

Supporting Information

A simple method for arylation of secondary amides/amines through NaH-initiated aryne generation strategy

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Table of Contents

1. General Information	S2
2. Synthesis of starting materials (Figure S1-S3)	S2
3. Table S1. Optimization of the reaction conditions for amides	S10
4. The reaction of <i>N</i> -arylation of amides	S11
5. Table S2. Optimization of the reaction conditions for amines	S43
6. The reaction of <i>N</i> -arylation of amines	S43
7. Synthetic applications	S49
8. Reference	S53
9. NMR spectra	S55

1. General information

Unless otherwise noted, reagents, catalysts, and solvents were obtained from commercial suppliers and used without further purification. Reactions which proceeded under heating were performed in an oil bath. Reaction progress was monitored by thin layer chromatography (TLC) performed on glass plates coated with silica gel GF254 with 0.2 mm thickness. Chromatograms were visualized by fluorescence quenching with UV light at 254 nm or by staining using potassium permanganate. Flash column chromatography was performed using silica gel 60 (200-300 mesh). Mass spectra were obtained using a TOF MS instrument ESI source.

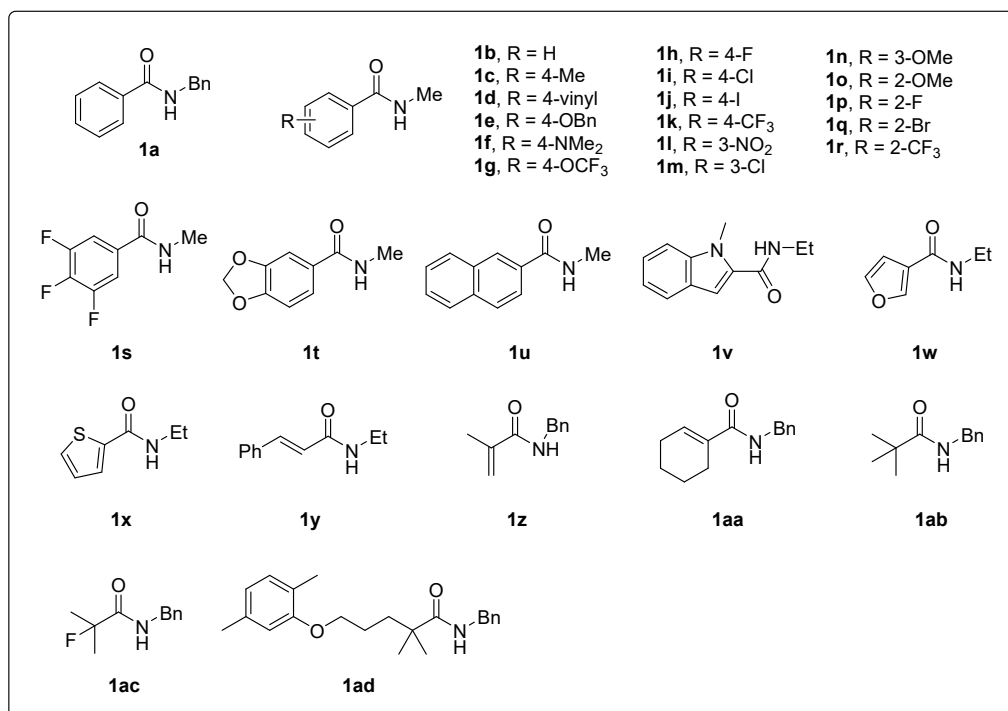
NMR spectra were recorded on Bruker AV-400 spectrometer (^1H , ^{13}C , ^{19}F at 400 MHz, 101 MHz, 376 MHz, respectively). ^1H NMR chemical shifts were determined in reference to $(\text{CH}_3)_4\text{Si}$ (TMS) at δ 0.00 ppm, or to the residual un-deuterated solvent of CDCl_3 at δ 7.26 ppm, d_6 -DMSO at δ 2.50 ppm. ^{13}C NMR chemical shifts were determined in reference to the signal of the solvent CDCl_3 triplet at δ 77.16 ppm, d_6 -DMSO multiplet at δ 39.52 ppm. Data for ^1H , ^{13}C , ^{19}F NMR were recorded as follows: chemical shift (δ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, qt = quartet of triplets, tq = triplet of quartets, br = broad).

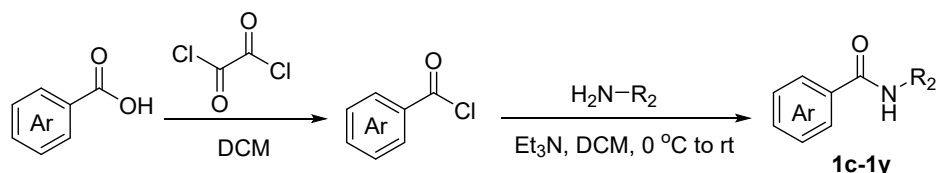
2. Synthesis of starting materials (Figure S1-S3)

Amides **1a**, **1b**, pyridones **1dd** and **1de**, iodobenzene **2a-2c**, **2j**, **2n**, **2o** and dibromobenzene **2e** are commercially available. Other substrates are synthesized according to the literature procedures as follows.

