3 mg), CsOH·H₂O (1.25 mmol, 212.0 mg), CHCl₃

(0.75 mmol, 61 μ L), PivOH (0.1875 mmol, 21 μ L), and PEG-400 (2.0 mL). The vial was capped and heated at 120 $^{\circ}$ C in a heating block for the indicated time. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (3 \times 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel (petroleum ether: diethyl ether = 100 : 1 to 10 : 1).

3. Effect of A Free-Radical Probe

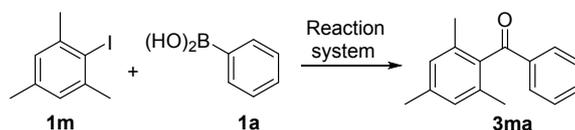


Following *general procedure A*, a cyclization product, 3-methyl-2,3-dihydrobenzofuran was obtained based on GC-MS analysis and no any carbonylated product was observed.



4. Comparison of catalyst systems

Table S1. Comparison of Catalyst Systems



Entry	Reaction system	Ref.	yield (%) ^a
1	Pd ₂ (dba) ₃ /CO	6h	72
2	Pd(OAc) ₂ /CHCl ₃	17	50
3	FeCl ₂ /CHCl ₃	16	6
4	CHCl ₃	This work	94

^a Isolated yields after column chromatography are given.

Entry 1: A 25 mL flask equipped with a magnetic stir bar was charged with **1m** (0.25 mmol, 62.8 mg),

2a (0.25 mmol, 31.4 mg), Pd₂(dba)₃ (0.0025 mmol, 2.3 mg), K₂CO₃ (0.75 mmol, 105.8 mg), and anisole (2.0 mL) before standard three cycles of evacuation and back-filling with dry and pure carbon monoxide. The mixture was then stirred at 100 °C for 48 h. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (3 × 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel to give the desired product **3ma** (40.3 mg, 72%).

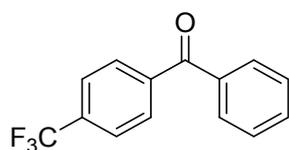
Entry 2: With no precautions to exclude air or moisture, a 10-ml screw-cap vial equipped with a magnetic stir bar was charged with **1m** (0.25 mmol, 62.8 mg), **2a** (0.30 mmol, 37.7 mg), Pd(OAc)₂ (0.005 mmol, 1.1 mg), DMAP (0.05 mmol, 6.2 mg), KOH (1.5 mmol, 85.9 mg), CHCl₃ (0.75 mmol, 61 μL), and toluene (2.0 mL). The vial was capped and heated at 80 °C in a heating block for 48 h. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (3 × 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel to give the desired product **3ma** (27.9 mg, 50%).

Entry 3: With no precautions to exclude air or moisture, a 10-ml screw-cap vial equipped with a magnetic stir bar was charged with **1m** (0.25 mmol, 62.8 mg), **2a** (0.375 mmol, 47.1 mg), FeCl₂ (0.025 mmol, 3.2 mg), NaI (0.125 mmol, 18.9 mg), Na₂CO₃ (0.50 mmol, 53.3 mg), CsOH·H₂O (1.25 mmol, 212.0 mg), CHCl₃ (0.75 mmol, 61 μL), PivOH (0.375 mmol, 42 μL), and PEG-400 (2.0 mL). The vial was capped and heated at 120 °C in a heating block for 48 h. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (3 × 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel to give the desired product **3ma** in 6% yield.

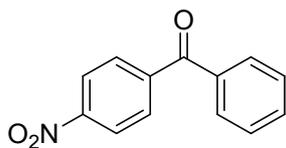
Entry 4: With no precautions to exclude air or moisture, a 10-ml screw-cap vial equipped with a magnetic stir bar was charged with **1m** (0.25 mmol, 62.8 mg), **2a** (0.375 mmol, 47.1 mg), NaI (0.25 mmol, 37.8 mg), Na₂CO₃ (0.75 mmol, 80.0 mg), CsOH·H₂O (1.25 mmol, 212.0 mg), CHCl₃ (0.75

mmol, 61 μ L), PivOH (0.1875 mmol, 21 μ L), and Glycol (2.0 mL). The vial was capped and heated at 120 °C in a heating block for 48 h. The vial was capped and heated at 80 °C in a heating block for 48 h. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (3 \times 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel to give the desired product **3ma** (52.6 mg, 94%).

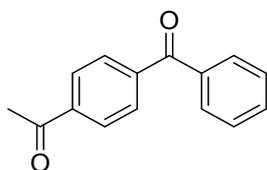
5. Analytical Data of Products



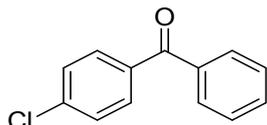
Phenyl(4-(trifluoromethyl)phenyl)methanone (3aa): Following *general procedure A*, **3aa** was isolated as a white solid (53.1 mg, 85%), known compound. The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 7.90 (d, J = 8.0 Hz, 2 H), 7.82-7.79 (m, 2 H), 7.76 (d, J = 8.0 Hz, 2 H), 7.63 (tt, J = 7.2 Hz, 1.0 Hz, 1 H), 7.51 ppm (t, J = 7.6 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 195.5, 140.7 (d, J = 1 Hz), 136.7, 133.7 (q, J = 32 Hz), 133.1, 130.12, 130.09, 128.5, 125.3 (q, J = 3 Hz), 123.7 ppm (q, J = 271 Hz); ¹⁹F NMR (400 MHz, CDCl₃): δ -63.0 ppm; Mp: 114.2-115.1 °C.



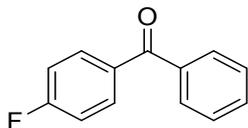
(4-Nitrophenyl)(phenyl)methanone (3ba): Following *general procedure A*, **3ba** was isolated as light yellow solid (48.2 mg, 85%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 8.34 (d, J = 8.8 Hz, 2 H), 7.94 (d, J = 8.8 Hz, 2 H), 7.81-7.79 (m, 2 H), 7.66 (t, J = 7.6 Hz, 1 H), 7.53 ppm (t, J = 7.6 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 194.8, 149.8, 142.8, 136.2, 133.5, 130.7, 130.1, 128.7, 123.5 ppm; Mp: 135.3-136.1 °C.



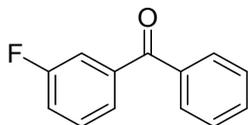
1-(4-Benzoylphenyl)ethanone (3ca): Following *general procedure B*, **3ca** was isolated as light yellow solid (36.4 mg, 65%), known compound; The NMR spectroscopic data agree with those described in ref.^[S2]. ¹H NMR (400 MHz, CDCl₃): δ 8.06 (d, *J* = 8.0 Hz, 2 H), 7.87 (d, *J* = 8.0 Hz, 2 H), 7.81-7.79 (m, 2 H), 7.63 (t, *J* = 7.6 Hz, 1 H), 7.50 (t, *J* = 7.6 Hz, 2 H), 2.67 ppm (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 197.6, 196.0, 141.2, 139.5, 136.8, 133.0, 130.1, 130.0, 128.5, 128.1, 26.9 ppm; Mp: 79.5-80.7 °C.



(4-Chlorophenyl)(phenyl)methanone (3da): Following *general procedure A*, **3da** was isolated as a white solid (47.5 mg, 88%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 7.79-7.74 (m, 4 H), 7.61 (tt, *J* = 7.2 Hz, 1.2 Hz, 1 H), 7.51-7.45 ppm (m, 4 H); ¹³C NMR (100 MHz, CDCl₃): δ 195.5, 138.9, 137.2, 135.9, 132.6, 131.4, 129.9, 128.6, 128.4 ppm; Mp: 71.7-73.2 °C.

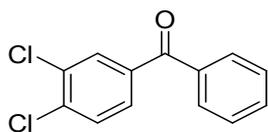


(4-Fluorophenyl)(phenyl)methanone (3ea): Following *general procedure A*, **3ea** was isolated as a light yellow oil (46.5 mg, 93%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 7.85-7.83 (m, 2 H), 7.78-7.76 (m, 2 H), 7.60 (td, *J* = 7.2 Hz, 1.2 Hz, 1 H), 7.49 (t, *J* = 8.4 Hz, 2 H), 7.17 ppm (td, *J* = 8.8 Hz, 1.6 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 195.3, 165.4 (d, *J* = 253 Hz), 137.5, 133.7 (d, *J* = 4 Hz), 132.7, 132.6 (d, *J* = 14 Hz), 129.9, 128.3, 115.5 ppm (d, *J* = 22 Hz); ¹⁹F NMR (400 MHz, CDCl₃): δ -106.0 ppm.

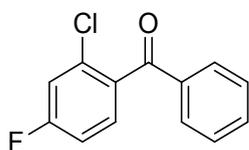


(3-Fluorophenyl)(phenyl)methanone (3fa): Following *general procedure A*, **3fa** was isolated as a yellow oil (46.0 mg, 92%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 7.80 (dt, *J* = 7.6 Hz, 1.6 Hz, 2 H), 7.63-7.56 (m, 2 H), 7.52-7.44 (m, 4 H), 7.29 ppm (tdd, *J* = 8.2 Hz, 2.4 Hz, 0.8 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃): δ 195.3 (d, *J* = 2 Hz), 162.5 (d, *J* = 247 Hz), 139.6 (d, *J* = 6 Hz), 137.0, 132.8, 130.0, 129.9, 128.4, 125.8 (d, *J*

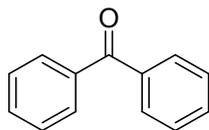
= 3 Hz), 119.4 (d, $J = 21$ Hz), 116.7 ppm (d, $J = 22$ Hz); ^{19}F NMR (400 MHz, CDCl_3): δ -112.0 ppm.



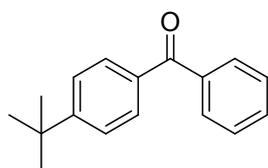
(3,4-Dichlorophenyl)(phenyl)methanone (3ga): Following *general procedure B*, **3ga** was isolated as a white solid (56.3 mg, 90%), known compound; The NMR spectroscopic data agree with those described in ref.^[S3]. ^1H NMR (400 MHz, CDCl_3): δ 7.89 (d, $J = 2.0$ Hz, 1 H), 7.78-7.76(m, 2 H), 7.65-7.61 (m, 2 H), 7.57 (d, $J = 8.4$ Hz, 1 H), 7.51 ppm (t, $J = 7.6$ Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3): δ 194.3, 137.1, 137.0, 136.6, 132.98, 132.96, 131.8, 130.4, 129.9, 129.1, 128.5 ppm; Mp: 99.7-100.4 °C.



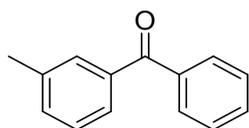
(2-Chloro-4-fluorophenyl)(phenyl)methanone (3ha): Following *general procedure A*, **3ha** was isolated as a yellow oil (54.4 mg, 93%), known compound; The NMR spectroscopic data agree with those described in ref.^[S4]. ^1H NMR (400 MHz, CDCl_3): δ 7.81-7.78 (m, 2 H), 7.62 (t, $J = 7.6$ Hz, 1 H), 7.48 (t, $J = 7.6$ Hz, 2 H), 7.40 (dd, $J = 8.4$ Hz, 6.0 Hz, 1 H), 7.22 (dd, $J = 8.4$ Hz, 2.4 Hz, 1 H), 7.10 ppm (td, $J = 8.4$ Hz, 2.4 Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 194.4, 163.3 (d, $J = 252$ Hz), 136.4, 134.6 (d, $J = 4$ Hz), 133.8, 132.9 (d, $J = 11$ Hz), 130.9 (d, $J = 9$ Hz), 130.0, 128.6, 117.6 (d, $J = 24$ Hz), 114.2 ppm (d, $J = 22$ Hz); ^{19}F NMR (400 MHz, CDCl_3): δ -107.8 ppm.



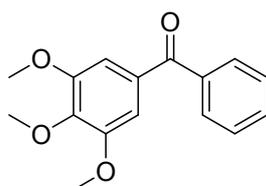
Benzophenone (3ia): Following *general procedure A*, **3ia** was isolated as a white solid (41.4 mg, 91%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ^1H NMR (400 MHz, CDCl_3): δ 7.81 (dt, $J = 8.0$ Hz, 1.6 Hz, 4 H), 7.60 (tt, $J = 7.2$ Hz, 1.2 Hz, 2 H), 7.49 ppm (t, $J = 7.6$ Hz, 4 H); ^{13}C NMR (100 MHz, CDCl_3): δ 196.8, 137.5, 132.4, 130.0, 128.2 ppm; Mp: 45.8-46.1 °C.



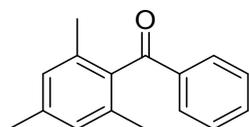
(4-(*tert*-Butyl)phenyl)(phenyl)methanone (3ja): Following *general procedure A*, **3ja** was isolated as a white solid with low melting point (42.8 mg, 72%), known compound; The NMR spectroscopic data agree with those described in ref.^[S5]. ¹H NMR (400 MHz, CDCl₃): δ 7.81-7.75 (m, 4 H), 7.58(tt, *J* = 7.6 Hz, 1.2 Hz, 1 H), 7.51-7.46 (m, 4 H), 1.37 ppm (s, 9 H); ¹³C NMR (100 MHz, CDCl₃): δ 196.5, 156.2, 137.9, 134.8, 132.2, 130.1, 130.0, 128.2, 125.2, 35.1, 31.1 ppm.



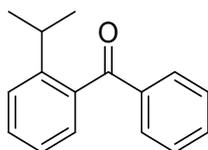
Phenyl(*m*-tolyl)methanone (3ka): Following *general procedure A*, **3ka** was isolated as a yellow oil (42.6 mg, 87%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 7.81-7.79 (m, 2 H), 7.63-7.57 (m, 3 H), 7.48 (t, *J* = 7.6 Hz, 2 H), 7.41 (d, *J* = 7.6 Hz, 1 H), 7.36 (t, *J* = 7.6 Hz, 1 H), 2.42 ppm (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 197.0, 138.1, 137.7, 137.6, 133.2, 132.3, 130.4, 130.0, 128.2, 128.1, 127.3, 21.3 ppm.



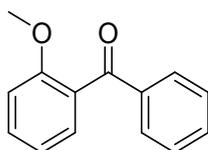
Phenyl(3,4,5-trimethoxyphenyl)methanone (3la): Following *general procedure B*, **3la** was isolated as a white solid with low melting point (51.0 mg, 75%), known compound; The NMR spectroscopic data agree with those described in ref.^[S6]. ¹H NMR (400 MHz, CDCl₃): δ 7.81-7.78 (m, 2 H), 7.59 (tt, *J* = 7.6 Hz, 1.2 Hz, 1 H), 7.51-7.47 (m, 2 H), 7.06 (s, 2 H), 3.93 (s, 3 H), 3.87 ppm (s, 6 H); ¹³C NMR (100 MHz, CDCl₃): δ 195.8, 152.8, 141.9, 137.7, 132.5, 132.3, 129.8, 128.2, 107.6, 60.9, 56.2 ppm.



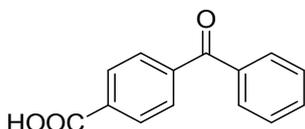
Mesityl(phenyl)methanone (3ma): Following *general procedure B*, **3ma** was isolated as a white solid with low melting point (52.6 mg, 94%), known compound; The NMR spectroscopic data agree with those described in ref.^[S7]. ¹H NMR (400 MHz, CDCl₃): δ 7.82-7.80 (m, 2 H), 7.58 (tt, *J* = 7.6 Hz, 1.6 Hz, 1 H), 7.44 (t, *J* = 8.0 Hz, 2 H), 6.90 (s, 2 H), 2.34 (s, 3 H), 2.09 ppm (s, 6 H); ¹³C NMR (100 MHz, CDCl₃): δ 200.8, 138.4, 137.2, 136.8, 134.1, 133.5, 129.3, 128.7, 128.3, 21.1, 19.3 ppm.



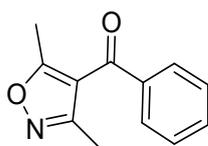
(2-Isopropylphenyl)(phenyl)methanone (3na): Following *general procedure B*, **3na** was isolated as a yellow oil (45.9 mg, 82%), known compound (CAS: 19103-09-4). ^1H NMR (400 MHz, CDCl_3): δ 7.83-7.81 (m, 2 H), 7.59 (tt, $J = 7.2$ Hz, 1.2 Hz, 1 H), 7.48-7.44 (m, 4 H), 7.25-7.20 (m, 2 H), 3.09-2.99 (m, 1 H), 1.19 ppm (d, $J = 6.8$ Hz, 6 H); ^{13}C NMR (100 MHz, CDCl_3): δ 199.1, 147.1, 138.3, 137.7, 133.3, 130.1, 130.1, 128.4, 127.5, 126.0, 125.1, 30.2, 24.1 ppm.



(2-Methoxyphenyl)(phenyl)methanone (3oa): Following *general procedure B*, **3oa** was isolated as a yellow solid with low melting point (42.9 mg, 81%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ^1H NMR (400 MHz, CDCl_3): δ 7.83-7.80 (m, 2 H), 7.55 (tt, $J = 7.2$ Hz, 1.2 Hz, 1 H), 7.50-7.41 (m, 3 H), 7.36 (dd, $J = 7.6$ Hz, 1.6 Hz, 1 H), 7.04 (t, $J = 7.2$ Hz, 1 H), 7.00 (d, $J = 8.4$ Hz, 1 H), 3.73 ppm (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 196.5, 157.3, 137.7, 132.9, 131.9, 129.8, 129.6, 128.7, 128.2, 120.4, 111.4, 55.6 ppm.

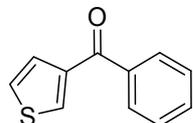


4-Benzoylbenzoic acid (3pa): Following *general procedure C*, **3pa** was isolated as a light white solid (33.9 mg, 60%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ^1H NMR (400 MHz, CDCl_3): δ 8.24 (dd, $J = 6.4$ Hz, 2.0 Hz, 2 H), 7.88 (dd, $J = 6.8$ Hz, 2.0 Hz, 2 H), 7.84-7.81 (m, 2 H), 7.64 (tt, $J = 7.2$ Hz, 1.2 Hz, 1 H), 7.52 ppm (t, $J = 7.6$ Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3): δ 196.0, 170.8, 142.1, 136.7, 133.1, 132.2, 130.14, 130.12, 129.8, 128.5 ppm; Mp: 195.3-196.1 $^\circ\text{C}$.

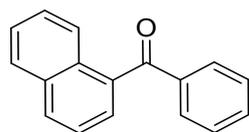


(3,5-Dimethylisoxazol-4-yl)(phenyl)methanone (3qa): Following *general procedure B*, **3qa** was isolated as a yellow solid (40.7 mg, 81%), known compound; The NMR spectroscopic data agree with

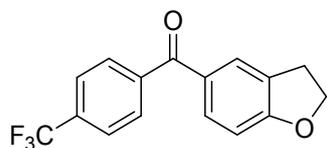
those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 7.72-7.70 (m, 2 H), 7.61 (tt, *J* = 7.6 Hz, 1.2 Hz, 1 H), 7.50 (t, *J* = 8.0 Hz, 2 H), 2.32 (s, 3 H), 2.30 ppm (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 190.4, 172.3, 159.6, 138.3, 133.2, 128.9, 128.7, 116.4, 13.3, 11.3 ppm; Mp: 55.2-56.9 °C.



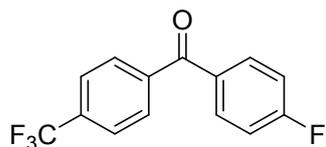
Phenyl(thiophen-3-yl)methanone (3ra): Following *general procedure B*, **3ra** was isolated as a light yellow oil (31.5 mg, 67%), known compound. The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 7.93 (dd, *J* = 2.8 Hz, 1.2 Hz, 1 H), 7.86-7.84 (m, 2 H), 7.58 (dd, *J* = 2.8 Hz, 1.2 Hz, 1 H), 7.55 (dt, *J* = 7.6 Hz, 1.2 Hz, 1 H), 7.47 (t, *J* = 8.0 Hz, 2 H); 7.36 ppm (dd, *J* = 5.2 Hz, 2.8 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃): δ 190.0, 141.2, 138.5, 134.0, 132.3, 129.3, 128.6, 128.3, 126.2 ppm.



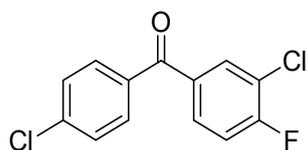
Naphthalen-1-yl(phenyl)methanone (3sa): Following *general procedure B*, **3sa** was isolated as a white solid (46.4 mg, 80%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 8.11 (dd, *J* = 7.6 Hz, 0.8 Hz, 1 H), 8.01 (d, *J* = 8.0 Hz, 1 H), 7.93 (dd, *J* = 7.2 Hz, 2.0 Hz, 1 H), 7.87 (dt, *J* = 8.4 Hz, 1.2 Hz, 2 H), 7.63-7.45 ppm (m, 7 H); ¹³C NMR (100 MHz, CDCl₃): δ 198.0, 138.2, 136.2, 133.6, 133.2, 131.2, 130.9, 130.4, 128.40, 128.35, 127.8, 127.2, 126.4, 125.6, 124.3 ppm; Mp: 68.3-69.8 °C.