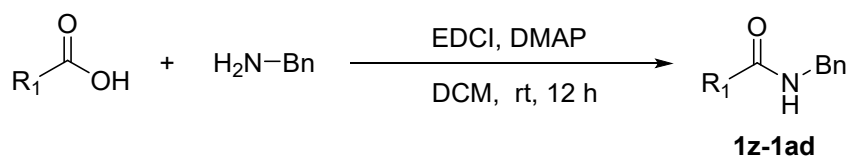
2.1 Preparation of amides

Figure S1. compounds **1a-1ad**



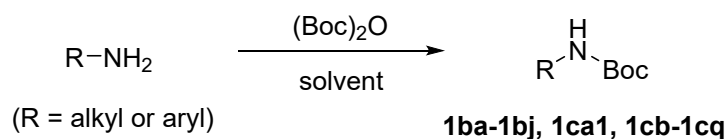
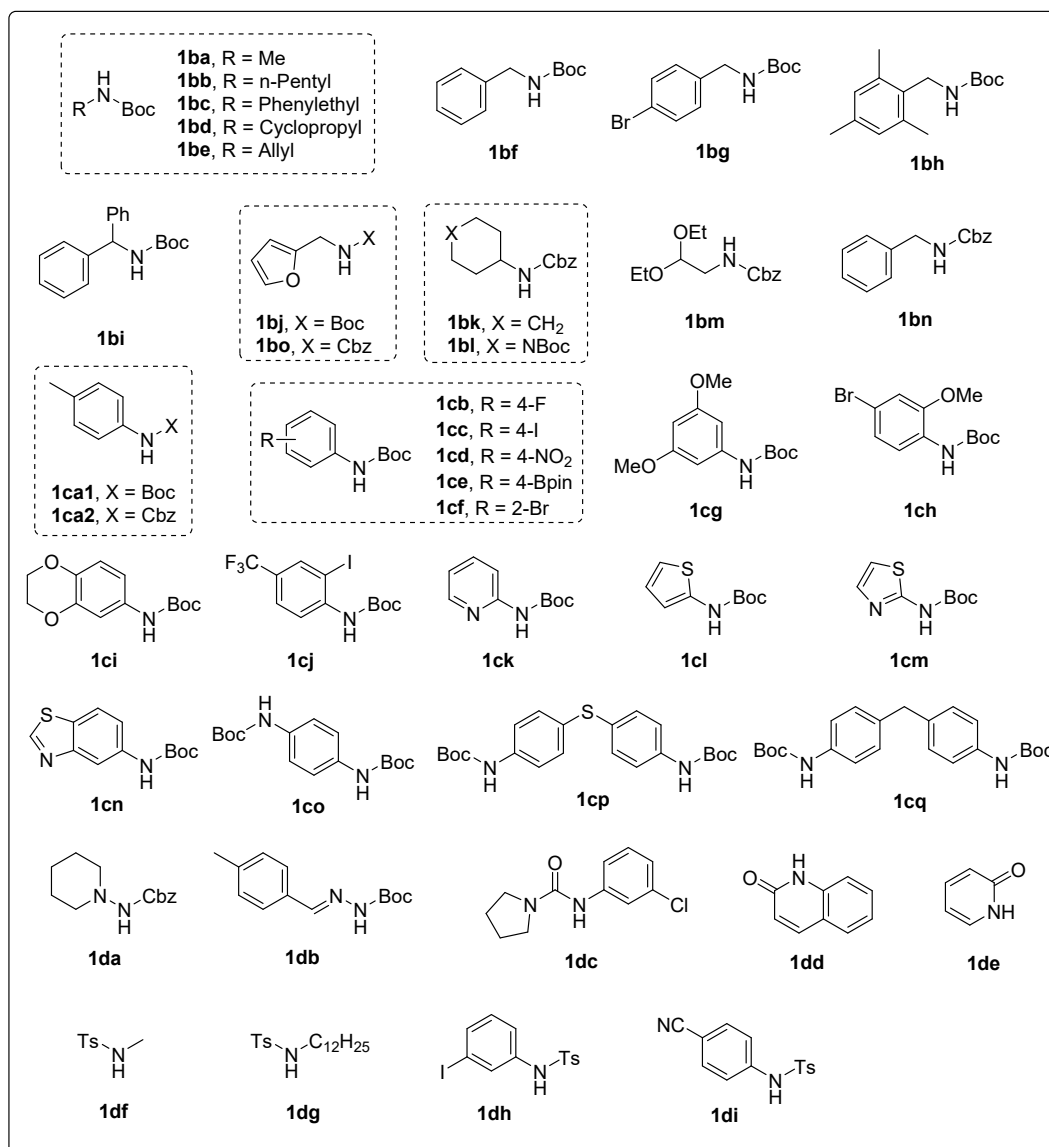


General procedure for the synthesis of substrates **1c-1y**:^[1] To a solution of carboxylic acid (10 mmol, 1.0 equiv) in CH₂Cl₂ (20 mL, 0.5 M), 1 drop of DMF was added, followed by the slow addition of oxalyl chloride (15 mmol, 1.5 equiv). The reaction mixture was stirred vigorously for 1-2 hours. After the suspension was turned into a homogeneous solution, the volatiles were removed under vacuum. The resulting crude acyl chloride was redissolved in CH₂Cl₂ (20 ml, 0.5 M). Methylamine hydrochloride (15 mmol, 1.5 equiv, for **1c-1u**) or ethylamine hydrochloride (15 mmol, 1.5 equiv, for **1v-1y**) and TEA (30 mmol, 3.0 equiv) was added sequentially dropwise into the reaction mixture under ice-water bath and then stirred at room temperature overnight. Water (80 mL) was added into the suspension and the resulting mixture was extracted with ethyl acetate (3 x 100 mL). The combined organic phase was dried over Na₂SO₄, filtrated and evaporated. The pure product was obtained by column chromatography on silica gel or crystallization using a mixture of ethyl acetate and hexane or pentane.



General procedure for the synthesis of substrates **1z-1ad**:^[2] The corresponding carboxylic acid (10 mmol, 1.0 equiv), DMAP (5 mmol, 0.5 equiv) and EDCI (15 mmol, 1.5 equiv) were dissolved in CH₂Cl₂ (20 ml, 0.5 M), and the mixture was stirred for 20 min. Then, benzylamine (10 mmol, 1.0 equiv) was added, and the mixture was stirred at room temperature for 12 h. Then 1 M HCl solution (15 mL) was added into the suspension and the resulting mixture was extracted with DCM (3 x 50 mL), followed by washing with 1 M HCl solution for two times and then saturated NaHCO₃ solution for three times. The organic phase was washed with brine, dried over Na₂SO₄, filtered and evaporated. The product was purified by column chromatography on silica gel using ethyl acetate and petroleum ether as the eluent.

Figure S2. compounds **1ba-1di**



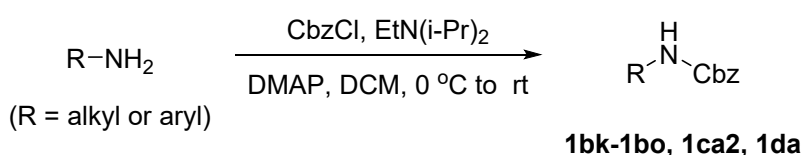
General procedure for the synthesis of substrates **1ba-1bj**, **1ca1**, **1cb-1cc**, **1ce-1ci**, **1cl-1cn**.^[3] To a solution of primary amine (10 mmol, 1.0 equiv) in EtOH (20 mL, 0.5 M) was added (Boc)₂O (12 mmol, 1.2 equiv), then the mixture was stirred at 30-50 °C for 1-12 h. The reaction mixture was concentrated directly in vacuo. The pure product was obtained by column chromatography on silica gel or crystallization using a mixture of ethyl acetate and hexane.

General procedure for the synthesis of substrates **1cd** and **1cj**.^[4] To a solution of arylamine (10 mmol, 1.0 equiv) in CH₃OH (30 mL, 0.33 M) was added (Boc)₂O (12 mmol, 1.2 equiv). After reflux at 100 °C for 4-6 h, the reaction mixture was concentrated directly in vacuo. The pure product was obtained by column

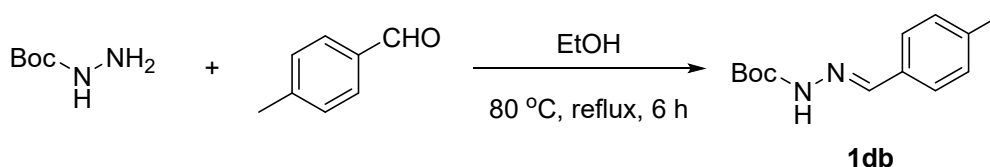
chromatography on silica gel using ethyl acetate and petroleum ether or hexane as the eluent.

Procedure for the synthesis of substrate **1ck**:^[5] To a solution of pyridin-2-amine (10 mmol, 1.0 equiv) in *t*-BuOH (20 mL, 0.5 M) was added (Boc)₂O (12 mmol, 1.2 equiv), then the mixture was stirred at 50 °C for 2 h. The reaction mixture was concentrated directly in vacuo. The pure product was obtained by crystallization using ethyl acetate and hexane.

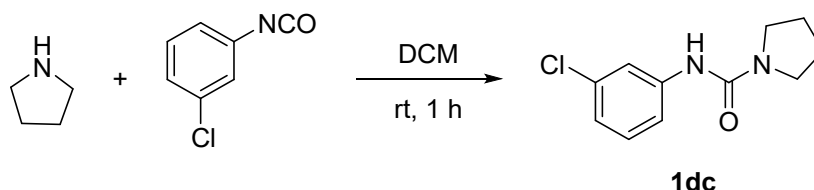
General procedure for the synthesis of substrates **1co-1cq**:^[3] To a solution of arylamine (10 mmol, 1.0 equiv) in EtOH (20 mL, 0.5 M) was added (Boc)₂O (24 mmol, 2.4 equiv), then the mixture was stirred at 40 °C for 1 h. The reaction mixture was concentrated directly in vacuo. The pure product was obtained by crystallization using a mixture of ethyl acetate and hexane.



General procedure for the synthesis of substrates **1bk-1bo, 1ca2, and 1da**:^[6] Benzyl chloroformate (11 mmol, 1.1 equiv) was added dropwise to a solution of amine (10 mmol, 1.0 equiv), DMAP (10 mmol, 1.0 equiv) and DIPEA (21 mmol, 2.1 equiv) in CH₂Cl₂ (25 mL, 0.4 M) at 0 °C. The solution was warmed to room temperature and stirred overnight. The reaction mixture was quenched with 1 M HCl solution (15 mL) and extracted with CH₂Cl₂ (3 x 20 mL). The combined organic layer was washed with NaHCO₃ saturated solution and brine successively. Then, the organic layer was dried over Na₂SO₄, filtered off and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel using ethyl acetate and petroleum ether as the eluent.

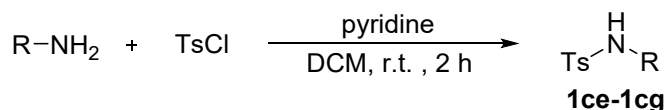


Procedure for the synthesis of substrate **1db**:^[7] To the solution of *tert*-butyl hydrazinecarboxylate (10 mmol, 1.0 equiv) in EtOH (40 mL, 0.25 M) was added 4-methylbenzaldehyde (10 mmol, 1.0 equiv) at room temperature. After reflux at 80 °C for 6 h, the reaction mixture was concentrated directly in vacuo. The pure product was obtained by crystallization using a mixture of ethyl acetate and hexane or pentane.