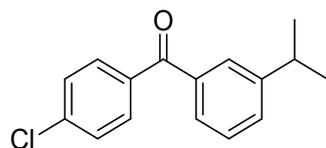
(2,3-Dihydrobenzofuran-5-yl)(4-(trifluoromethyl)phenyl)methanone (3ab): Following *general procedure D*, **3ab** was isolated as a yellow solid (62.1 mg, 85%), known compound (CAS: 1094286-50-6). ¹H NMR (400 MHz, CDCl₃): δ 7.82 (d, *J* = 8.0 Hz, 2 H), 7.73 (d, *J* = 8.8 Hz, 3 H), 7.63 (dd, *J* = 8.4 Hz, 1.2 Hz, 1 H), 6.83 (d, *J* = 8.4 Hz, 1 H), 4.69 (t, *J* = 8.4 Hz, 2 H), 3.27 ppm (t, *J* = 8.4 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 194.3, 164.7, 141.8, 133.1 (q, *J* = 32 Hz), 132.6, 129.7, 129.6, 127.9, 127.4, 125.2 (q, *J* = 4 Hz), 123.7 (q, *J* = 271 Hz), 109.0, 72.3, 28.9 ppm; ¹⁹F NMR (400 MHz, CDCl₃): δ -62.9 ppm; Mp: 144.9-145.7 °C.



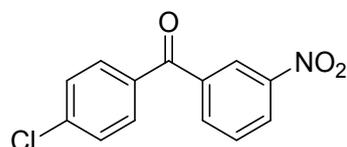
(4-Fluorophenyl)(4-(trifluoromethyl)phenyl)methanone (3ac): Following *general procedure A*, **3ac** was isolated as a white solid (54.3 mg, 81%), known compound; The NMR spectroscopic data agree with those described in ref.^[S8]. ¹H NMR (400 MHz, CDCl₃): δ 7.88-7.83 (m, 4 H), 7.76 (d, *J* = 8.4 Hz, 2 H), 7.19 ppm (tt, *J* = 8.4 Hz, 2.0 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 194.1, 165.7 (d, *J* = 254 Hz), 140.5 (d, *J* = 1 Hz), 133.8 (q, *J* = 32 Hz), 132.9 (d, *J* = 3 Hz), 132.7 (d, *J* = 9 Hz), 130.0, 125.4 (q, *J* = 4 Hz), 123.6 (q, *J* = 271 Hz), 115.8 ppm (d, *J* = 22 Hz); ¹⁹F NMR (400 MHz, CDCl₃): δ -63.0, -104.6 ppm ; Mp: 96.0-97.3 °C.



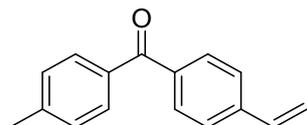
(3-Chloro-4-fluorophenyl)(4-chlorophenyl)methanone (3dd): Following *general procedure A*, **3dd** was isolated as a yellow solid (56.1 mg, 84%), known compound (CAS: 951890-04-3). ¹H NMR (400 MHz, CDCl₃): δ 7.87 (dd, *J* = 7.2 Hz, 2.0 Hz, 1 H), 7.74-7.67 (m, 3 H), 7.48 (dt, *J* = 8.4 Hz, 2.0 Hz, 2 H), 7.26 ppm (t, *J* = 8.4 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃): δ 192.8, 160.8 (d, *J* = 256 Hz), 139.4, 135.0, 134.2 (d, *J* = 4 Hz), 132.6 (d, *J* = 1 Hz), 131.2, 130.2 (d, *J* = 9 Hz), 128.9, 121.7 (d, *J* = 19 Hz), 116.7 ppm (d, *J* = 22 Hz); ¹⁹F NMR (400 MHz, CDCl₃): δ -107.8 ppm ; Mp: 75.5-76.6 °C.



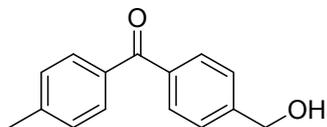
(4-Chlorophenyl)(3-isopropylphenyl)methanone (3de): Following *general procedure A*, **3de** was isolated as a yellow oil (47.1 mg, 73%), known compound (CAS: 343221-62-5). ¹H NMR (400 MHz, CDCl₃): δ 7.76 (dt, *J* = 8.4 Hz, 2.0 Hz, 2 H), 7.66 (s, 1 H), 7.55 (dt, *J* = 7.6 Hz, 1.6 Hz, 1 H), 7.48-7.45 (m, 3 H), 7.40 (t, *J* = 7.6 Hz, 1 H), 3.03-2.93 (m, 1 H), 1.28 ppm (d, *J* = 7.2 Hz, 6 H); ¹³C NMR (100 MHz, CDCl₃): δ 195.8, 149.3, 138.7, 137.2, 136.0, 131.5, 130.9, 128.6, 128.2, 127.9, 127.7, 34.0, 23.9 ppm.



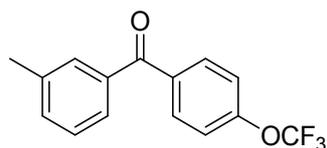
(4-Chlorophenyl)(3-nitrophenyl)methanone (3df): Following *general procedure A*, **3df** was isolated as a white solid (44.4 mg, 68%), known compound; The NMR spectroscopic data agree with those described in ref.^[S9]. ¹H NMR (400 MHz, CDCl₃): δ 8.59 (t, *J* = 2.0 Hz, 1 H), 8.46 (dq, *J* = 8.4 Hz, 1.2 Hz, 1 H), 8.11 (dt, *J* = 7.6 Hz, 1.2 Hz, 1 H), 7.77-7.70 (m, 3 H), 7.51 ppm (dt, *J* = 8.8 Hz, 2.0 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 193.0, 148.0, 140.0, 138.6, 135.3, 134.4, 131.4, 129.8, 129.1, 126.9, 124.6 ppm; Mp: 87.5-88.7 °C.



***p*-Tolyl(4-vinylphenyl)methanone (3tg):** Following *general procedure A*, **3tg** was isolated as a white solid (38.9 mg, 70%), known compound (CAS: 24993-89-3). ¹H NMR (400 MHz, CDCl₃): δ 7.77 (dd, *J* = 6.8 Hz, 1.6 Hz, 2 H), 7.71 (d, *J* = 8.0 Hz, 2 H), 7.50 (d, *J* = 8.0 Hz, 2 H), 7.28 (d, *J* = 8.0 Hz, 2 H), 6.78 (dd, *J* = 17.6 Hz, 10.8 Hz, 1 H), 5.89 (d, *J* = 17.6 Hz, 1 H), 5.40 (d, *J* = 10.8 Hz, 1 H), 2.44 ppm (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 196.0, 143.1, 141.2, 137.0, 136.0, 134.9, 130.4, 130.2, 128.9, 125.9, 116.4, 21.7 ppm; Mp: 61.4-62.3 °C.

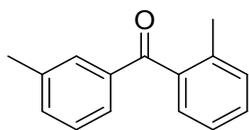


(4-(Hydroxymethyl)phenyl)(*p*-tolyl)methanone (3th): Following *general procedure A*, **3th** was isolated as a yellow solid (46.9 mg, 83%), known compound; The NMR spectroscopic data agree with those described in ref.^[S1]. ¹H NMR (400 MHz, CDCl₃): δ 7.78 (d, *J* = 8.0 Hz, 2 H), 7.71 (d, *J* = 8.4 Hz, 2 H), 7.47 (d, *J* = 8.0 Hz, 2 H), 7.28 (d, *J* = 8.0 Hz, 2 H), 4.80 (s, 2 H), 2.44 ppm (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 196.2, 145.1, 143.3, 137.1, 134.9, 130.3, 130.2, 129.0, 126.4, 64.7, 21.7 ppm; Mp: 83.6-84.3 °C.

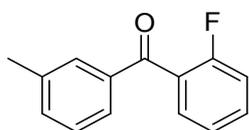


***m*-Tolyl(4-(trifluoromethoxy)phenyl)methanone (3ki):** Following *general procedure A*, **3ki** was isolated as a white solid (63.0 mg, 90%), known compound (CAS: 54362-81-1). ¹H NMR (400 MHz, CDCl₃): δ 7.86 (dt, *J* = 8.8 Hz, 2.4 Hz, 2 H), 7.61 (s, 1 H), 7.55 (d, *J* = 7.2 Hz, 1 H), 7.42 (d, *J* = 7.6 Hz, 1 H), 7.38 (t, *J* = 7.6 Hz, 1 H), 7.33-7.30 (m, 2 H), 2.43 ppm (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ

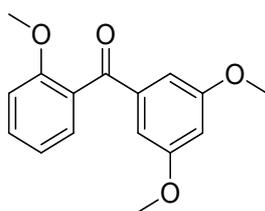
195.4, 152.0 (d, $J = 2$ Hz), 138.4, 137.1, 135.9, 133.5, 131.9, 130.3, 128.2, 127.2, 120.3 (q, $J = 257$ Hz), 120.2, 21.4 ppm; ^{19}F NMR (400 MHz, CDCl_3): δ -57.6 ppm; Mp: 56.4-57.8 °C.



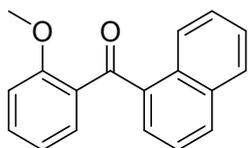
***m*-Tolyl(*o*-tolyl)methanone (3kj):** Following *general procedure A*, **3kj** was isolated as a yellow oil (39.4 mg, 75%), known compound; The NMR spectroscopic data agree with those described in ref.^[S10]. ^1H NMR (400 MHz, CDCl_3): δ 7.65 (s, 1 H), 7.56 (d, $J = 7.6$ Hz, 1 H), 7.41-7.23 (m, 6 H), 2.40 (s, 3 H), 2.33 ppm (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 198.9, 138.7, 138.3, 137.7, 136.6, 133.9, 130.9, 130.4, 130.1, 128.4, 128.3, 127.5, 125.1, 21.3, 20.0 ppm.



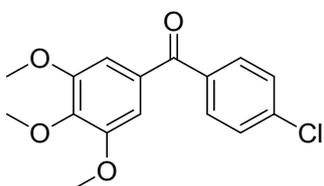
(2-Fluorophenyl)(*m*-tolyl)methanone (3kk): Following *general procedure A*, **3kk** was isolated as a yellow oil (41.2 mg, 77%), known compound (CAS: 726158-58-3). ^1H NMR (400 MHz, CDCl_3): δ 7.67 (s, 1 H), 7.60 (d, $J = 7.6$ Hz, 1 H), 7.56-7.50 (m, 2 H), 7.42 (d, $J = 7.6$ Hz, 1 H), 7.35 (t, $J = 7.6$ Hz, 1 H), 7.26 (td, $J = 7.6$ Hz, 0.8 Hz, 1 H), 7.16 (t, $J = 8.8$ Hz, 1 H), 2.41 ppm (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.7, 160.1 (d, $J = 251$ Hz), 138.3, 137.4, 134.2, 132.9 (d, $J = 8$ Hz), 130.7 (d, $J = 3$ Hz), 130.1, 128.3, 127.2 (d, $J = 1$ Hz), 124.2 (d, $J = 3$ Hz), 116.2 (d, $J = 22$ Hz), 100.0, 21.3 ppm; ^{19}F NMR (400 MHz, CDCl_3): δ -111.3 ppm.



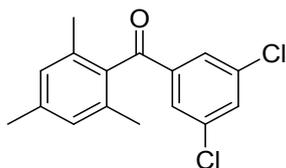
(3,5-Dimethoxyphenyl)(2-methoxyphenyl)methanone (3ol): Following *general procedure A*, **3ol** was isolated as a yellow oil (47.6 mg, 70%), known compound (CAS: 757961-86-7). ^1H NMR (400 MHz, CDCl_3): δ 7.47-7.43 (m, 1 H), 7.32 (dd, $J = 7.2$ Hz, 2.0 Hz, 1 H), 7.02 (td, $J = 7.6$ Hz, 0.8 Hz, 1 H), 6.98 (d, $J = 8.4$ Hz, 1 H), 6.96 (d, $J = 2.0$ Hz, 2 H), 6.65 (t, $J = 2.4$ Hz, 1 H), 3.80 (s, 6 H), 3.75 ppm (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 196.1, 160.5, 157.2, 139.6, 131.7, 129.3, 128.7, 120.3, 111.4, 107.6, 105.4, 55.6, 55.5 ppm.



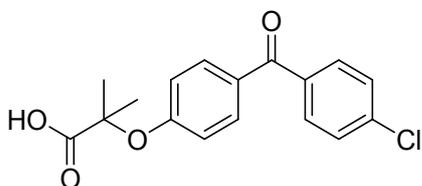
(2-Methoxyphenyl)(naphthalen-1-yl)methanone (30m): Following *general procedure A*, **30m** was isolated as a yellow solid (49.8 mg, 76%), known compound; The NMR spectroscopic data agree with those described in ref.^[S11]. ¹H NMR (400 MHz, CDCl₃): δ 8.62 (d, *J* = 8.4 Hz, 1 H), 7.98 (d, *J* = 8.4 Hz, 1 H), 7.92-7.89 (m, 1 H), 7.61-7.48 (m, 5 H), 7.42 (dd, *J* = 8.4 Hz, 7.6 Hz, 1 H), 7.04 (td, *J* = 7.2 Hz, 0.8 Hz, 1 H), 6.97 (d, *J* = 8.4 Hz, 1 H), 3.61 ppm (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 197.9, 158.3, 136.7, 133.7, 132.8, 132.2, 130.8, 130.7, 129.9, 129.8, 128.3, 127.6, 126.2, 125.9, 124.3, 120.4, 111.8, 55.6 ppm; Mp: 73.8-74.7 °C.



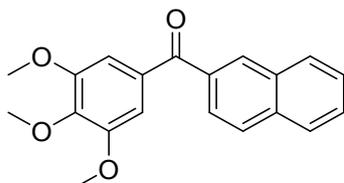
(4-Chlorophenyl)(3,4,5-trimethoxyphenyl)methanone (31n): Following *general procedure A*, **31n** was isolated as a white solid (50.5 mg, 66%), known compound; The NMR spectroscopic data agree with those described in ref.^[S12]. ¹H NMR (400 MHz, CD₃COCD₃): δ 7.82 (d, *J* = 8.4 Hz, 2 H), 7.58 (d, *J* = 8.4 Hz, 2 H), 7.09 (s, 2 H), 3.86 (s, 6 H), 3.83 ppm (s, 3 H); ¹³C NMR (100 MHz, CD₃COCD₃): δ 194.3, 154.0, 143.2, 138.7, 137.3, 133.0, 132.3, 129.4, 108.3, 60.7, 56.5 ppm; Mp: 98.2-99.7 °C.



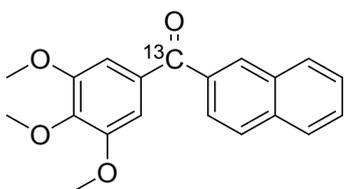
(3,5-Dichlorophenyl)(mesityl)methanone (3mo): Following *general procedure B*, **3mo** was isolated as a white solid with low melting point (63.5 mg, 87%), known compound (CAS: 1096971-39-9). ¹H NMR (400 MHz, CDCl₃): δ 7.64 (d, *J* = 1.6 Hz, 2 H), 7.56 (t, *J* = 2.0 Hz, 1 H), 6.91 (s, 2 H), 2.34 (s, 3 H), 2.07 ppm (s, 6 H); ¹³C NMR (100 MHz, CDCl₃): δ 198.1, 139.8, 139.3, 135.9, 135.3, 134.2, 133.2, 128.6, 127.5, 21.2, 19.4 ppm.



2-(4-(4-Chlorobenzoyl)phenoxy)-2-methylpropanoic acid (3un): Following *general procedure C*, **3un** was isolated as a white solid (57.2 mg, 72%), known compound; The NMR spectroscopic data agree with those described in ref.^[S13]. ¹H NMR (400 MHz, CD₃COCD₃): δ 7.79-7.75 (m, 4 H), 7.58 (dt, *J* = 8.8 Hz, 2.0 Hz, 2 H), 6.99 (dt, *J* = 8.8 Hz, 2.4 Hz, 2 H), 1.67 ppm (s, 6 H); ¹³C NMR (100 MHz, CD₃COCD₃): δ 194.0, 174.9, 160.7, 138.4, 137.7, 132.6, 132.1, 131.0, 129.3, 118.2, 79.9, 25.6 ppm; Mp: 177.1-178.6 °C.



Naphthalen-2-yl(3,4,5-trimethoxyphenyl)methanone (3lp): Following *general procedure B*, **3lp** was isolated as a white solid (51.5 mg, 64%), known compound; The NMR spectroscopic data agree with those described in ref.^[S9]. ¹H NMR (400 MHz, CD₃COCD₃): δ 8.38 (s, 1 H), 8.11 (d, *J* = 8.0 Hz, 1 H), 8.05 (d, *J* = 8.4 Hz, 1 H), 8.02 (d, *J* = 8.0 Hz, 1 H), 7.92 (dd, *J* = 8.4 Hz, 2.0 Hz, 1 H), 7.69-7.60 (m, 2 H), 7.18 (s, 2 H), 3.86 (s, 6 H), 3.85 ppm (s, 3 H); ¹³C NMR (100 MHz, CD₃COCD₃): δ 195.5, 154.0, 143.0, 136.0, 135.9, 133.7, 133.3, 132.1, 130.3, 129.14, 129.05, 128.6, 127.7, 126.5, 108.5, 60.7, 56.5 ppm; Mp: 106.4-107.2 °C.

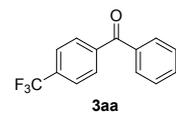
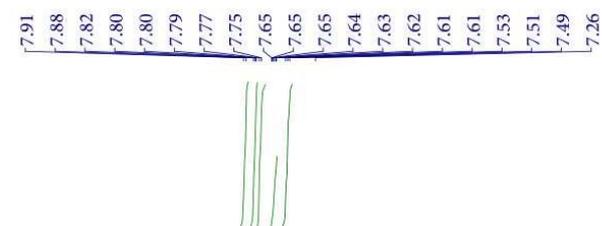


Naphthalen-2-yl(3,4,5-trimethoxyphenyl)methanone (¹³C-3lp): Following *general procedure B*, ¹³C-**3lp** was isolated as a white solid (51.7 mg, 64%). ¹H NMR (400 MHz, CD₃COCD₃): δ 8.38 (d, *J* = 3.6 Hz, 1 H), 8.11 (d, *J* = 8.0 Hz, 1 H), 8.05 (d, *J* = 8.4 Hz, 1 H), 8.02 (d, *J* = 8.0 Hz, 1 H), 7.92 (ddd, *J* = 8.4 Hz, 3.2 Hz, 1.6 Hz, 1 H), 7.69-7.60 (m, 2 H), 7.18 (d, *J* = 4.0 Hz, 2 H), 3.864 (s, 6 H), 3.855 ppm (s, 3 H); ¹³C NMR (100 MHz, CD₃COCD₃): δ 195.5, 154.0 (d, *J* = 6 Hz), 143.1, 136.0, 135.9 (d, *J* = 55 Hz), 133.5 (d, *J* = 56 Hz), 133.3 (d, *J* = 5 Hz), 132.1 (d, *J* = 3 Hz), 130.3, 129.2, 129.1 (d, *J* = 4 Hz), 128.6, 127.7, 126.5 (d, *J* = 3 Hz), 108.5 (d, *J* = 3 Hz), 60.7, 56.6 ppm; HRMS (ESI) calcd. for C₁₉¹³CH₁₈O₄⁺ [M + H⁺] *m/z* 324.1311, found 324.1316; Mp: 106.6-107.5 °C.