Procedure for the synthesis of substrate **1dc**:^[8] To the solution of pyrrolidine (10 mmol, 1.0 equiv) in CH₂Cl₂ (40 mL, 0.25 M) was added 3-chlorophenyl isocyanate (10 mmol, 1.0 equiv) at 0 °C, then the mixture was stirred at room temperature for 1 h. The reaction

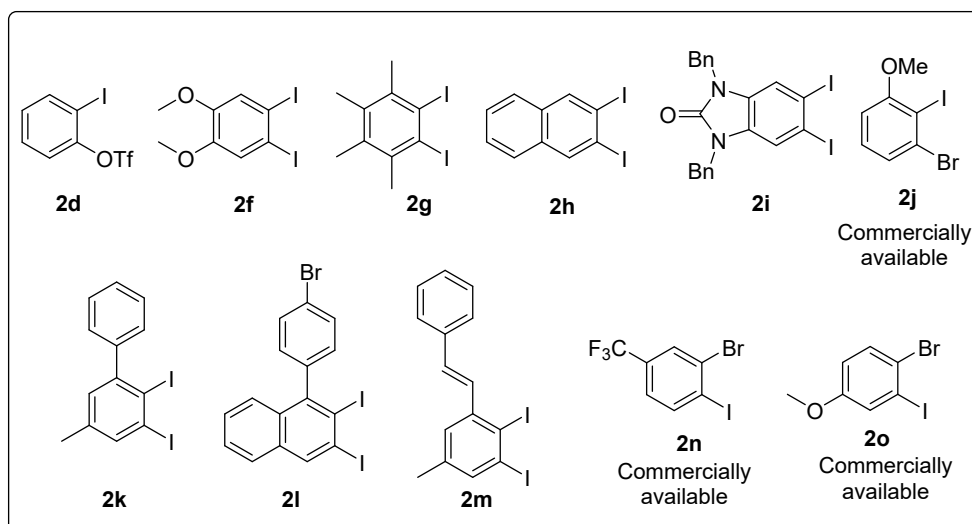
mixture was concentrated directly in vacuo. The pure product was obtained by crystallization using a mixture of ethyl acetate and hexane.



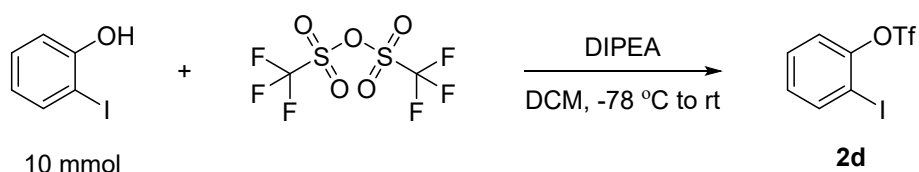
General procedure for the synthesis of substrates **1ce-1cg**:^[9] To a solution of aniline (5 mmol) in DCM (20 mL) was added pyridine (791 mg, 10 mmol) and TsCl (1.91 g, 10 mmol). The resulting solution was stirred for 2 h. Then the mixture was washed with HCl (1 M aqueous solution, 20 mL) and water (20 mL). After that, the organic layer was dried over Na₂SO₄ and concentrated under the reduced pressure. The obtained residue was purified by silica gel column chromatography using ethyl acetate and petroleum ether as the eluent to afford the corresponding secondary sulfonamide.

2.2 Preparation of *o*-diiodoarenes

Figure S3. compounds **2d-2o**



2-Iodophenyl trifluoromethanesulfonate (**2d**):

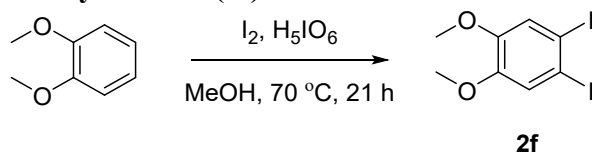


2-Iodophenol (2.20 g, 10.0 mmol, 1.0 equiv) was dissolved in CH₂Cl₂ (25 mL) in a nitrogen-filled Schlenk flask and cooled to -78 °C. After the dropwise addition of DIPEA (2.15 mL, 1.64 g, 12.7 mmol, 1.3 equiv) and Tf₂O (2.30 mL, 3.86 g, 13.7 mmol, 1.4 equiv), the reaction mixture was stirred at -78 °C for 45 minutes and warmed to room temperature overnight. Addition of water was followed by extraction with ethyl acetate (three times). The combined organic phases were dried over Na₂SO₄, filtered and concentrated. The pure product was obtained as a colorless oil by column chromatography on silica gel using pentane as eluent, yield: 89%.

¹H NMR (400 MHz, CDCl₃): δ 7.91 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.45 – 7.40 (m, 1H), 7.33 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.11 (td, *J* = 7.6, 1.4 Hz, 1H).

LR-MS (ESI): m/z 352.9 $[M+H]^+$.

1,2-Diiodo-4,5-dimethoxybenzene (2f):^[10]

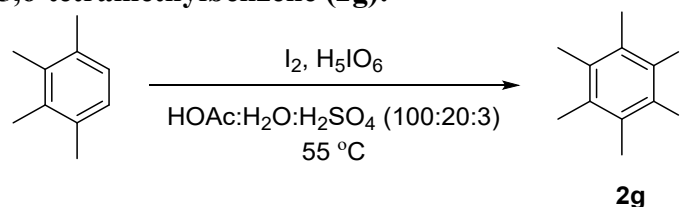


Iodine (10.6 g, 42 mmol, 0.8 equiv) and H_5IO_6 (4.8 g, 21 mmol, 0.4 equiv) were dissolved in MeOH (80 mL, 0.66 M). 1,2-Dimethoxybenzene (7.2 g, 52.1 mmol, 1.0 equiv) was added and the solution stirred at 70 °C for 21 h. The reaction mixture was cooled to room temperature. Saturated $NaHSO_3$ solution (5 mL) was added to reduce excessive iodine, filtered, and the filter cake was carefully collected and dried in an oven at 55 °C to obtain the title compound as a white solid (17.5 g, 86%).