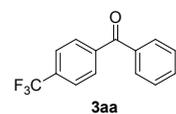
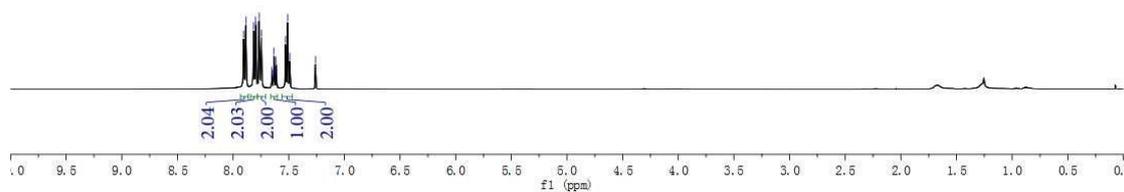
6. References

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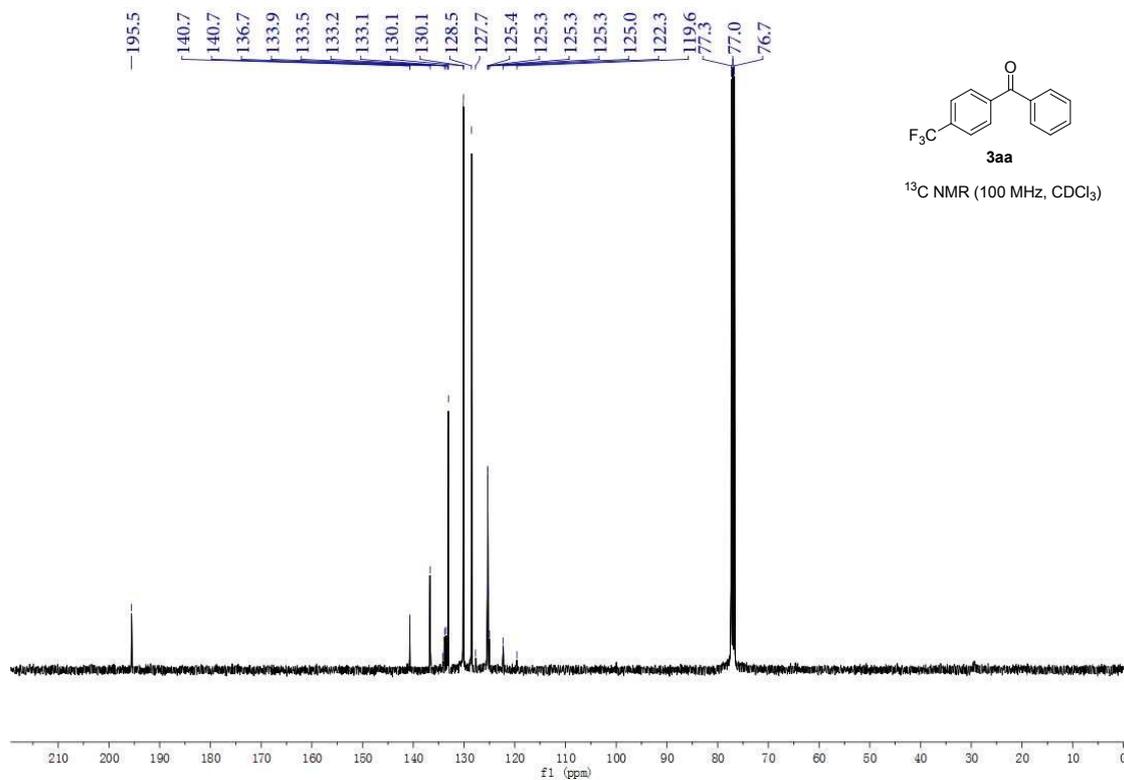
7. Copies of NMR Spectra

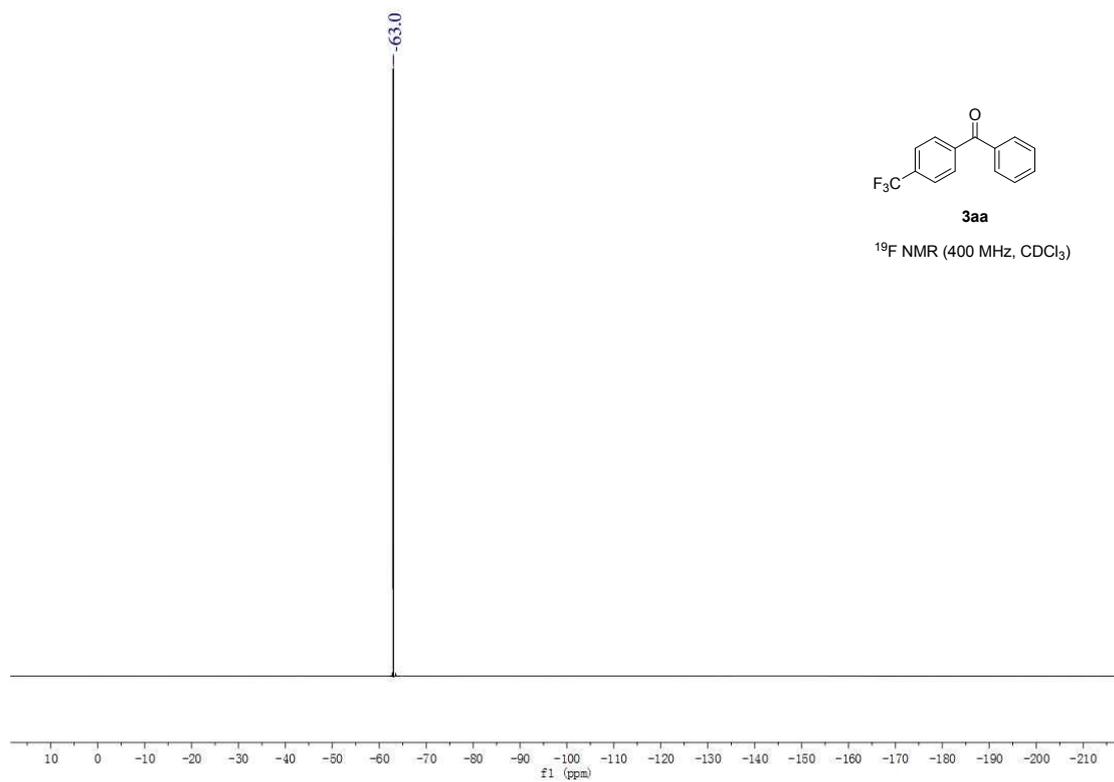


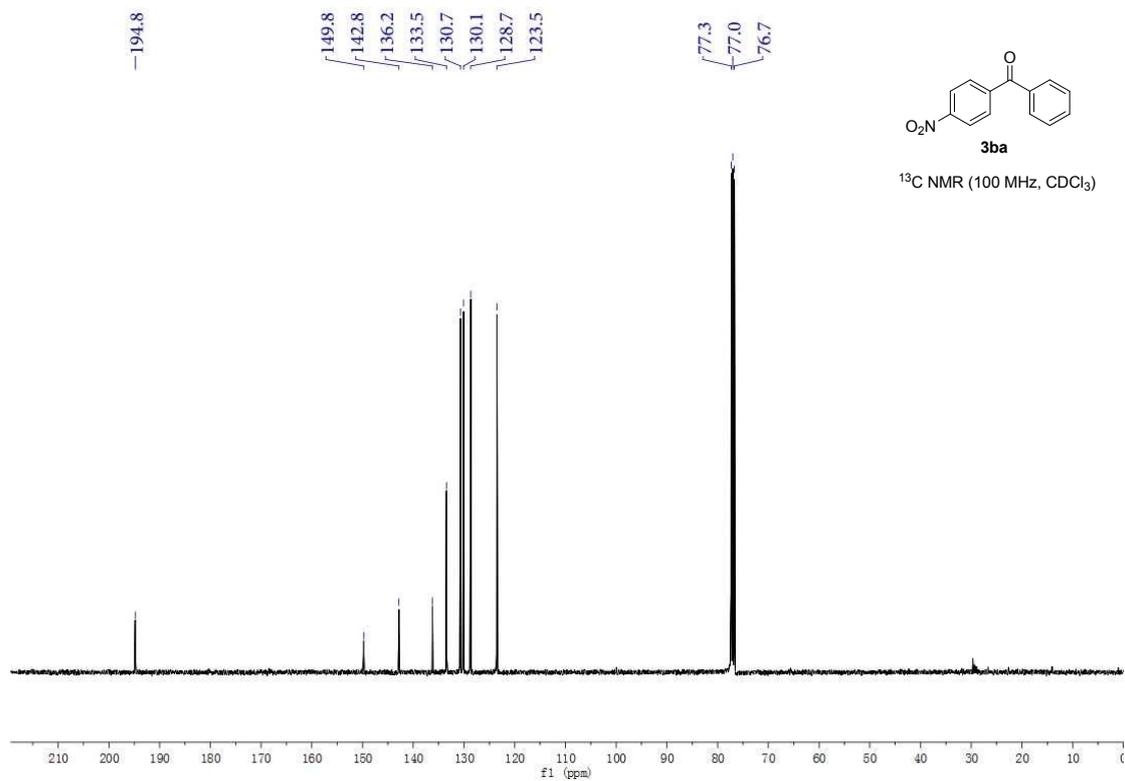
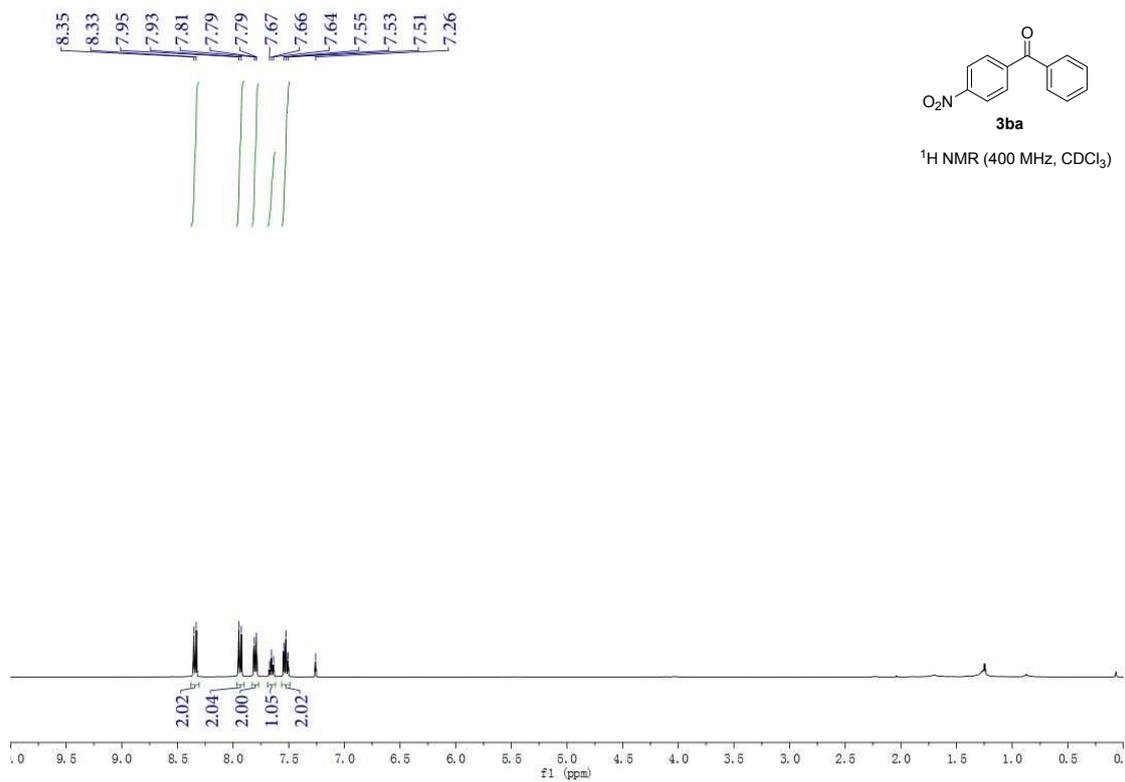
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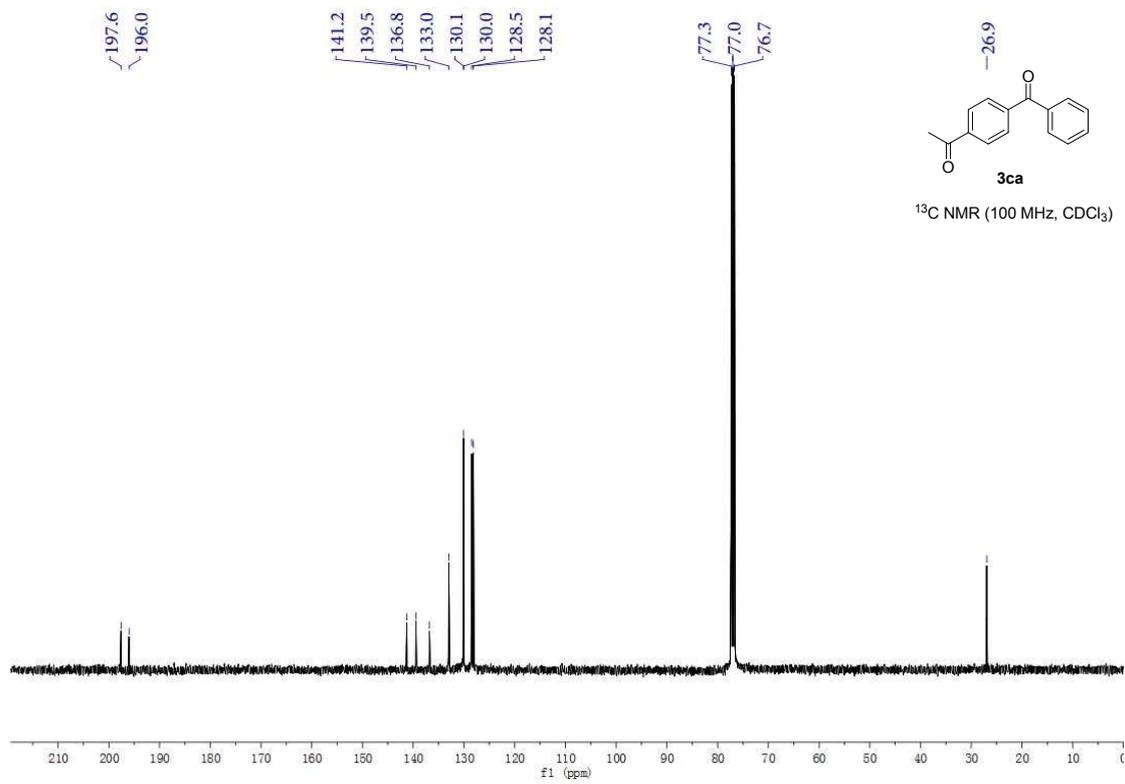
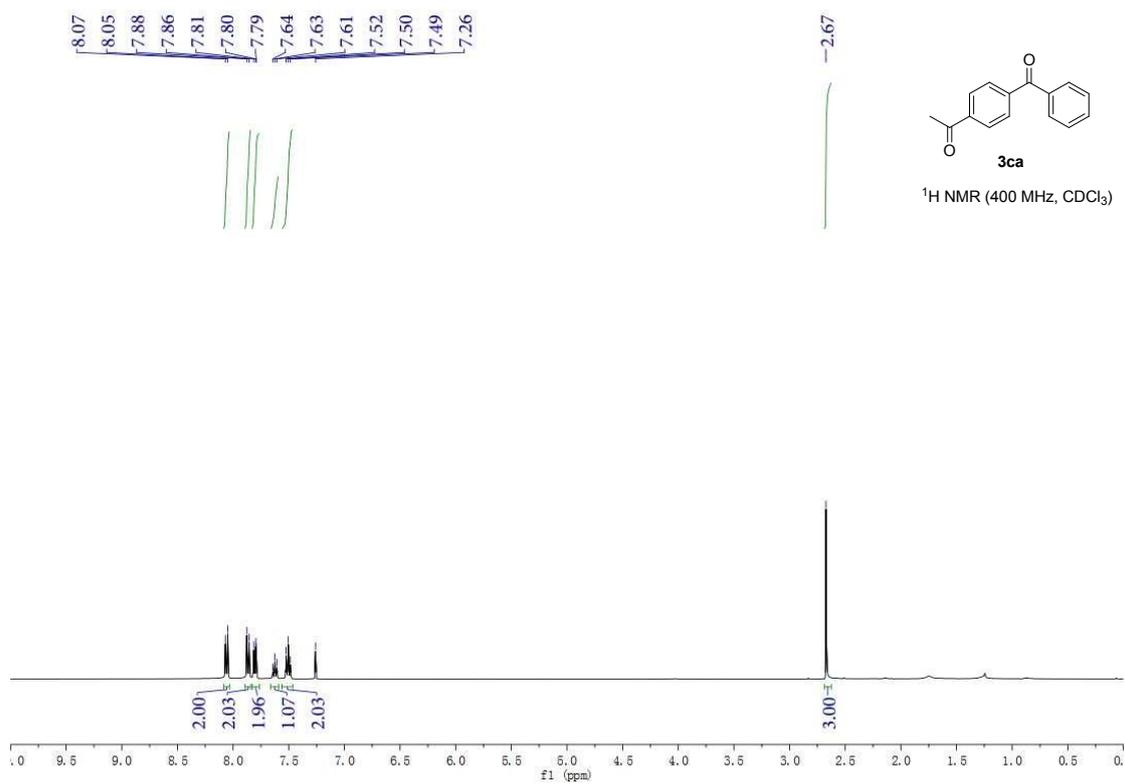


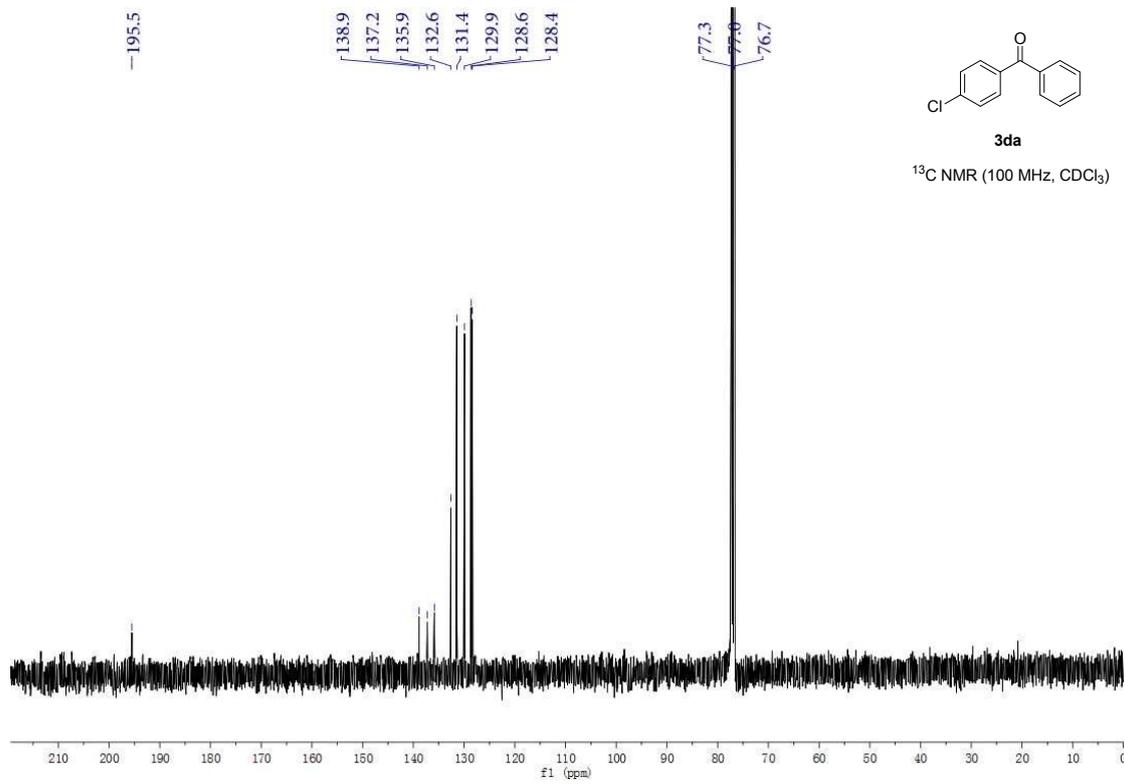
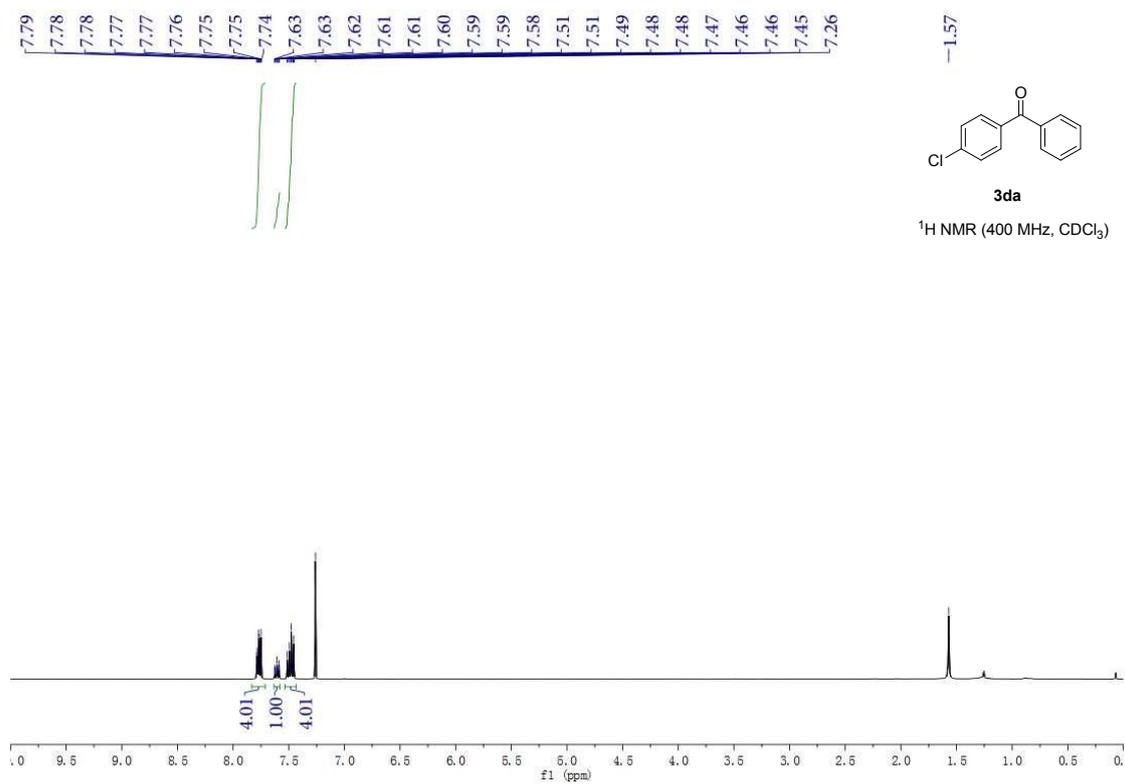
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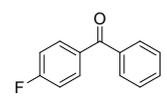
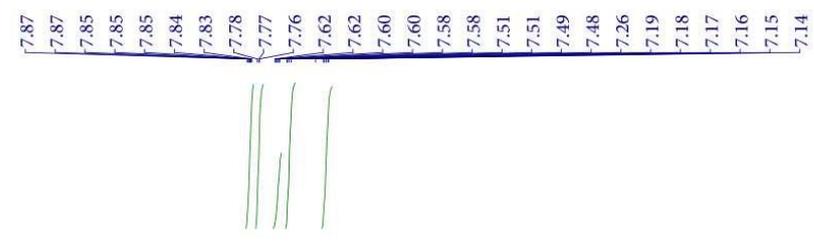