1H NMR (400 MHz, $CDCl_3$): δ 7.24 (s, 2H), 3.84 (s, 6H).

LR-MS (ESI): m/z 390.8 $[M+H]^+$.

1,2-Diiodo-3,4,5,6-tetramethylbenzene (2g):^[11]

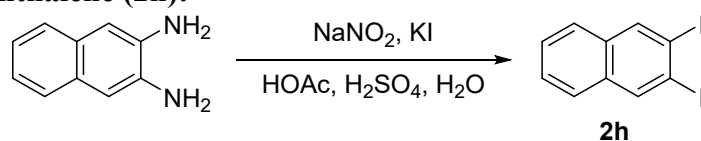


A mixture of 1,2,3,4-tetramethylbenzene (7.38 g, 55.0 mmol, 1.0 equiv), H_5IO_6 (3.58 g, 15.7 mmol, 0.29 equiv) and iodine (11.96 g, 47.1 mmol, 0.86 equiv) were heated in a mixture of acetic acid, water and sulfuric acid (100:20:3, 100 mL) to 55 °C. The mixture was stirred overnight at 55 °C and then poured into a dilute solution of sodium bisulfite. The tan solid precipitate was filtered, and the remainder of the product in filtrate was extracted with dichloromethane for three times. The organic layer was evaporated, combined with the above precipitate, and recrystallized in methanol to afford **2g** as a white solid (17.0 g, 80%).

1H NMR (400 MHz, $CDCl_3$): δ 2.69 (s, 6H), 2.28 (s, 6H).

LR-MS (ESI): m/z 386.9 $[M+H]^+$.

2,3-Diiodonaphthalene (2h):^[12]

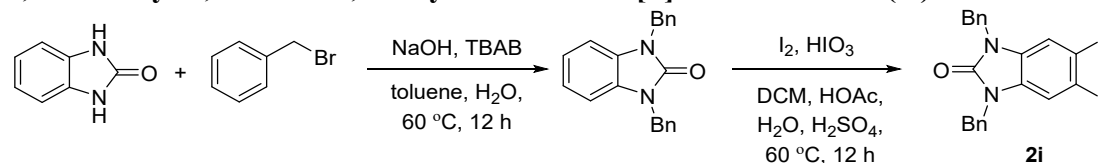


To a solution of $NaNO_2$ (1.06 g, 15.4 mmol, 2.3 equiv) in concentrated sulfuric acid (15 mL) was added a solution of 2,3-diaminonaphthalene (1.06 g, 6.70 mmol, 1.0 equiv) in acetic acid (20 mL) at 0 °C. After stirring for 10 min at 0 °C, the purple suspension was added to a solution of potassium iodide (11.1 g, 66.9 mmol, 10.0 equiv) in water (30 mL). After stirring at 60 °C for 1 h, a saturated aqueous solution of $NaHCO_3$ was added, and the reaction mixture was extracted with ethyl acetate. The extract was washed with a saturated aqueous solution of sodium thiosulfate and brine, and dried over anhydrous Na_2SO_4 . After removal of the solvent, the residue was purified by column chromatography to obtain the pure product **2h** as a white solid (1.01 g, 39%).

1H NMR (400 MHz, $CDCl_3$): δ 8.41 (s, 2H), 7.67 (dd, J = 6.0, 3.3 Hz, 2H), 7.49 (dd, J = 6.3, 3.2 Hz, 2H).

LR-MS (ESI): m/z 380.8 $[M+H]^+$.

1,3-Dibenzyl-5,6-diiodo-1,3-dihydro-2H-benzo[d]imidazol-2-one (2i):^[13]



A mixture of NaOH (3.05 g, 76.2 mmol, 3.8 equiv), TBAB (0.65 g, 2.0 mmol, 2.0 equiv), 2-hydroxybenzimidazole (2.68 g, 20.0 mmol, 1.0 equiv) and benzyl bromide (8.20 g, 48.0 mol, 2.4 equiv) in a mixture of water (15 mL) and toluene (15 mL) was stirred at 60 °C for 12 h and cooled to room temperature. The reaction mixture was quenched with water and extracted with ethyl acetate (three times). The combined organic layers were washed with brine, dried over Na₂SO₄, filtered and concentrated under reduced pressure. The pure intermediate (5.53 g, 88%) was obtained by column chromatography on silica gel using ethyl acetate and petroleum ether as the eluent.

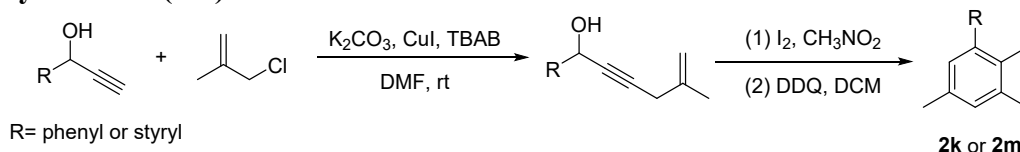
A mixture of the above intermediate (5.53 g, 17.6 mmol, 1.0 equiv), HIO₃ (1.55 g, 8.8 mmol, 0.5 equiv), and I₂ (4.52 g, 17.6 mmol, 1.0 equiv) in a mixture of solvents CH₂Cl₂ (20 mL), acetic acid (17.6 mL), and aqueous sulfuric acid solution (20% w/w, 10.6 mL) were stirred at 60 °C for 12 h and then cooled to room temperature. The reaction mixture was slowly poured into water, and extracted with CH₂Cl₂ for three times. The combined organic layers were washed with saturated aqueous NaHSO₃ solution and brine successively, dried over Na₂SO₄, filtered and concentrated under reduced pressure. The pure product **2i** (6.28 g, 63%) was obtained by crystallization using a mixture of ethyl acetate and hexane.

¹H NMR (400 MHz, CDCl₃): δ 7.37 – 7.25 (m, 12H), 5.03 (s, 4H).

¹³C NMR (101 MHz, CDCl₃): δ 154.0, 135.5, 130.8, 129.1, 128.1, 127.4, 118.2, 97.2, 45.2.

HRMS (ESI-TOF): calculated for [C₂₁H₁₇I₂N₂O (M + H)]⁺: 566.9430, found: 566.9431.

2,3-Diiodo-5-methyl-1,1'-biphenyl (2k) and (E)-1,2-diiodo-5-methyl-3-styrylbenzene (2m)^{[14]:}

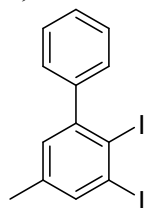


R= phenyl or styryl

To a stirred solution of 1-phenylprop-2-yn-1-ol (1.32 g, 10 mmol, 1.0 equiv) or (*E*)-1-phenylpent-1-en-4-yn-3-ol (1.58 g, 10 mmol, 1.0 equiv) in dry DMF (10 mL) under nitrogen were sequentially added K₂CO₃ (1.93 g, 14 mmol, 1.4 equiv), tetrabutylammonium bromide (483 mg, 1.5 mmol, 0.15 equiv), and copper(I) iodide (96 mg, 0.5 mmol, 0.05 equiv) at room temperature. After 15 min, 3-chloro-2-methylprop-1-ene (1.36 g, 15 mmol, 1.5 equiv) was added. The reaction mixture was stirred for 24 h. Then it was poured into water and extracted with EtOAc (3 x 30 mL). The combined organic layers were washed with water (3 x 30 mL) and brine, dried over Na₂SO₄, filtered, and the solvents were removed under reduced pressure. The residue was purified by flash column chromatography to give the intermediate as a yellow oil.

To a solution of the above intermediate (8.0 mmol, 1.0 equiv) in wet CH_3NO_2 (115 mL, 0.07 M) was added I_2 (3.66 g, 14.4 mmol, 1.8 equiv) at room temperature and stirred for 2 h. After completion of the reaction monitored by TLC, an appropriate amount of saturated NaHSO_3 solution was added to reduce excessive iodine, and then extracted with ethyl acetate (3 x 50 mL). The organic phase was washed with brine, dried over Na_2SO_4 , filtered and evaporated under reduced pressure. The residue was dissolved in CH_2Cl_2 (160 mL, 0.05 M), and DDQ (3.63 g, 16 mmol, 2.0 equiv) was added at room temperature. When the reaction was completed as determined by TLC, the reaction mixture was filtered, and the filtrate was evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford corresponding 2,3-diiodobenzenes **2k** or **2m**.

2,3-Diiodo-5-methyl-1,1'-biphenyl (**2k**):



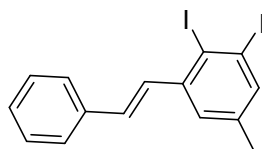
The title compound was obtained as colorless oil, yield 66%.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.73 (s, 1H), 7.43 – 7.34 (m, 3H), 7.25 – 7.20 (m, 2H), 7.02 (s, 1H), 2.25 (s, 3H).

LR-MS (ESI): m/z 420.8 $[\text{M}+\text{H}]^+$.

The $^1\text{H NMR}$ of **2k** are consistent with the reported spectra^[14].

(*E*)-1,2-diiodo-5-methyl-3-styrylbenzene (**2m**):



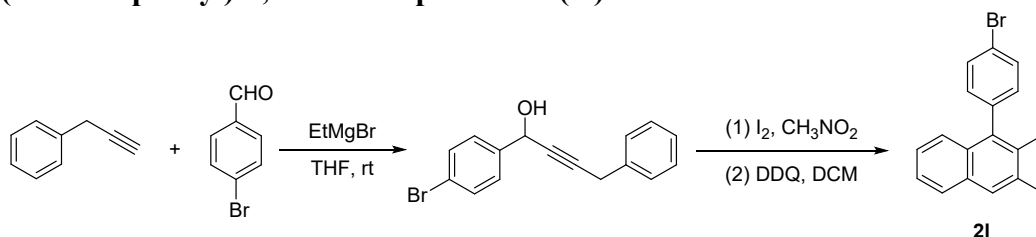
The title compound was obtained as a yellow oil, yield 61%.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.66 (s, 1H), 7.52 (d, $J = 7.7$ Hz, 2H), 7.39 – 7.25 (m, 5H), 6.79 (d, $J = 15.9$ Hz, 1H), 2.26 (s, 3H).

LR-MS (ESI): m/z 446.9 $[\text{M}+\text{H}]^+$.

The $^1\text{H NMR}$ of **2m** are consistent with the reported spectra^[14].