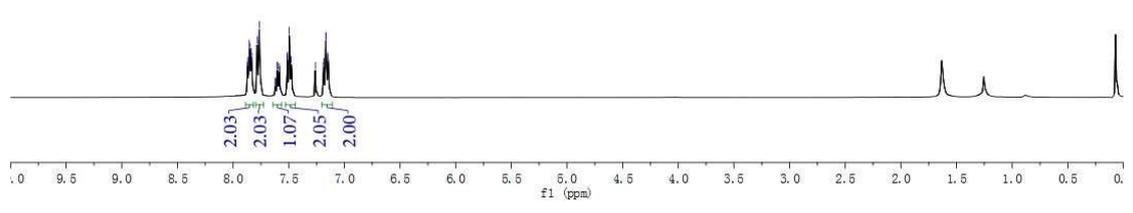




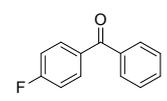


3ea

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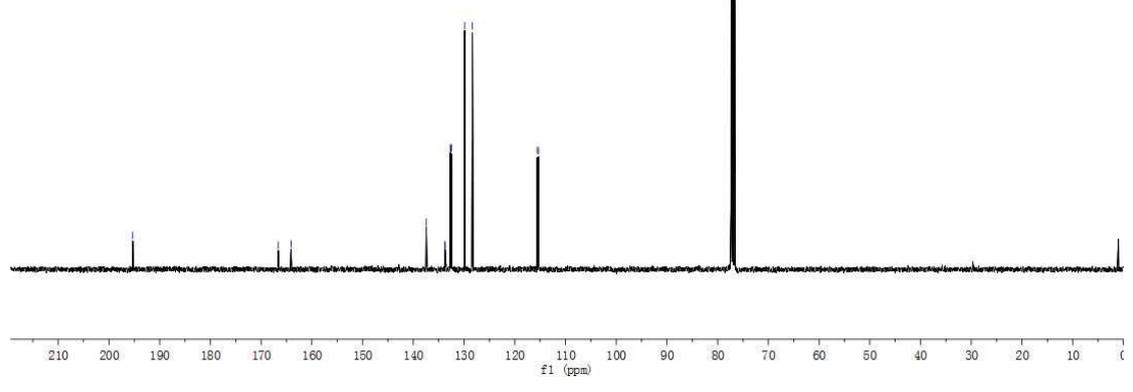


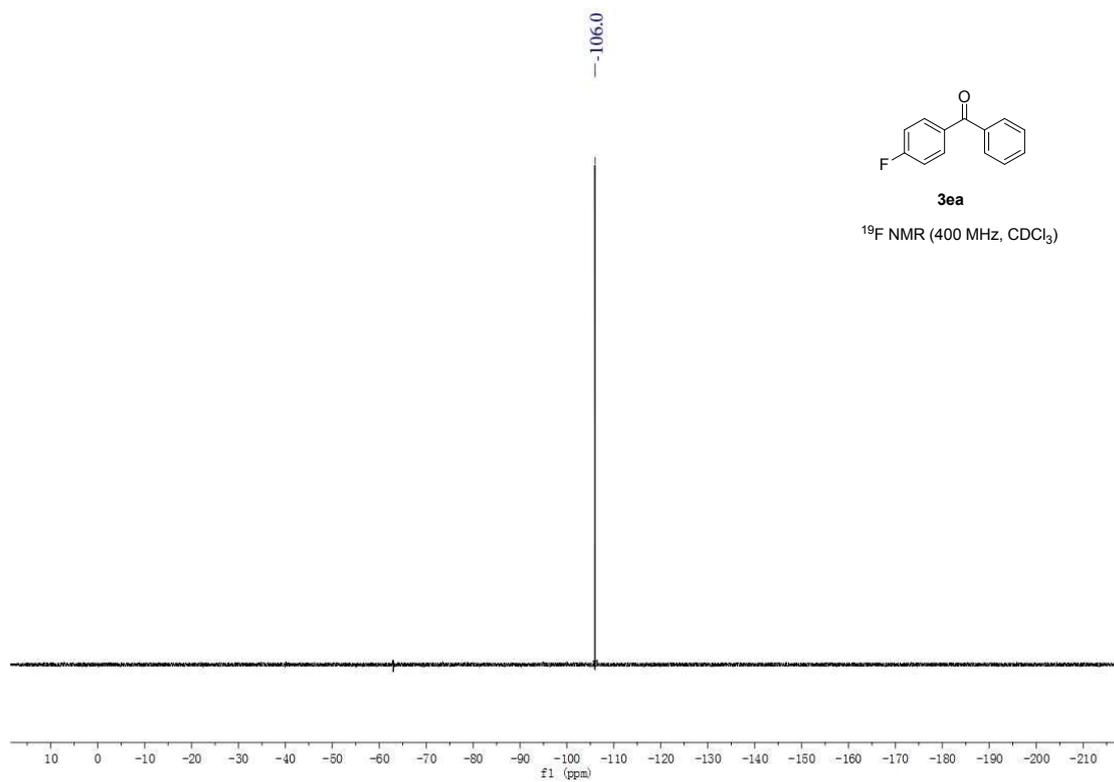
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166.6
164.1
137.5
132.7
132.6
132.5
129.9
128.3
115.6
115.3
77.3
77.0
76.7

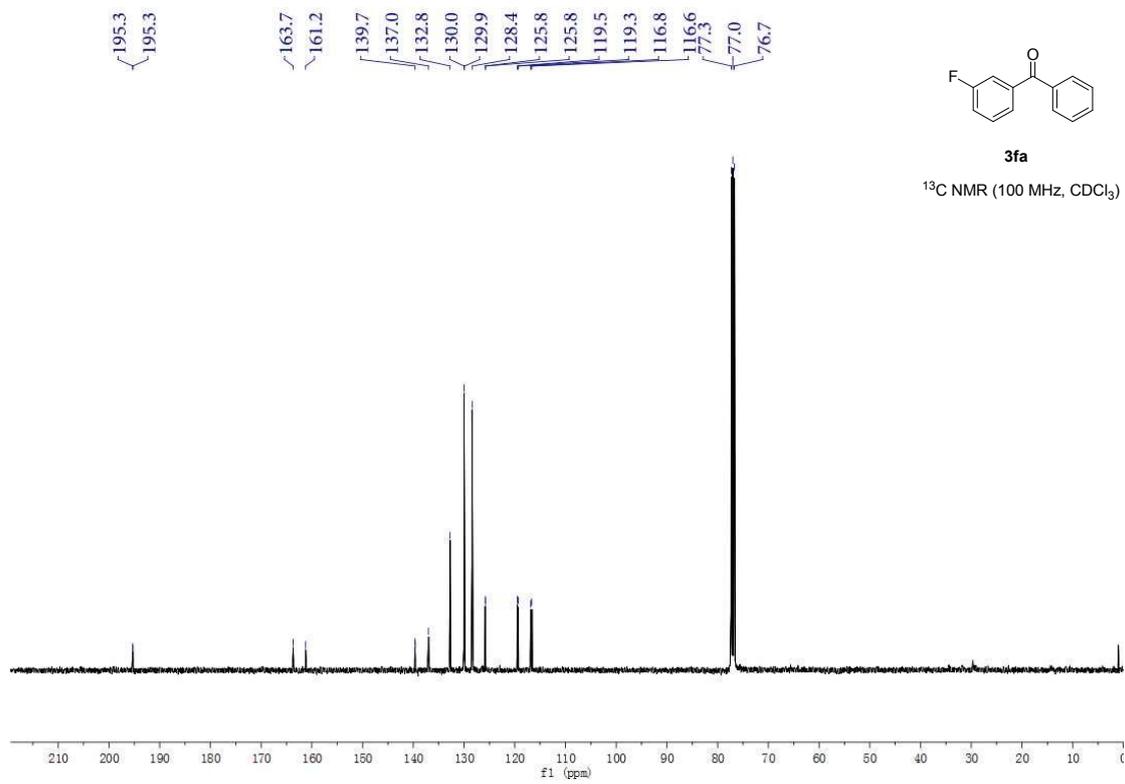
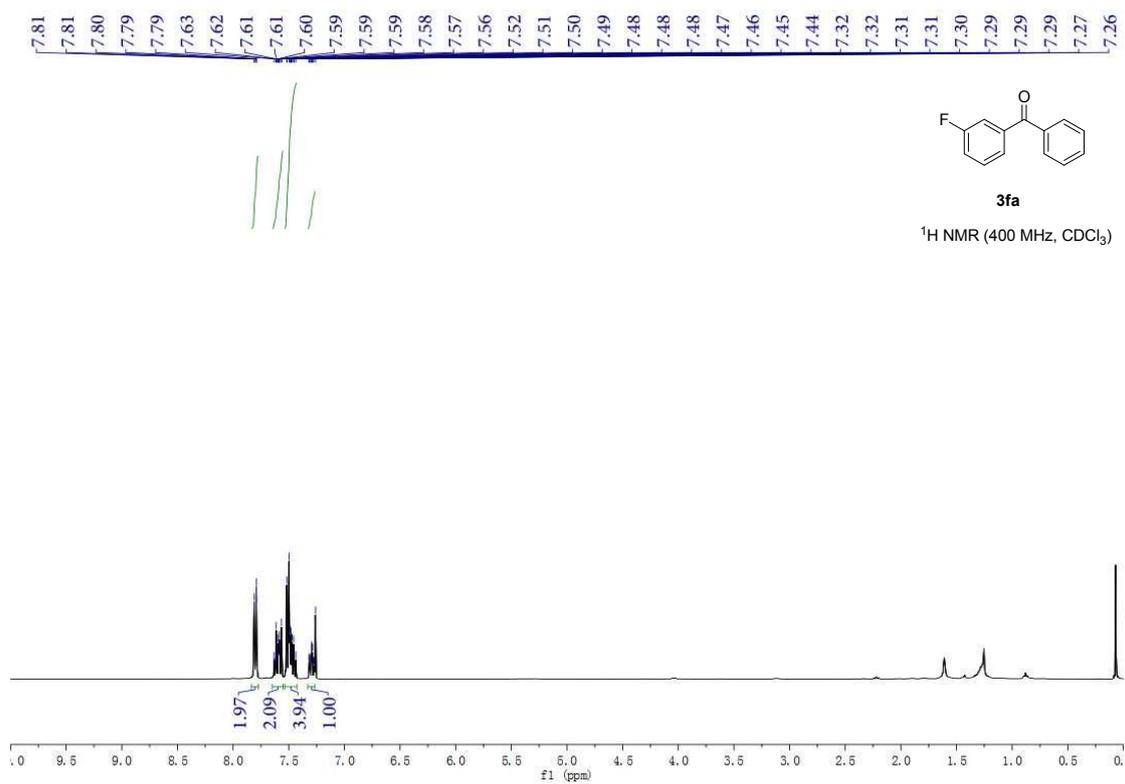


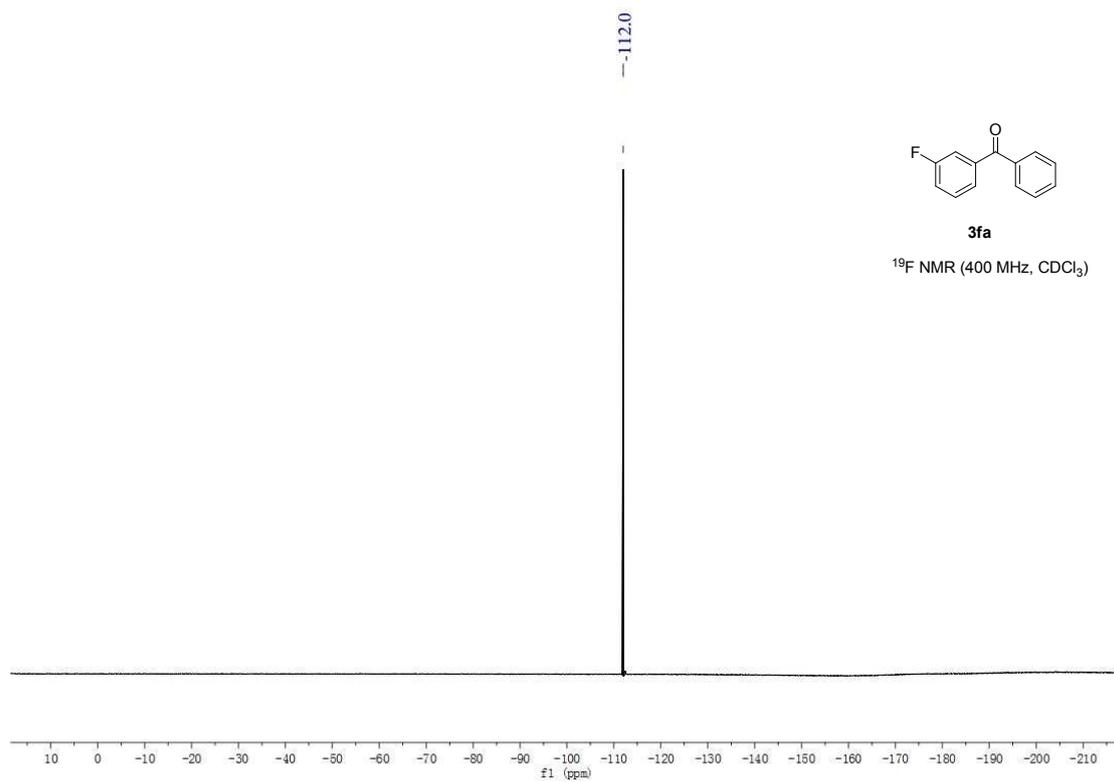
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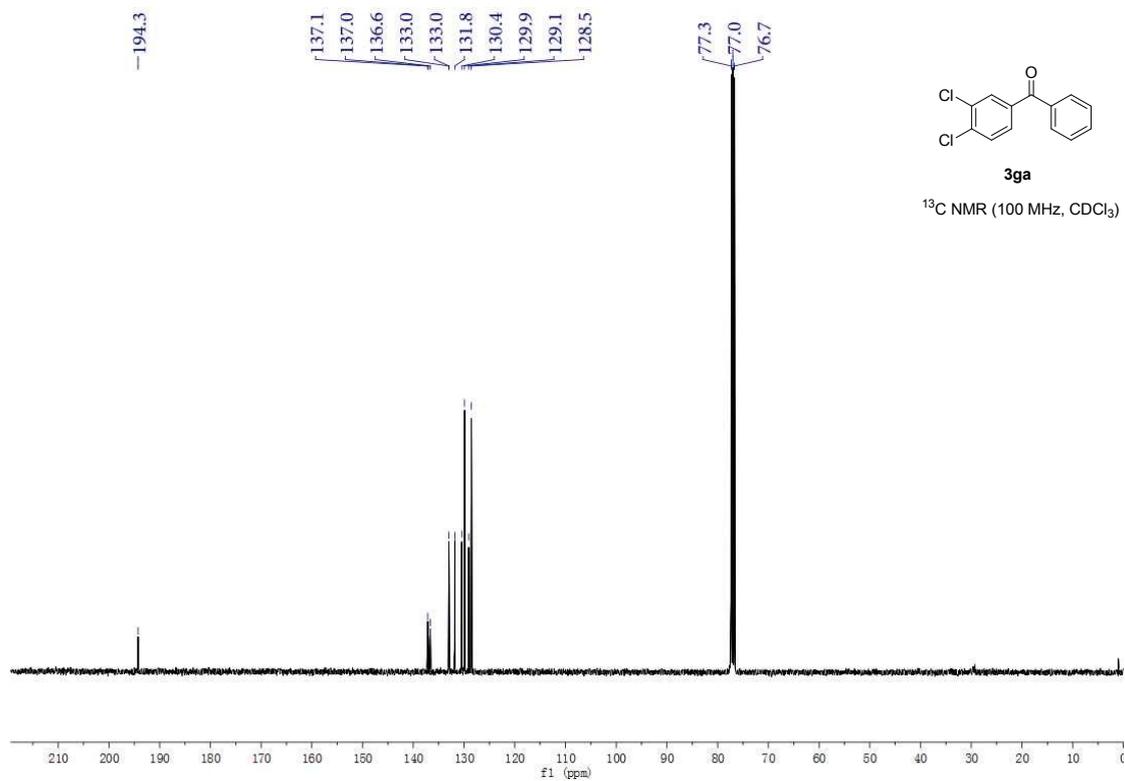
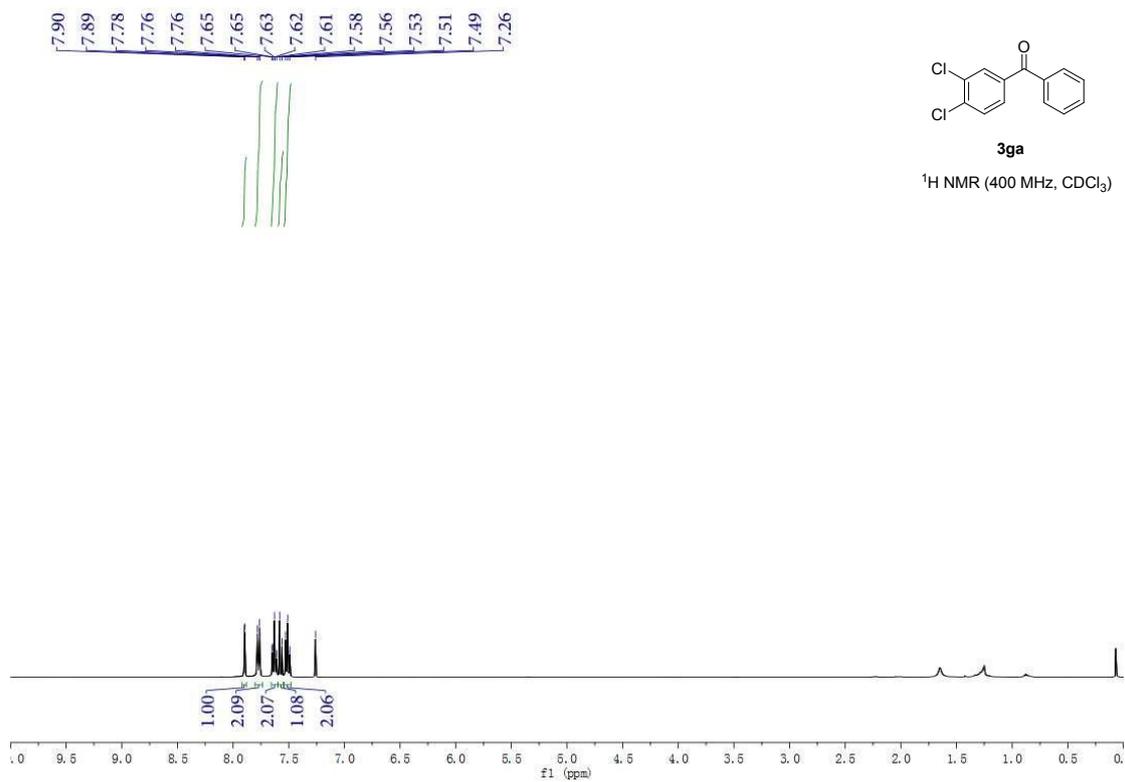
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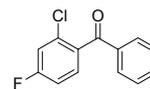
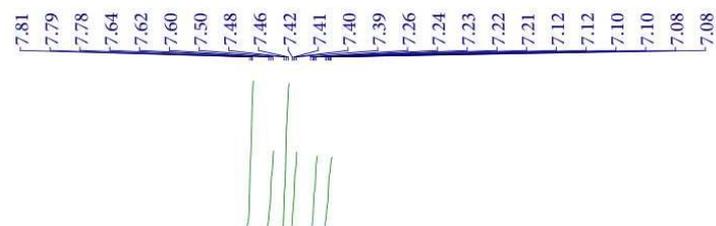






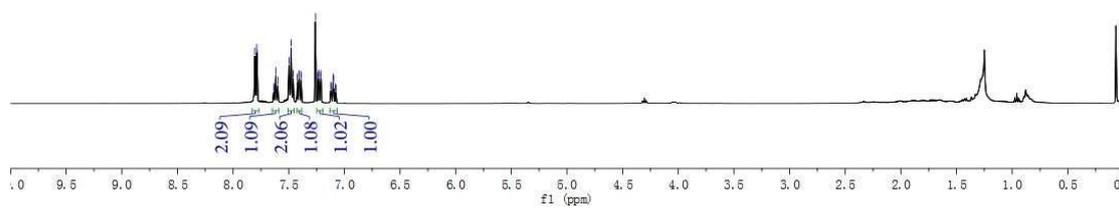




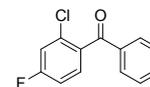


3ha

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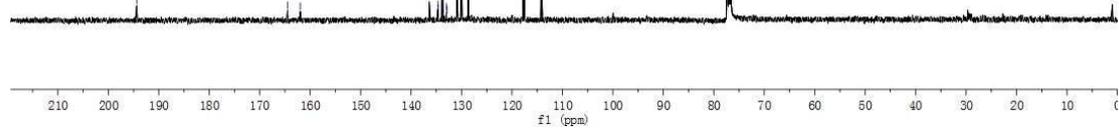