1-(4-Bromophenyl)-2,3-diiodonaphthalene (**2l**)^[15]:



To a solution of prop-2-yn-1-ylbenzene (6.0 mmol, 1.2 equiv) in THF (5 mL) was added EtMgBr (5.5 mL, 1.0 M in THF, 1.1 equiv) at room temperature, and the reaction mixture was stirred for 1 h at 50 °C. 4-Bromobenzaldehyde (925 mg, 5.0 mmol, 1.0 equiv) in THF (1 mL) was added. The reaction mixture was warmed to room temperature for 1 h and then quenched with aqueous NH_4Cl . The reaction mixture was diluted with ethyl acetate, washed with water and saturated brine, dried over Na_2SO_4

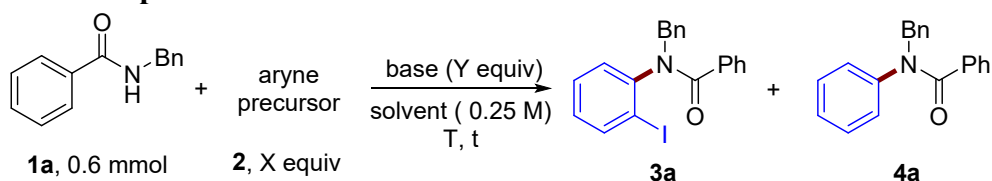
and evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford propargylic alcohol (1.2 g, 80%).

To a solution of propargylic alcohol (1.2 g, 4.0 mmol, 1.0 equiv) in wet CH_3NO_2 (60 mL, 0.067 M) was added I_2 (2.03 g, 8.0 mmol, 2.0 equiv) at room temperature and stirred for 2 h. After completion of the reaction monitored by TLC, an appropriate amount of saturated NaHSO_3 solution was added to reduce excessive iodine, and then extracted with ethyl acetate (3 x 30 mL). The organic phase was washed with brine, dried over Na_2SO_4 , filtered and evaporated under reduced pressure. The residue was dissolved in CH_2Cl_2 (80 mL) at room temperature without purification, and then DDQ (2.72 g, 12 mmol, 3.0 equiv) was added. When the reaction was completed as determined by TLC, the reaction mixture was filtered, and the filtrate was evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford the title compound **2I** as a yellow solid (1.35 g, 63%).

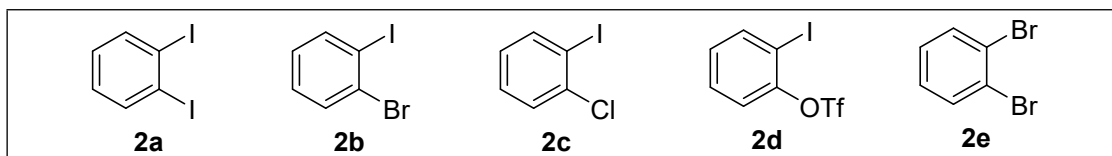
$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 8.53 (s, 1H), 7.72 (d, $J = 8.2$ Hz, 1H), 7.69 – 7.63 (m, 2H), 7.48 (ddd, $J = 8.1, 6.8, 1.1$ Hz, 1H), 7.35 (ddd, $J = 8.2, 6.8, 1.3$ Hz, 1H), 7.24 (d, $J = 8.7$ Hz, 1H), 7.09 – 7.04 (m, 2H).

LR-MS (ESI): m/z 535.7 $[\text{M}+\text{H}]^+$.

3. Table S1. Optimization of the reaction conditions for amides

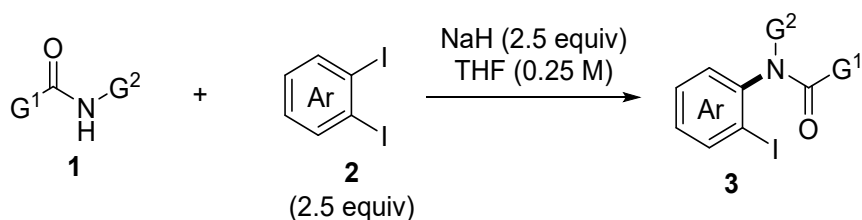


entry	aryne precursor (X equiv)	base (Y equiv)	temp (°C)	solvent	time	yield 3a (%) ^a	yield 4a (%) ^a
1	2a (2.0)	NaH (3.0)	30	THF	15 h	54	31
2	2a (2.0)	NaH (2.5)	30	THF	15 h	77	7
3	2a (2.0)	NaH (2.0)	30	THF	15 h	86	0
4	2a (2.5)	NaH (2.0)	30	THF	15 h	88	0
5	2a (3.0)	NaH (2.0)	30	THF	15 h	88	0
6	2a (2.5)	NaH (1.5)	30	THF	15 h	73	0
7	2a (2.5)	NaH (2.5)	30	THF	15 h	90	0
8	2a (2.5)	NaH (3.0)	30	THF	15 h	85	4
9	2a (3.0)	NaH (2.5)	30	THF	15 h	88	0
10	2a (2.5)	NaH (2.5)	30	THF	1 h	90	0
11	2b (3.0)	NaH (2.5)	30	THF	15 h	74	0
12	2c (3.0)	NaH (2.5)	30	THF	15 h	8	4
13	2d (3.0)	NaH (2.5)	30	THF	15 h	67	7
14	2e (3.0)	NaH (2.5)	30	THF	15 h	0	0
15	2a (2.5)	NaH (2.5)	0	THF	24 h	0	0
16	2a (2.5)	NaH (2.5)	50	THF	1 h	88	0
17	2a (2.0)	KH (2.5)	30	THF	1 h	11	2
18	2a (2.0)	CaH_2 (2.5)	50	THF	1 h	<5	0
19	2a (2.0)	LiH (2.5)	50	THF	1 h	<5	0
20	1a (2.0)	NaH (2.5)	30	DMA	1 h	70	8
21	1a (2.0)	NaH (2.5)	30	DME	1 h	68	2



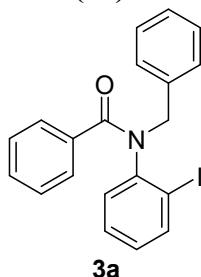
^aIsolated yield. DMA = *N,N*-Dimethylacetamide, DME = 1,2-Dimethoxyethane.