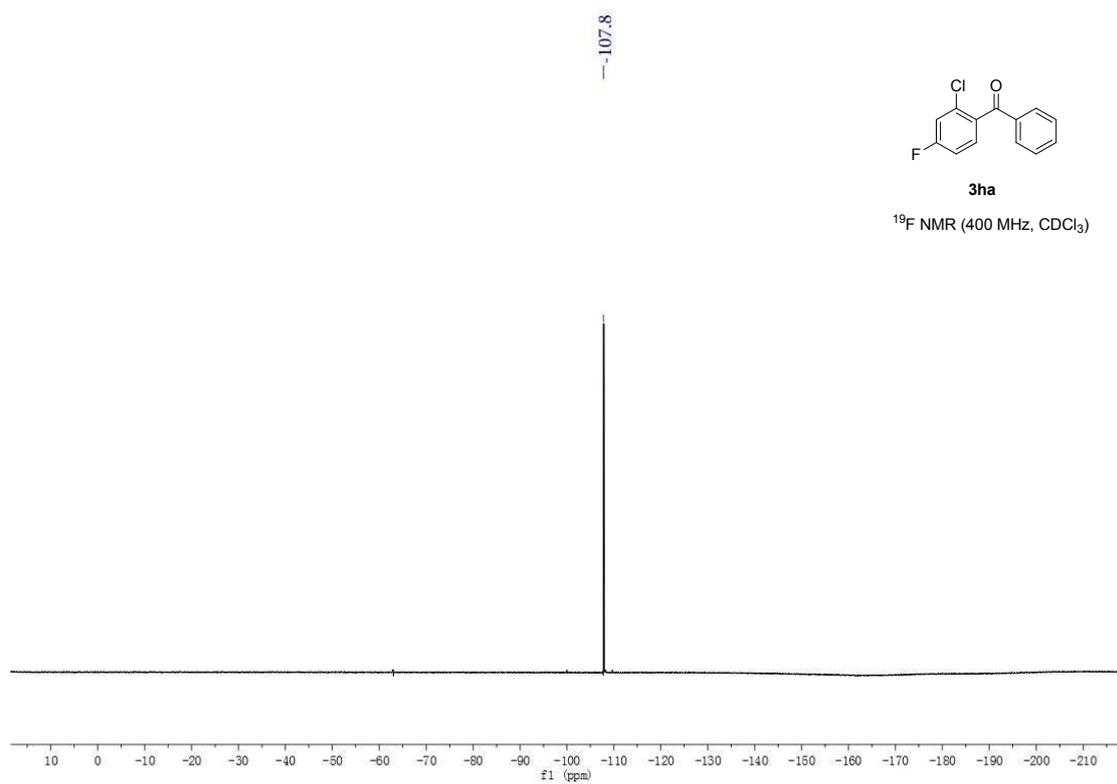
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164.5
162.0
136.4
134.7
133.8
133.0
131.0
130.9
130.0
128.6
117.8
117.5
114.3
114.1
77.3
77.0
76.7

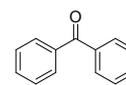
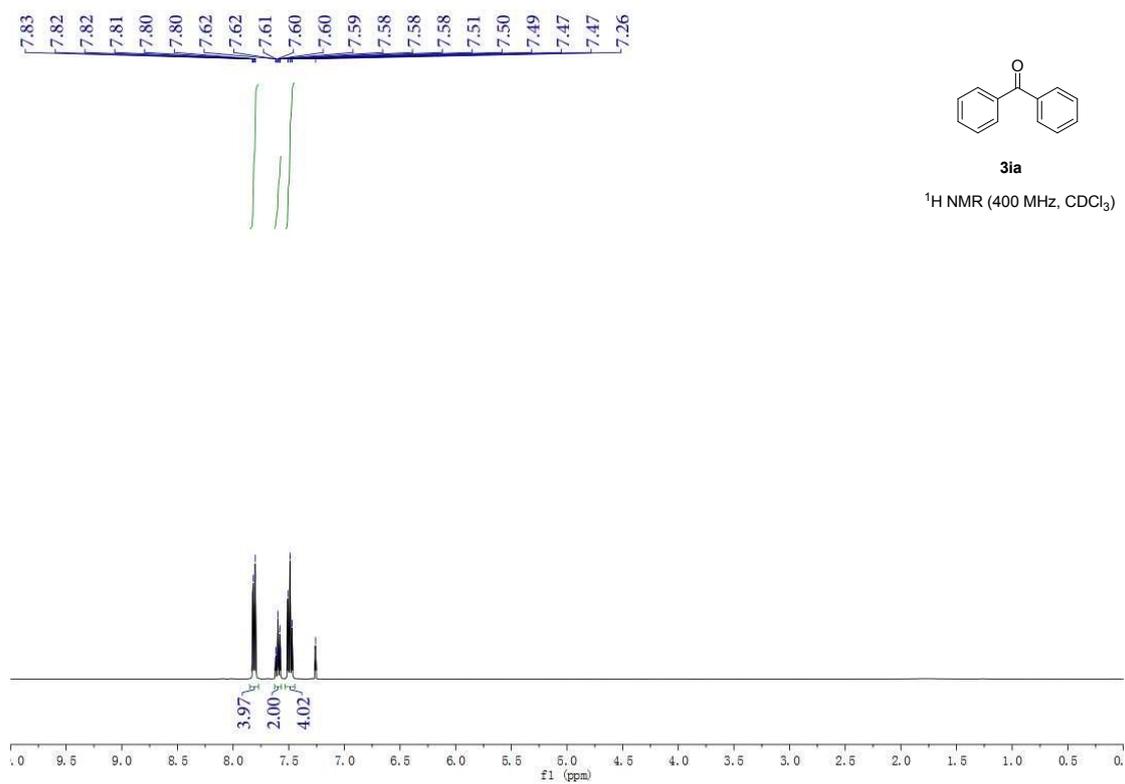


3ha

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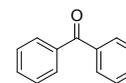
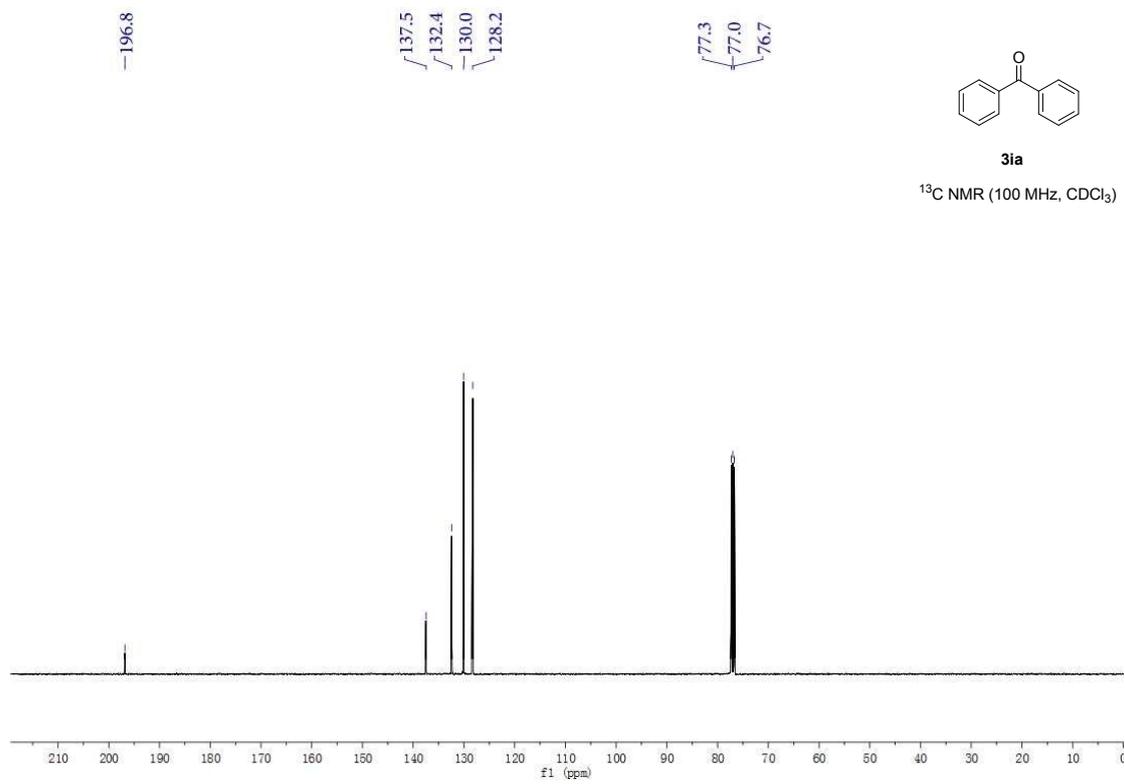






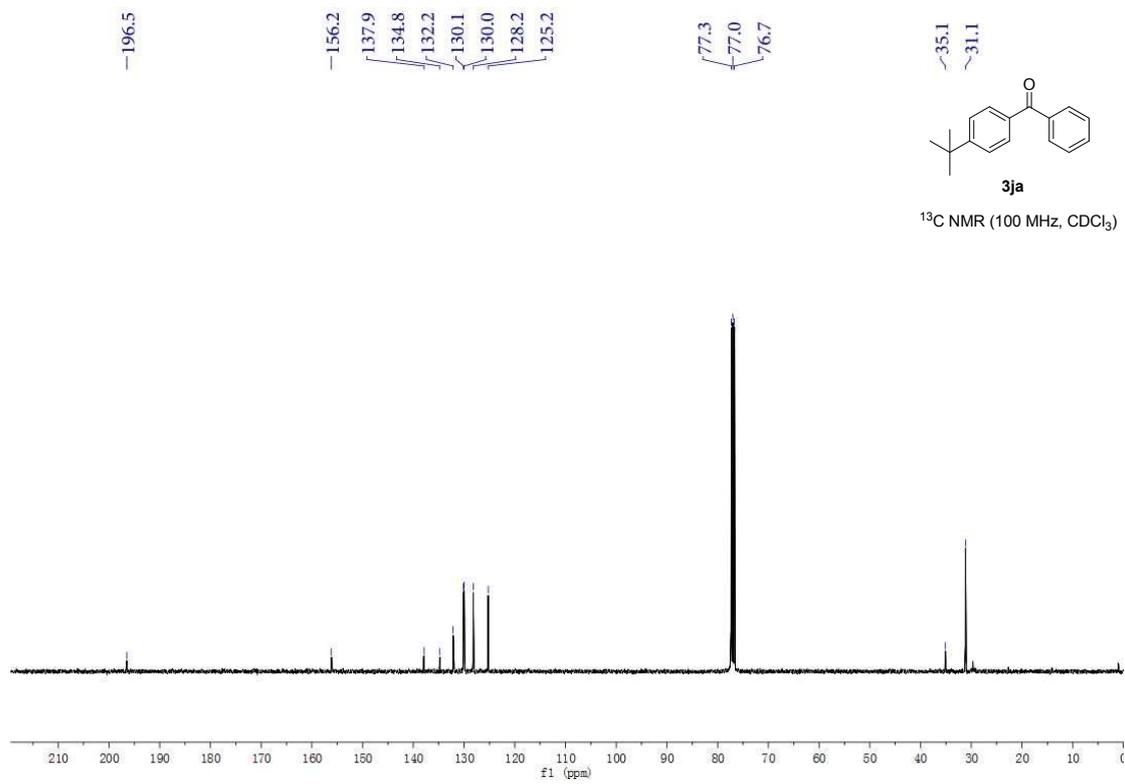
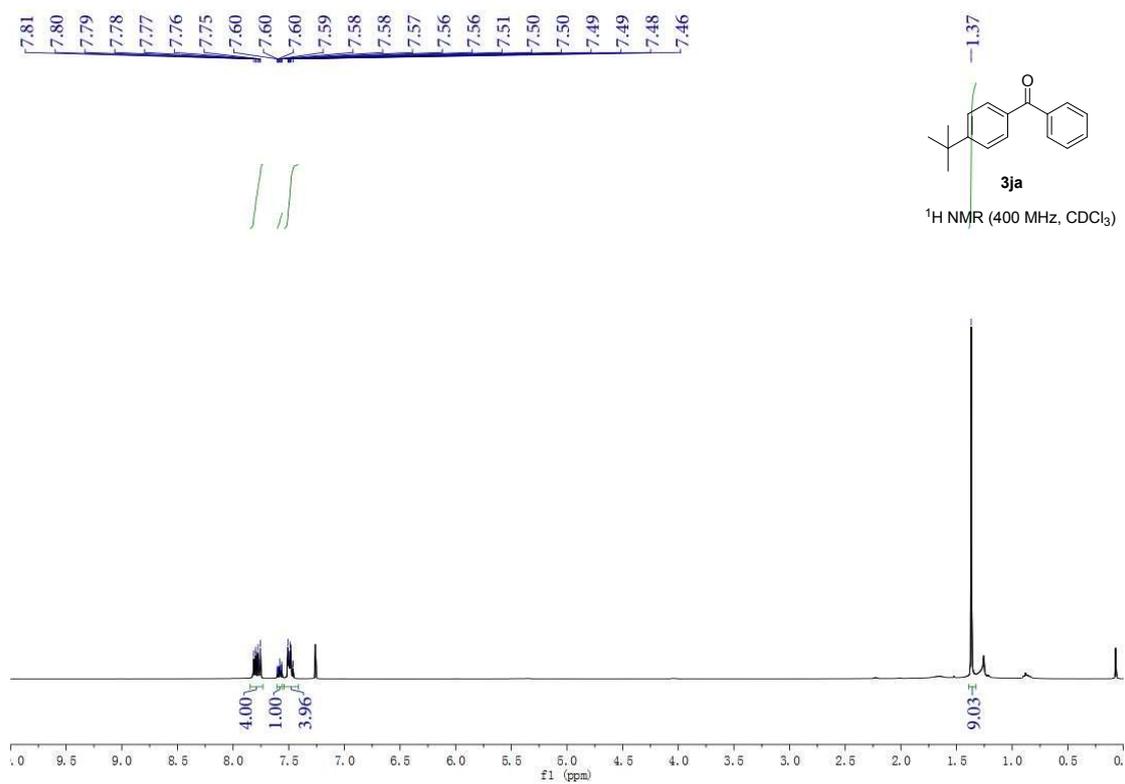
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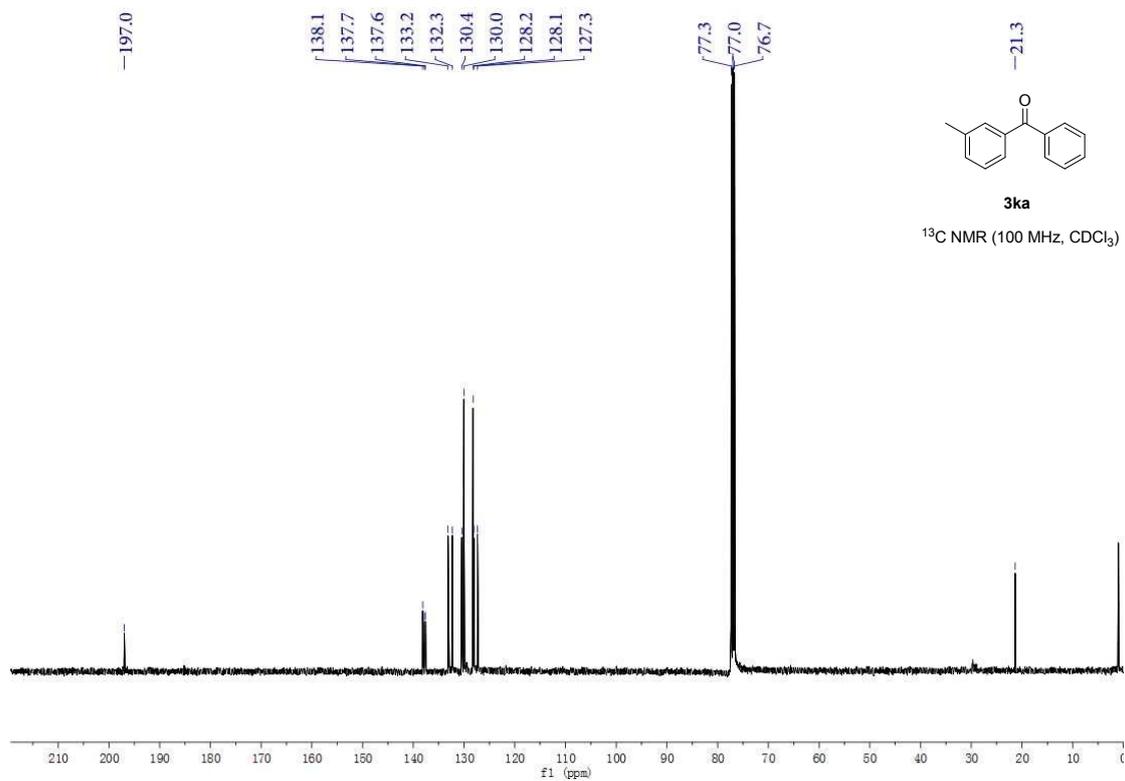
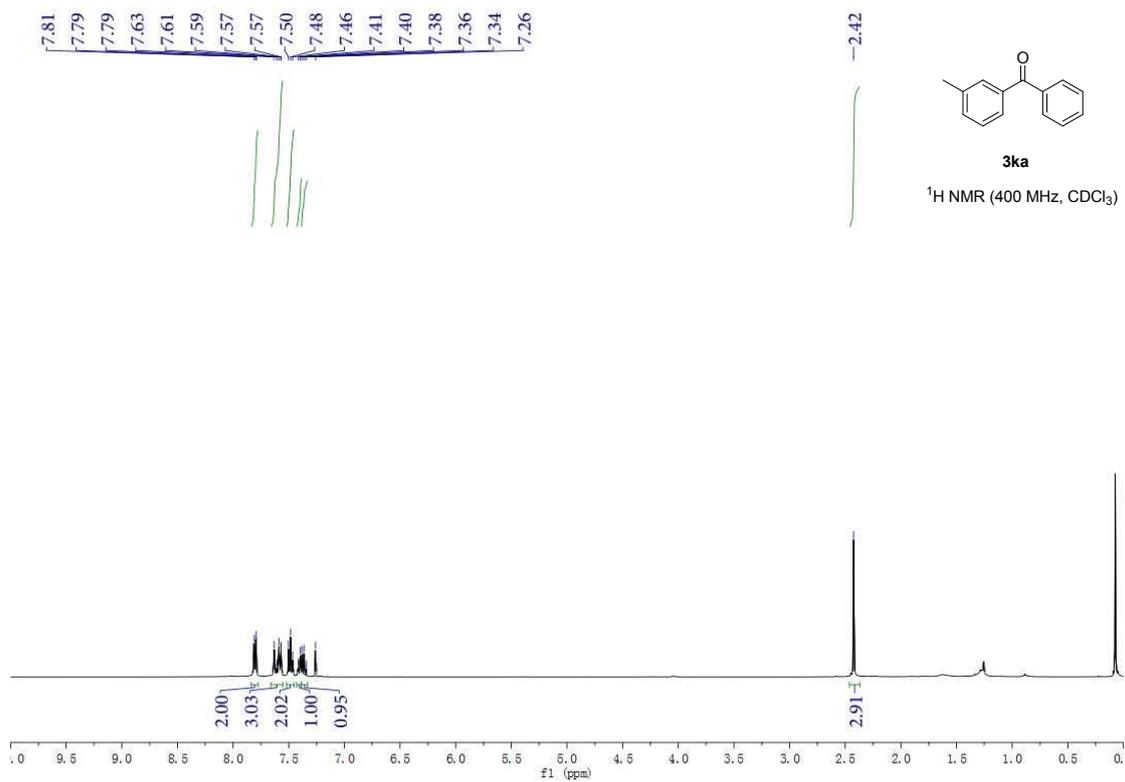
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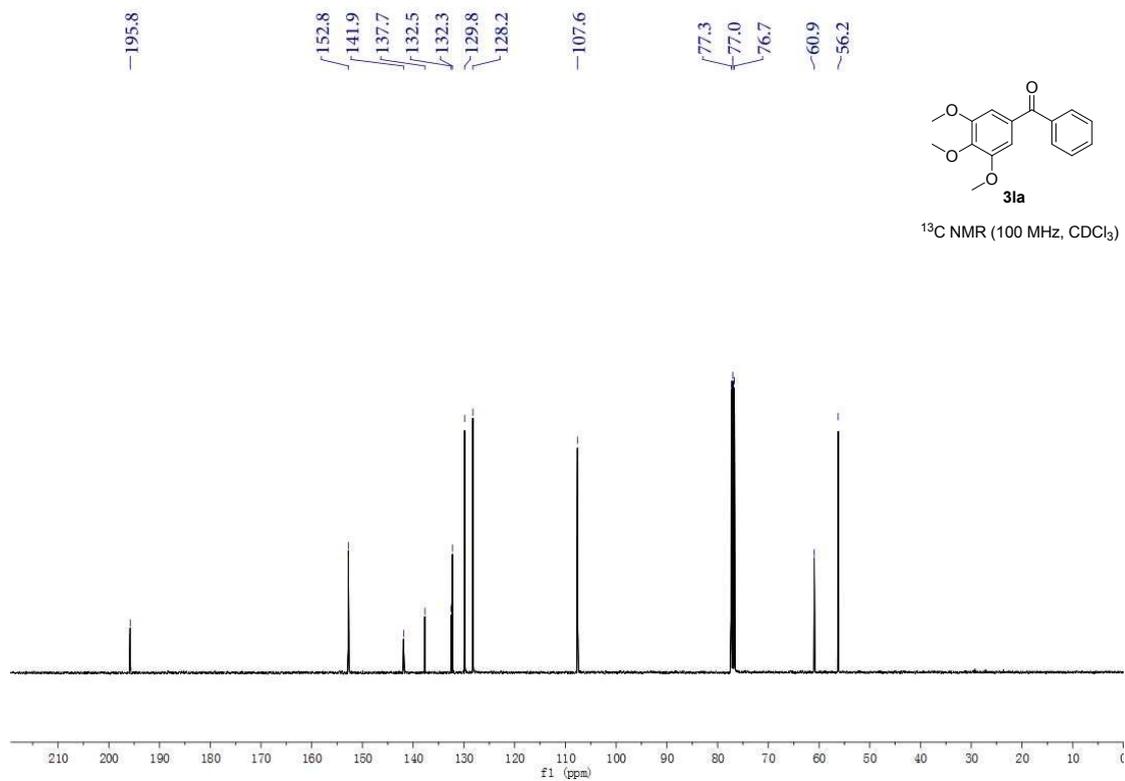
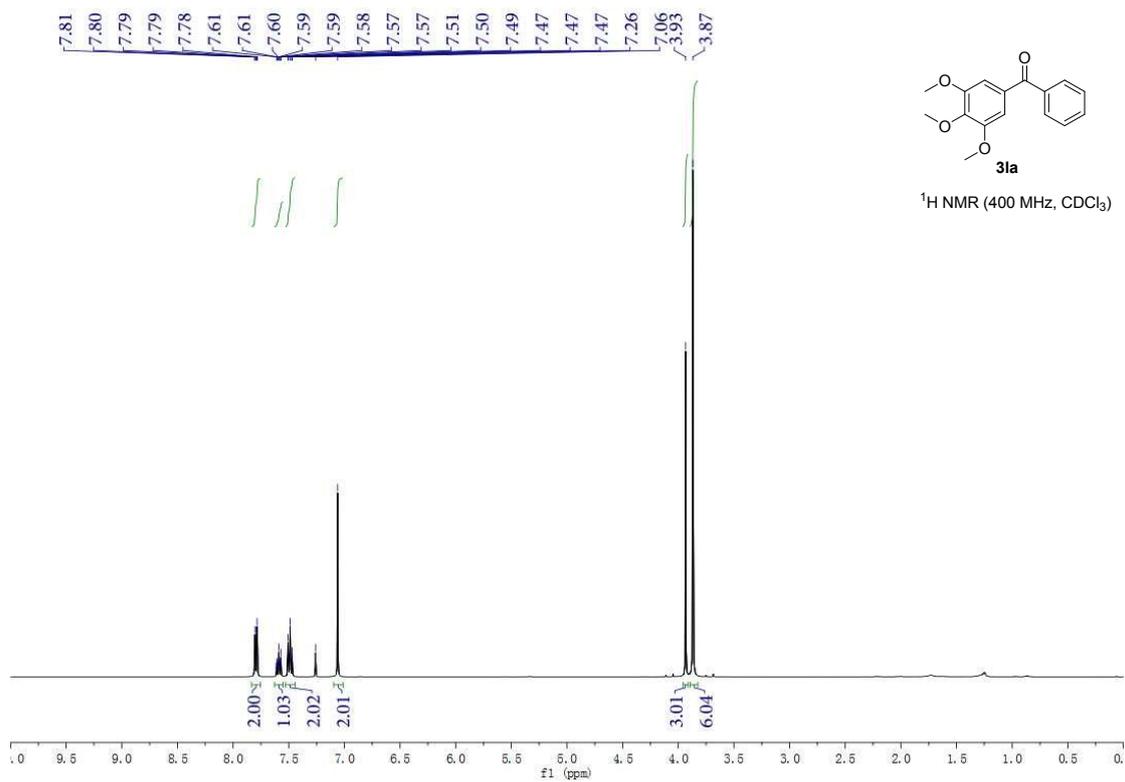


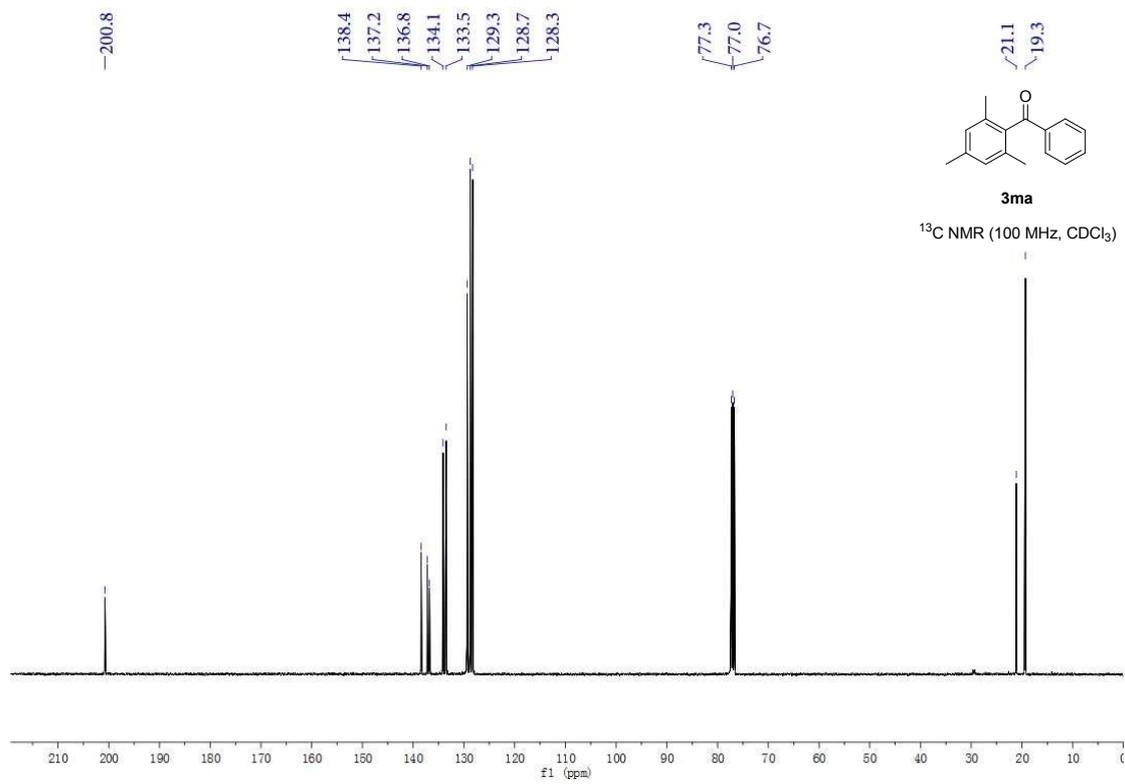
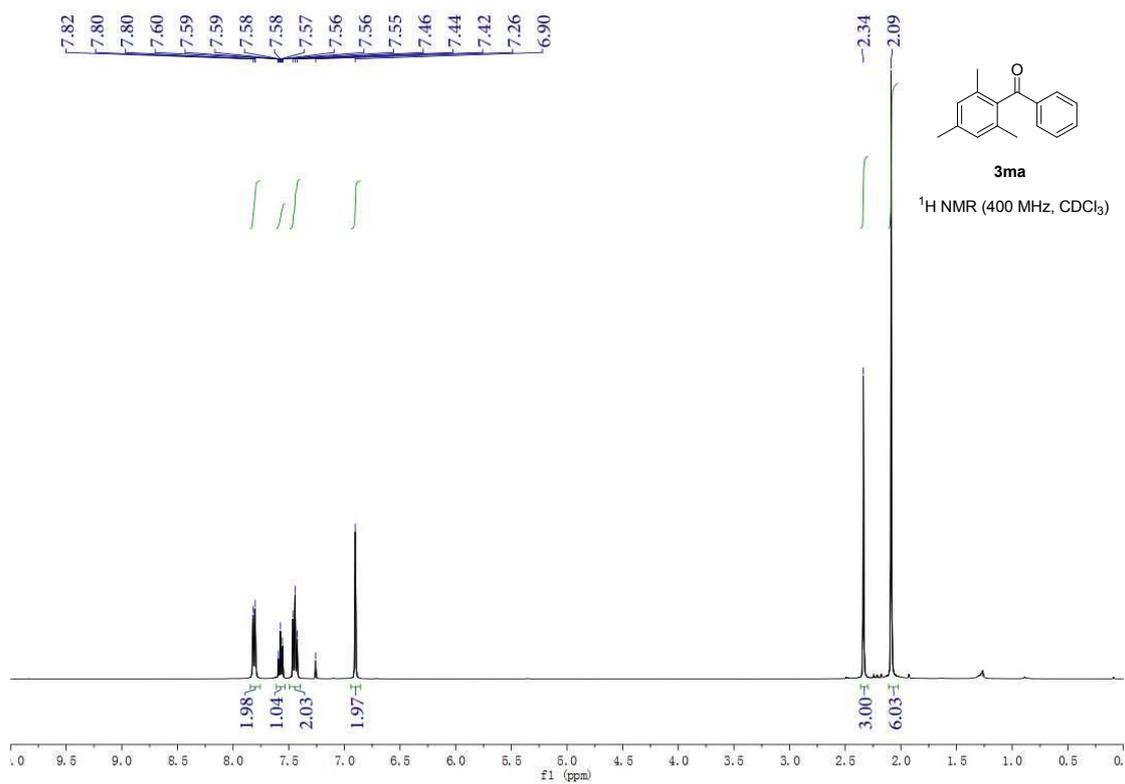
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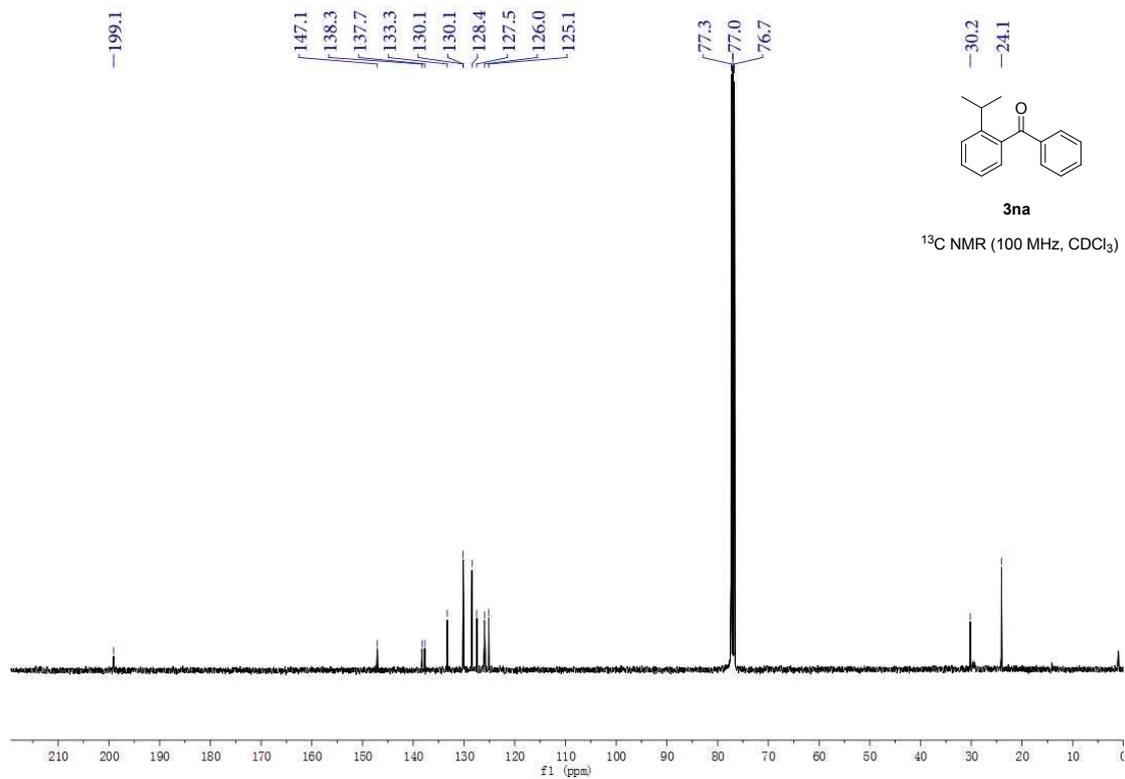
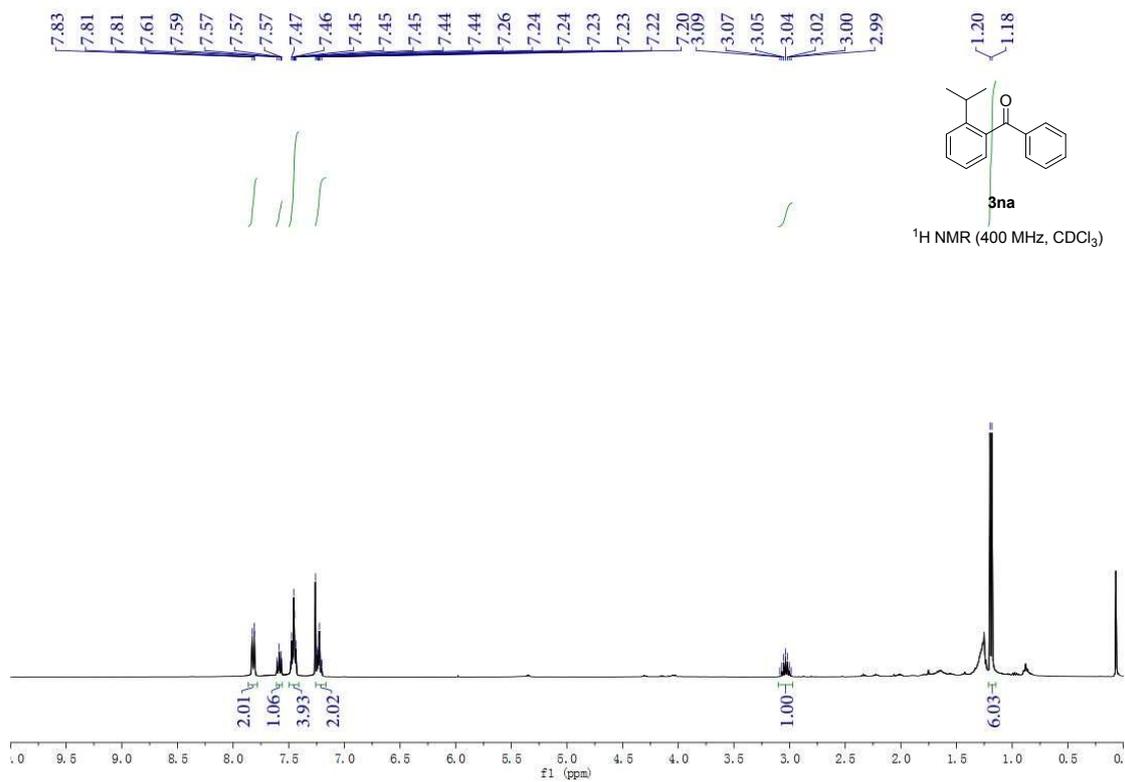
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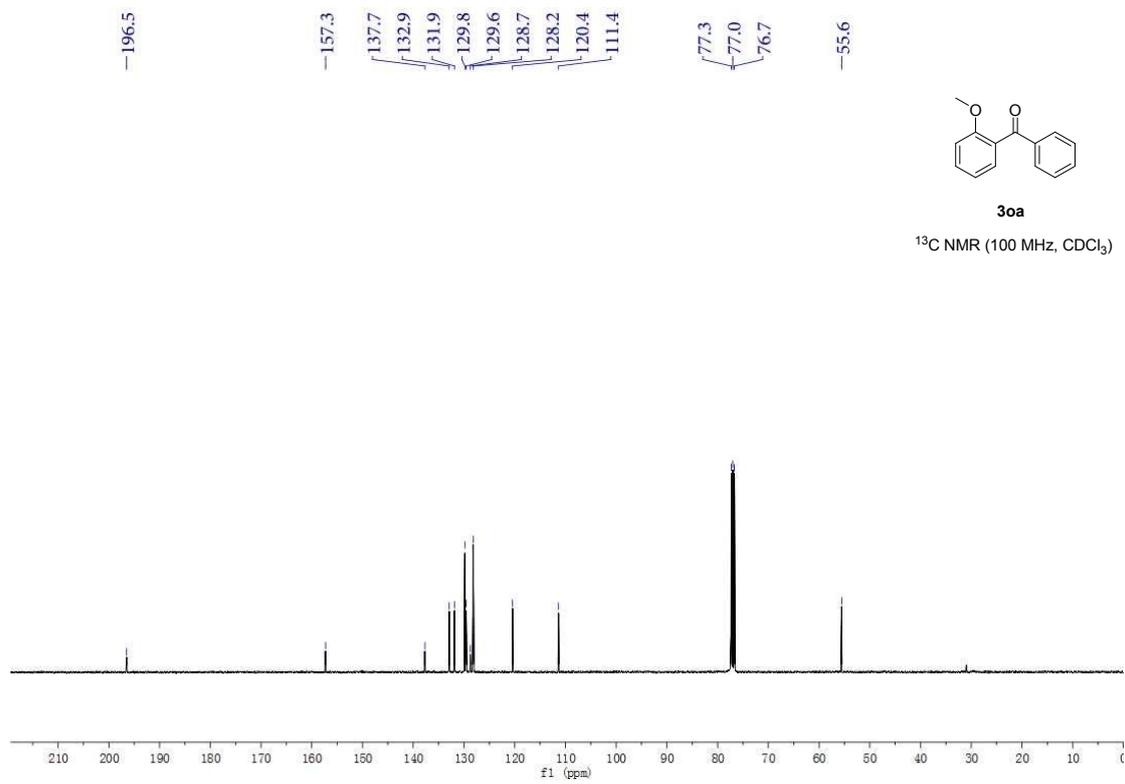
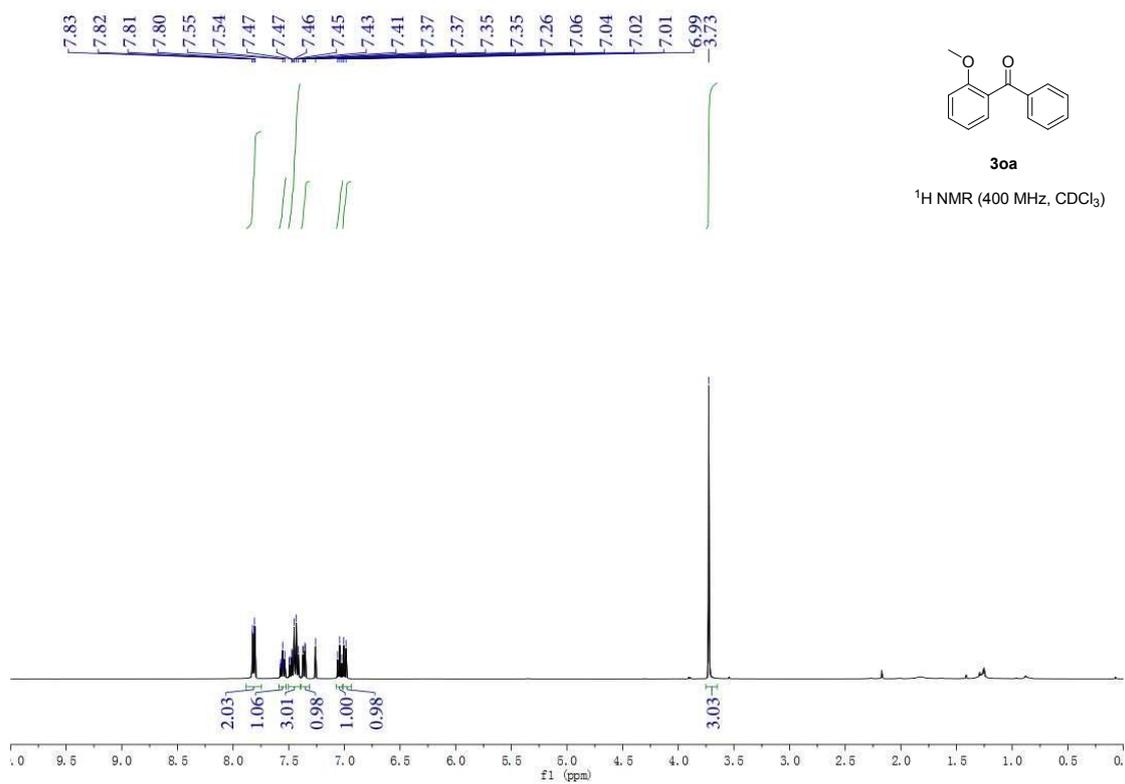


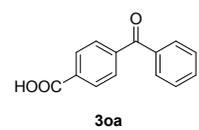
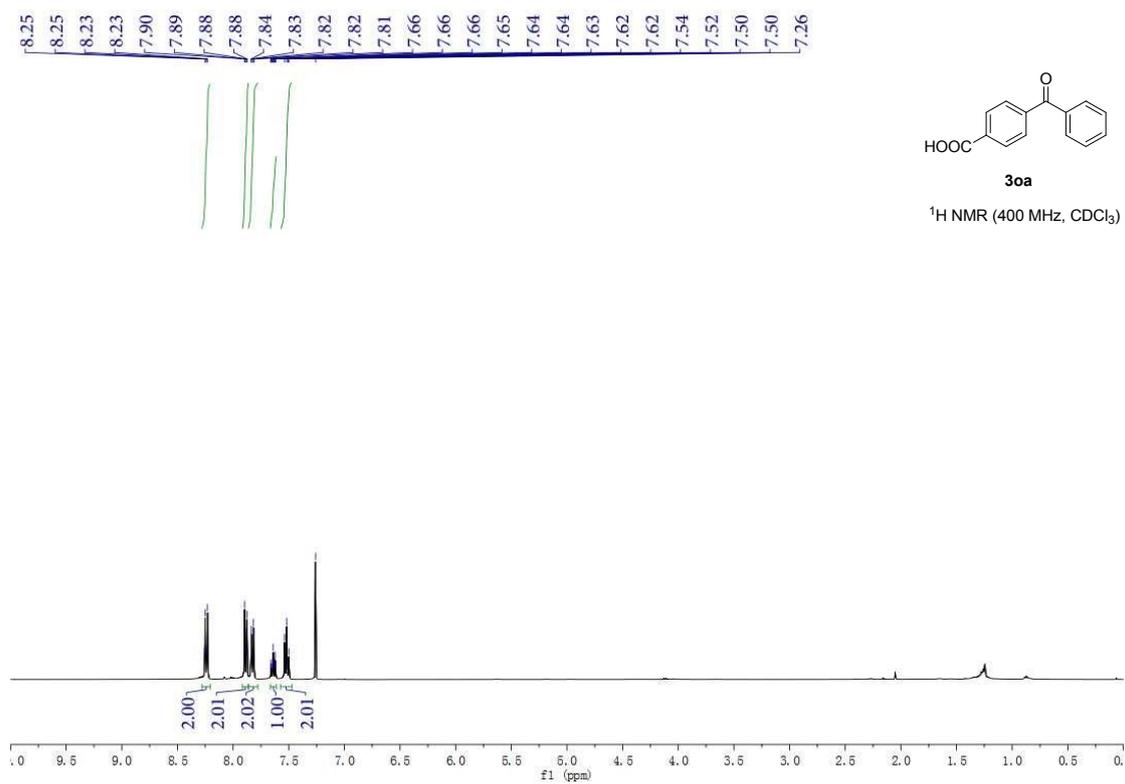




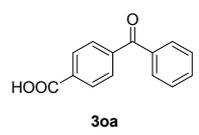
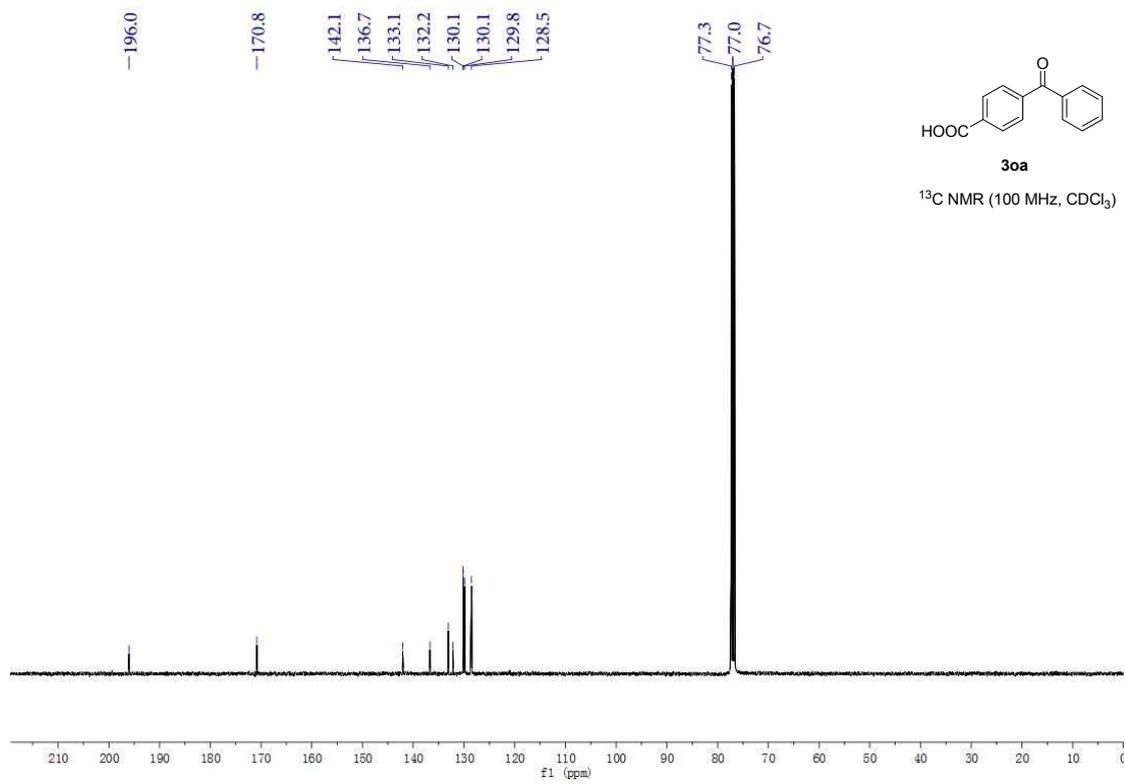




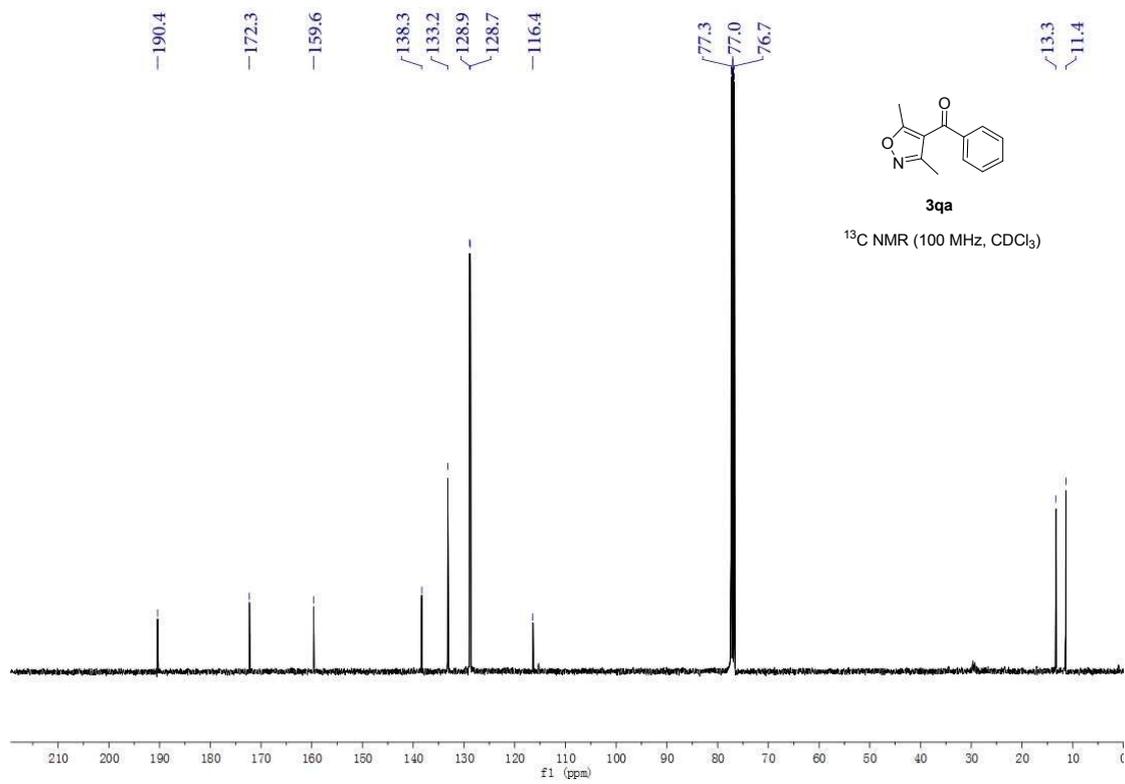
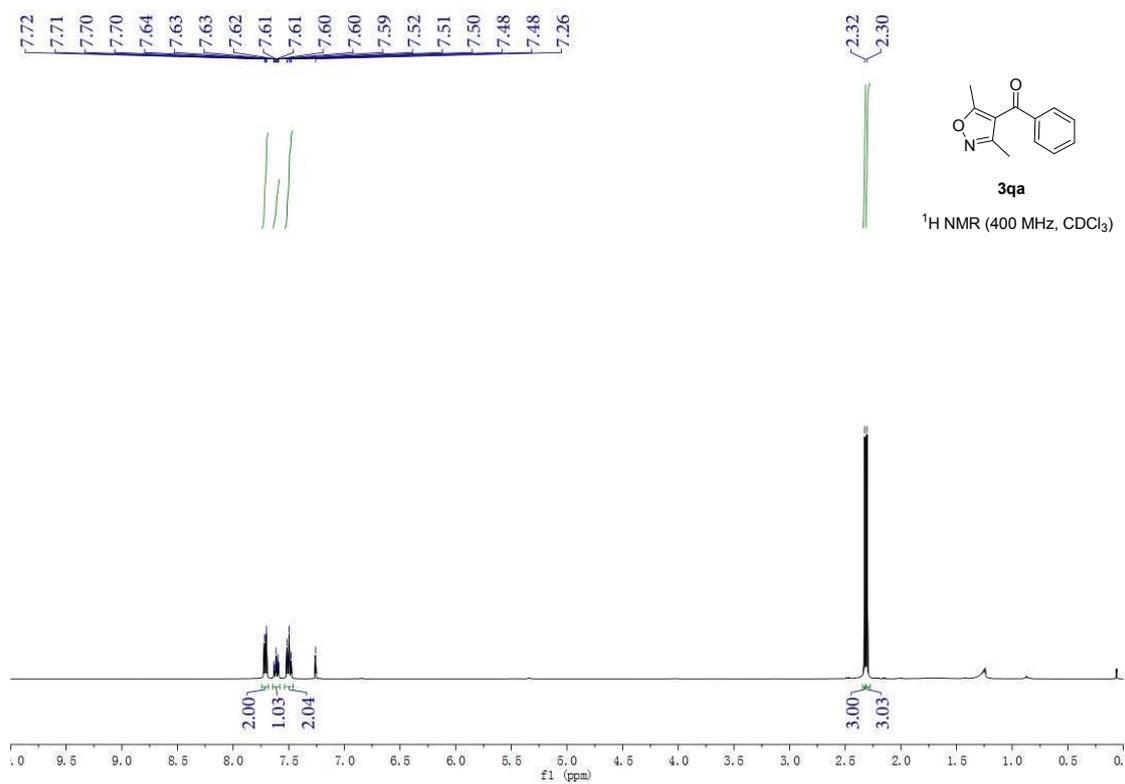


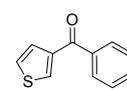
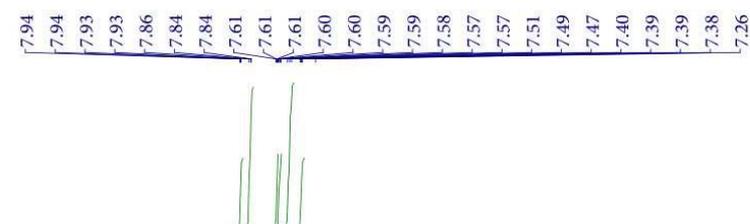


¹H NMR (400 MHz, CDCl₃)



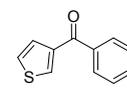
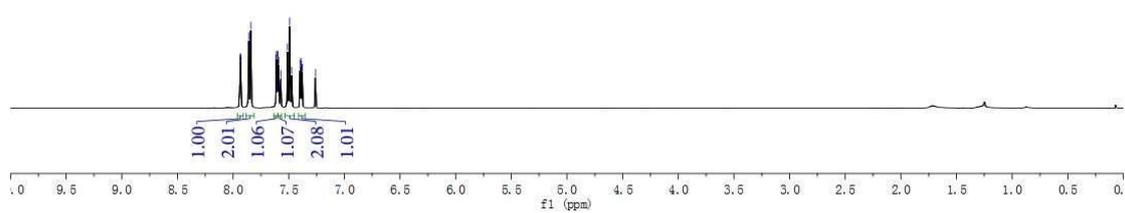
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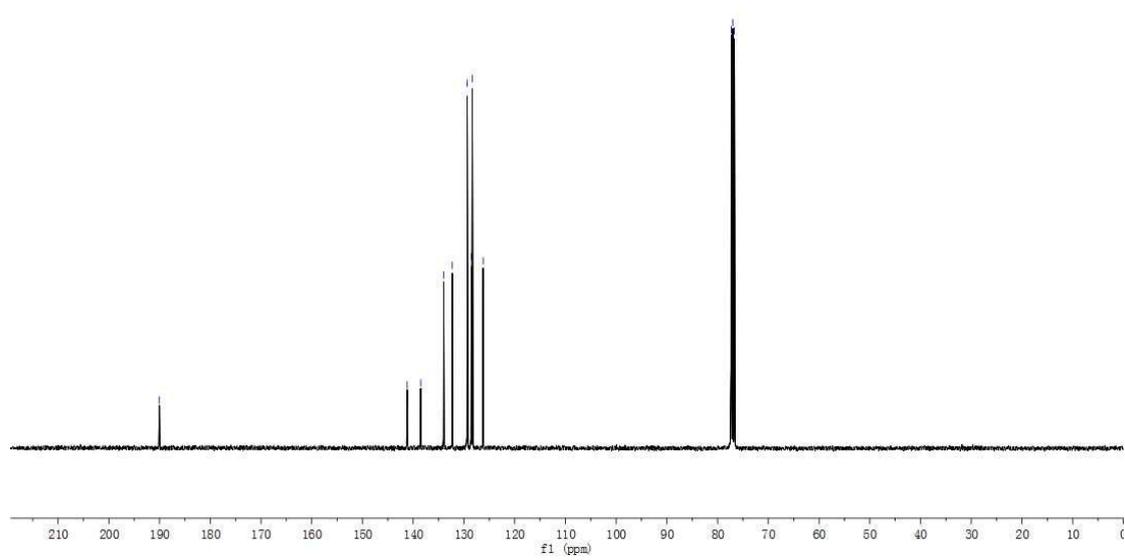
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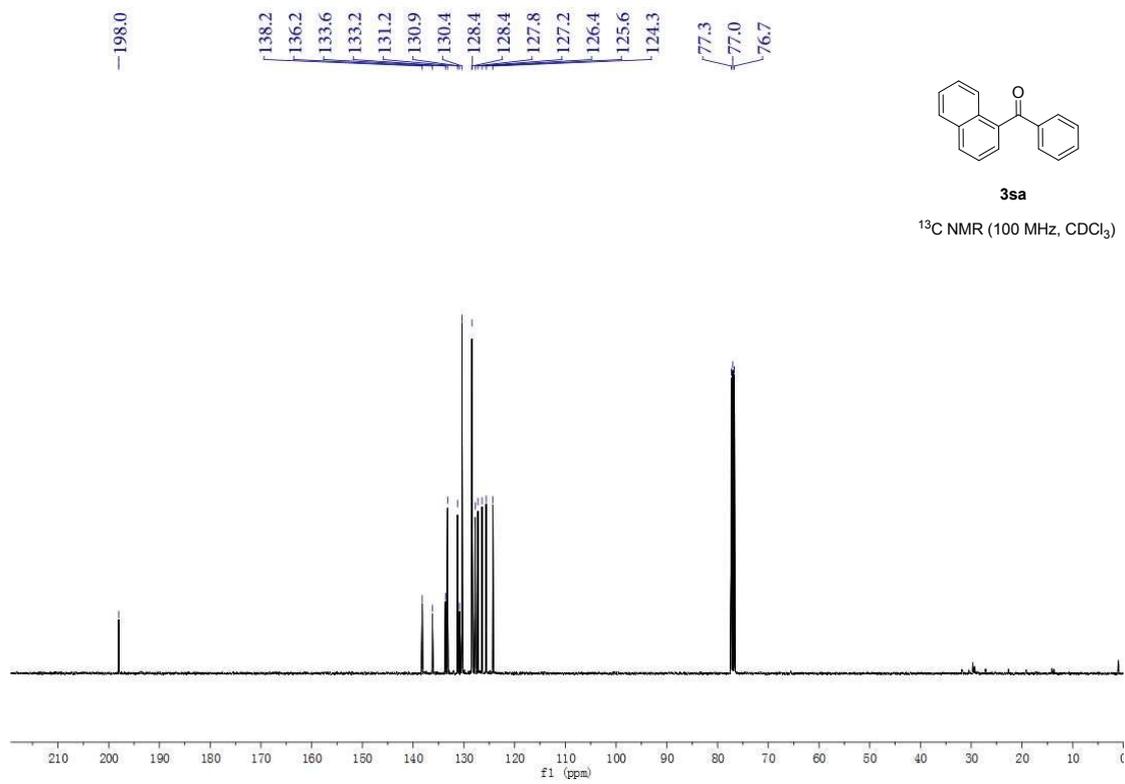
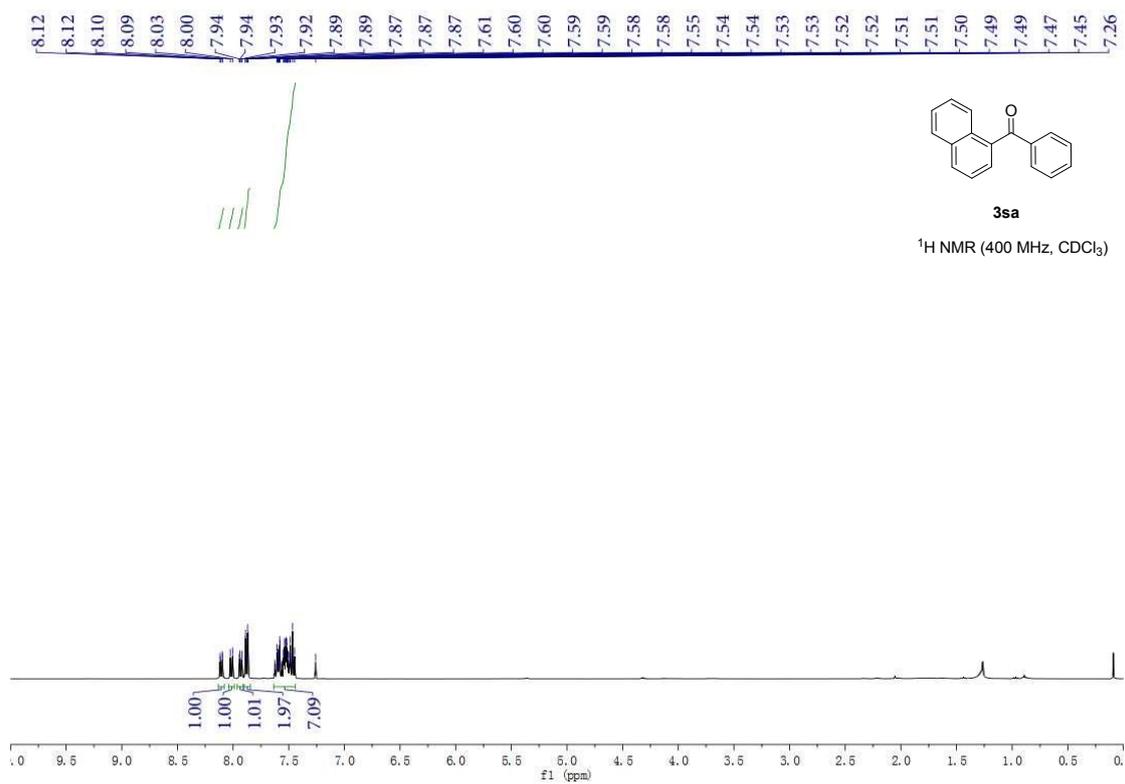
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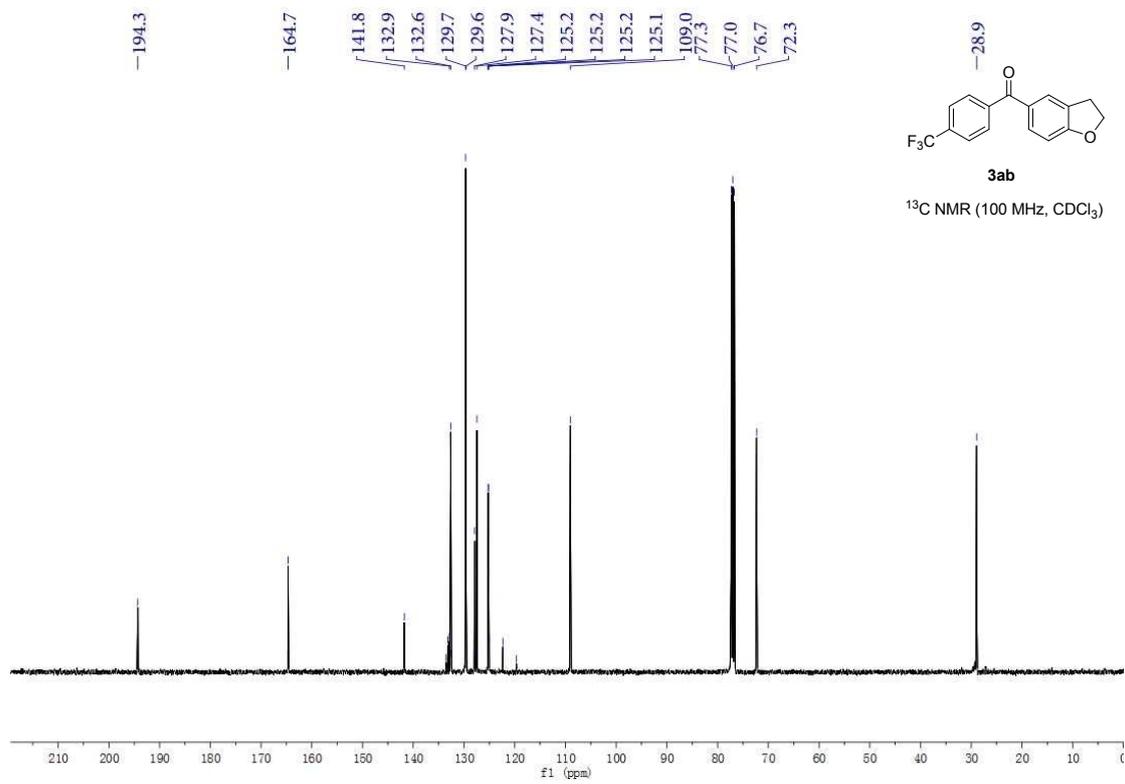
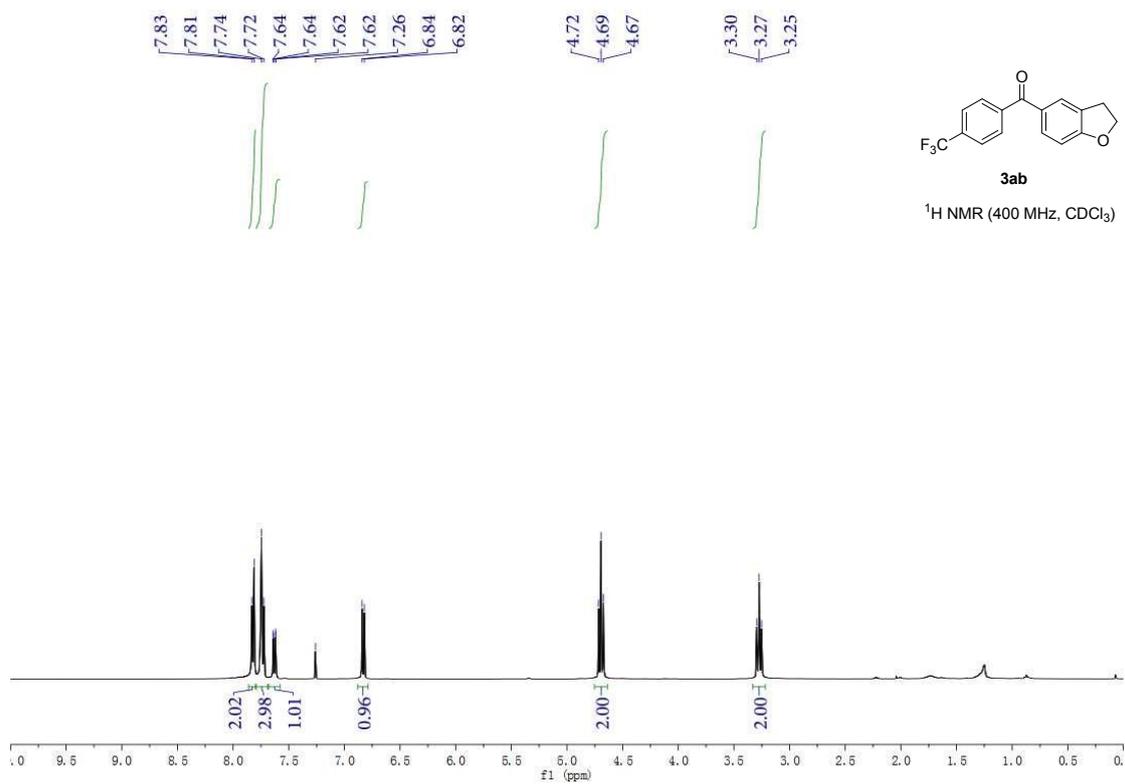


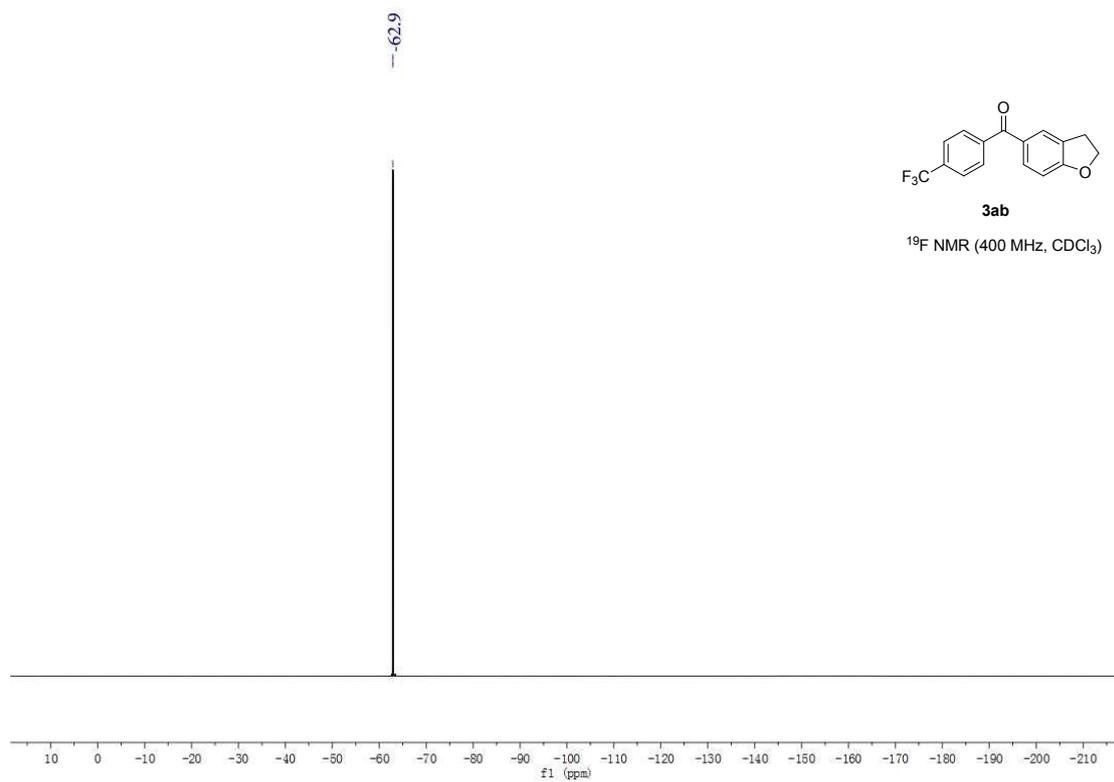
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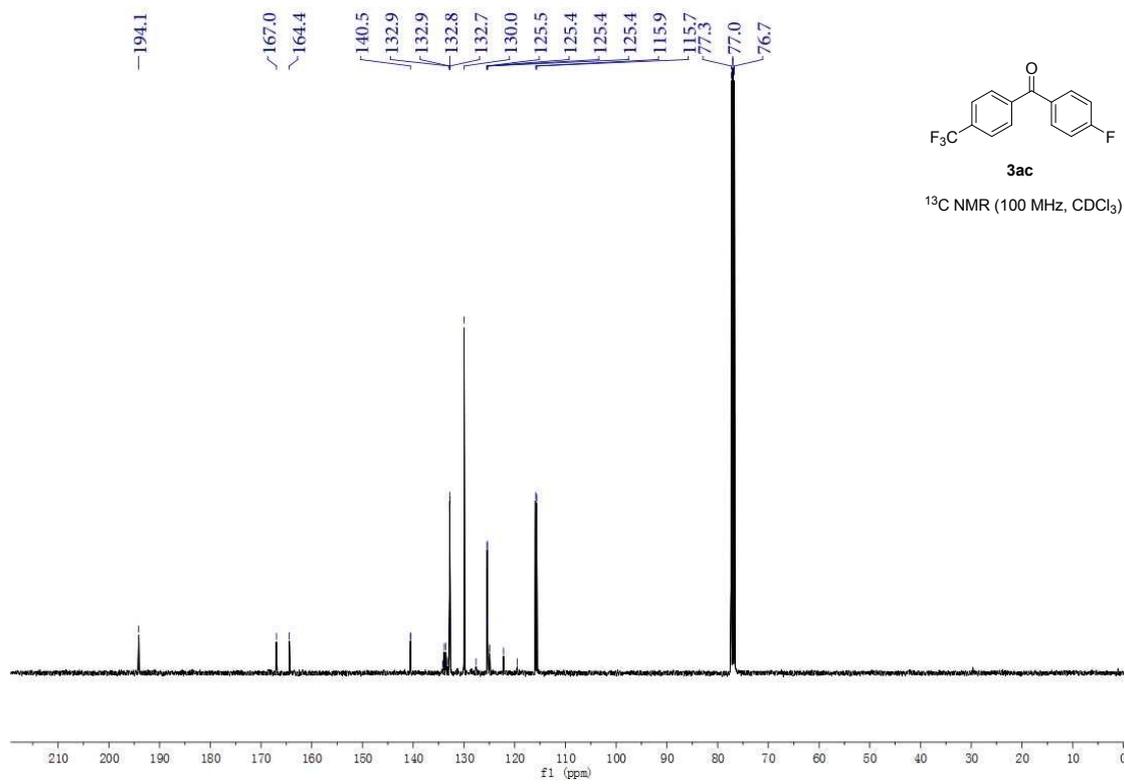
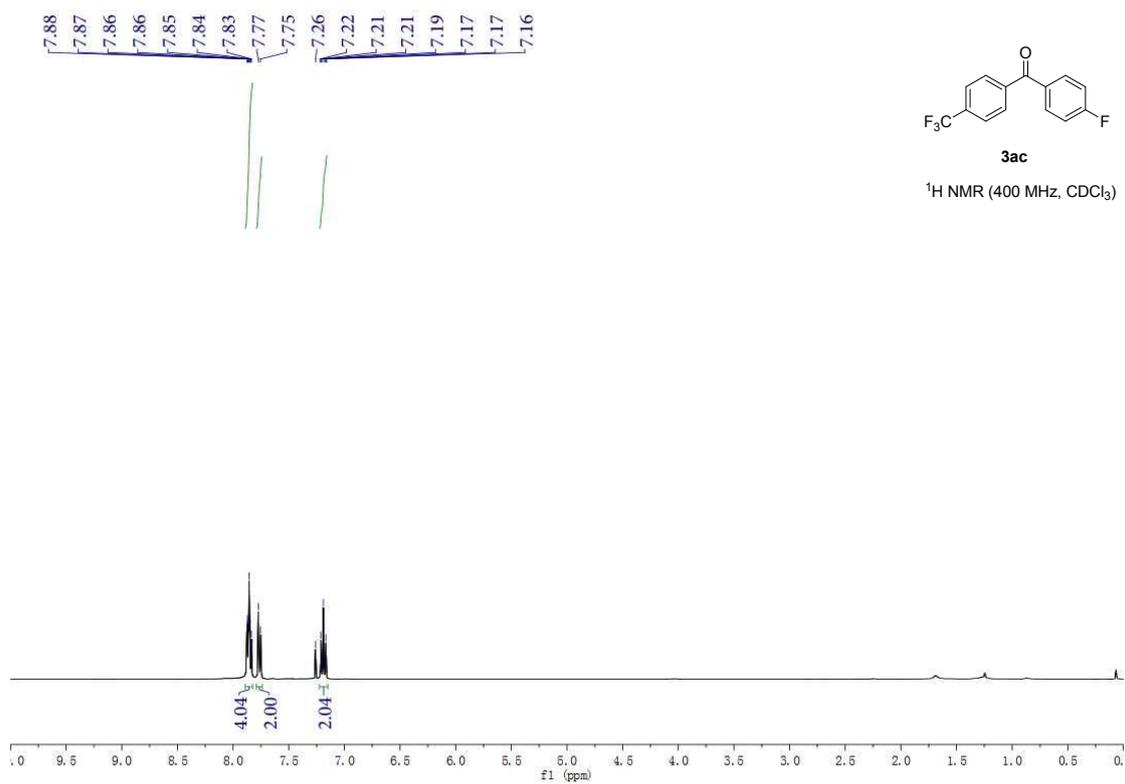
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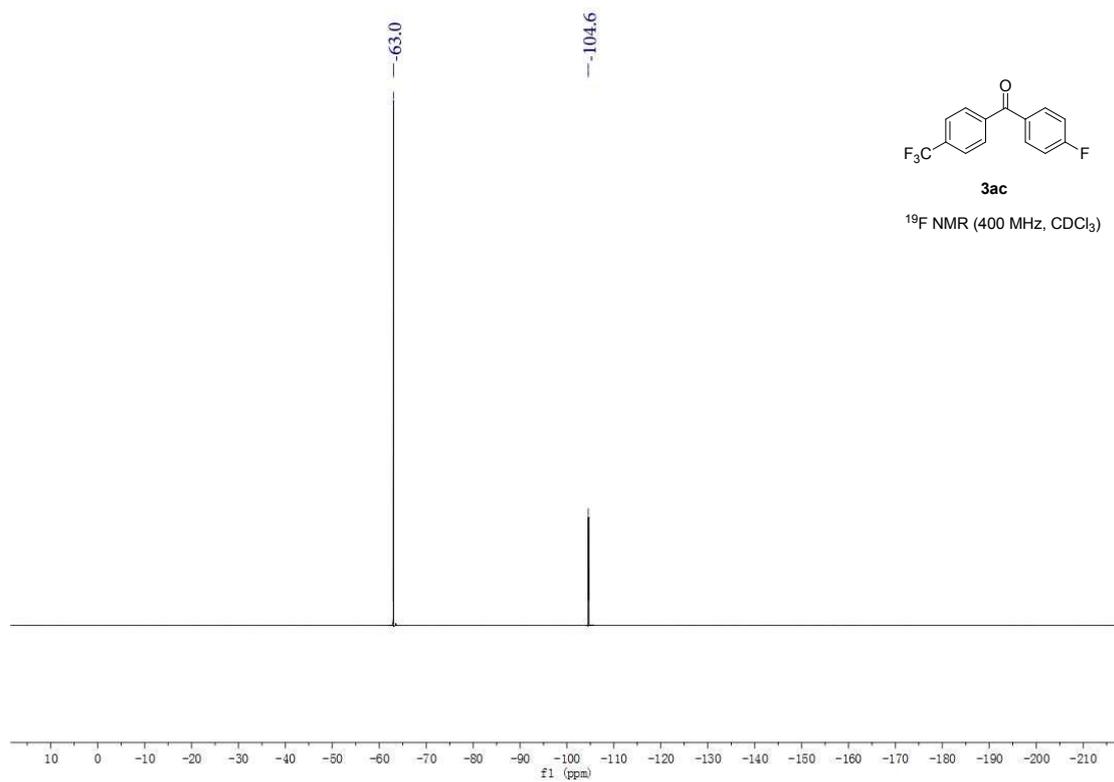


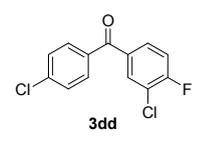
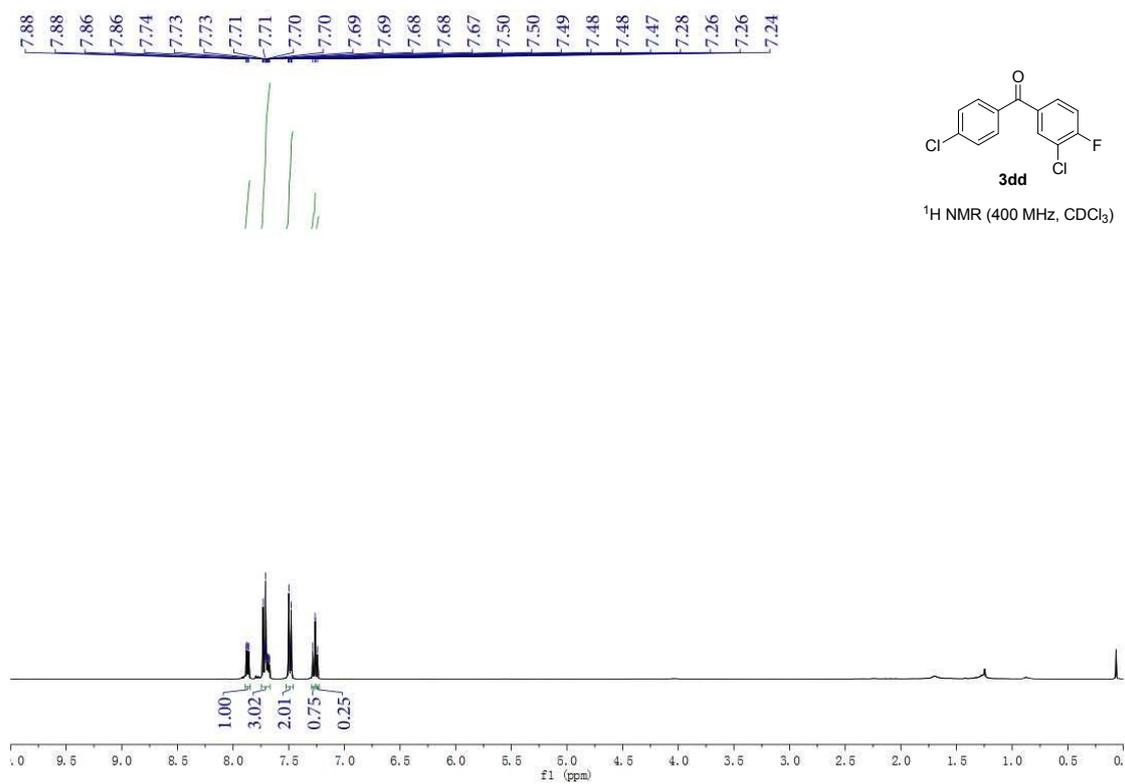




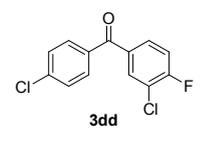
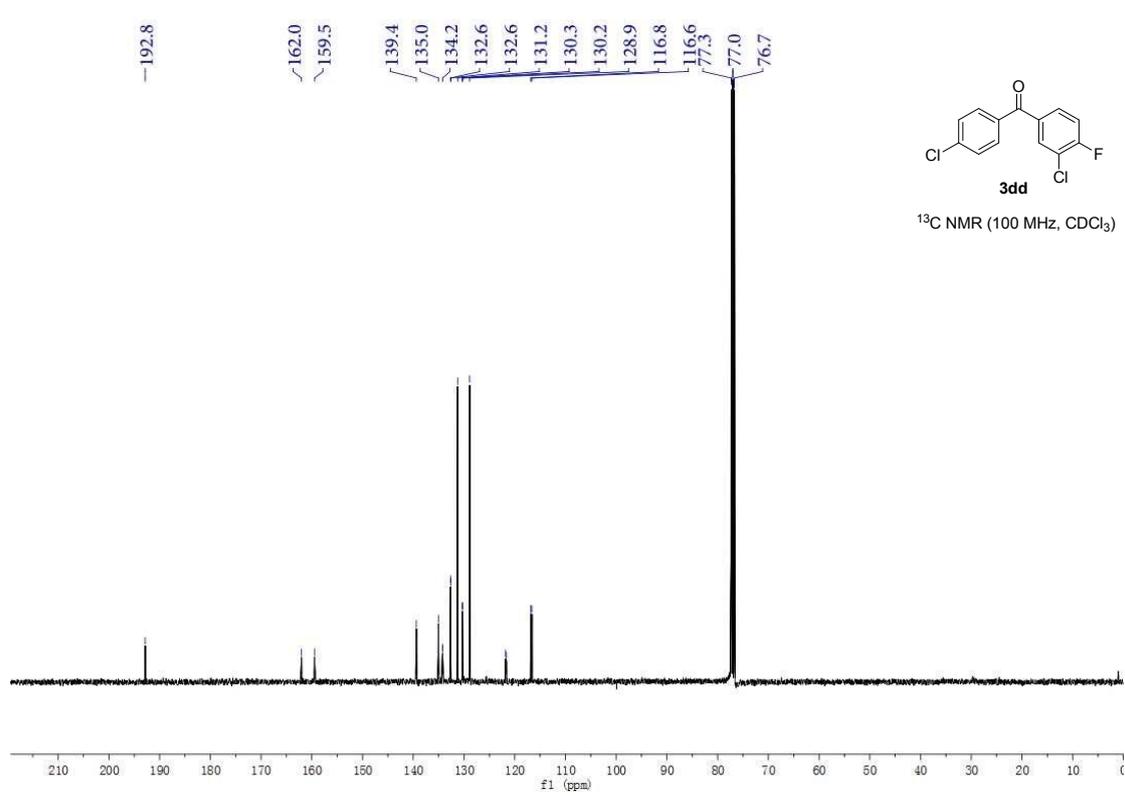






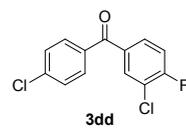


¹H NMR (400 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)

-107.8



¹⁹F NMR (400 MHz, CDCl₃)