4. The reaction of *N*-arylation of amides



General procedure: To NaH (60% in oil, 60 mg, 1.5 mmol, 2.5 equiv) in a vial was slowly added a solution of amides **1** (0.6 mmol, 1.0 equiv) in anhydrous THF (2.0 mL). After stirred at rt for 1-3 min, aryne precursor **2** (1.5 mmol, 2.5 equiv) in anhydrous THF (0.4 mL) was added and the mixture was warmed to 30 °C. After stirring at 30 °C for the indicated time, the reaction was slowly poured into ice water (5 mL). The mixture was extracted with ethyl acetate for three times. The combined organic layers were dried over Na₂SO₄, concentrated under vacuum. The pure product **3** was obtained by column chromatography on silica gel.

N-benzyl-*N*-(2-iodophenyl)benzamide (**3a**):



The title compound was prepared according to the general procedure for 1 h, obtained as a yellow solid, 223 mg, yield 90%. Melting Point: 88-89 °C.

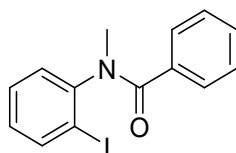
Scale-up: when **1a** (1.0 g, 4.74 mmol) was employed in this reaction, 1.8 g of **3a** was obtained, 92% yield.

¹H NMR (400 MHz, CDCl₃): δ 7.77 (d, *J* = 7.8 Hz, 1H), 7.37 (d, *J* = 7.3 Hz, 2H), 7.28 (m, 5H), 7.20 (t, *J* = 7.2 Hz, 1H), 7.13 (t, *J* = 7.2 Hz, 2H), 7.01 (t, *J* = 7.4 Hz, 1H), 6.83 (t, *J* = 7.3 Hz, 1H), 6.62 (d, *J* = 7.8 Hz, 1H), 5.82 (d, *J* = 14.2 Hz, 1H), 4.27 (d, *J* = 14.2 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 170.5, 144.6, 140.2, 136.9, 136.0, 132.2, 129.8, 129.6, 129.2, 128.6, 128.5, 128.3, 127.7, 127.6, 100.1, 52.5.

HRMS (ESI-TOF): calculated for [C₂₀H₁₇INO (M + H)]⁺: 414.0355, found: 414.0351. The ¹H NMR and ¹³C NMR of **3a** are consistent with the reported spectra^[16].

N-(2-iodophenyl)-*N*-methylbenzamide (**3b**):



3b

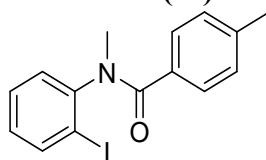
The title compound was prepared according to the general procedure for 3 h, obtained as a yellowish solid, 172 mg, yield 85%. Melting Point: 139-140 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.79 (d, *J* = 7.8 Hz, 1H), 7.36 (d, *J* = 7.3 Hz, 2H), 7.20 (t, *J* = 7.2 Hz, 2H), 7.15 (t, *J* = 7.2 Hz, 2H), 7.09 (d, *J* = 7.6 Hz, 1H), 6.89 (t, *J* = 7.4 Hz, 1H), 3.38 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.8, 147.0, 140.2, 135.7, 130.2, 129.8, 129.4, 129.1, 128.4, 127.7, 99.1, 37.6.

HRMS (ESI-TOF): calculated for [C₁₄H₁₃INO (M + H)]⁺: 338.0042, found: 338.0039. The ¹H NMR and ¹³C NMR of **3b** are consistent with the reported spectra^[17].

***N*-(2-iodophenyl)-*N*,4-dimethylbenzamide (3c):**



3c

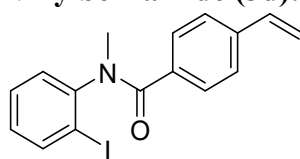
The title compound was prepared according to the general procedure for 10 h, obtained as a yellowish solid, 196 mg, yield 93%. Melting Point: 102-103 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.80 (d, *J* = 7.4 Hz, 1H), 7.30 – 7.16 (m, 3H), 7.09 (d, *J* = 7.2 Hz, 1H), 6.98 – 6.85 (m, 3H), 3.36 (s, 3H), 2.23 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.8, 147.2, 140.1, 139.9, 132.7, 130.1, 129.4, 128.9, 128.5, 128.3, 99.1, 37.6, 21.4.

HRMS (ESI-TOF): calculated for [C₁₅H₁₅INO (M + H)]⁺: 352.0198, found: 352.0197. The ¹H NMR and ¹³C NMR of **3c** are consistent with the reported spectra^[18, 19].

***N*-(2-iodophenyl)-*N*-methyl-4-vinylbenzamide (3d):**



3d

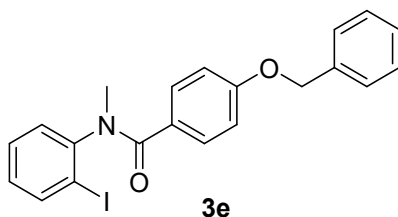
The title compound was prepared according to the general procedure for 4 h, obtained as a yellowish solid, 179 mg, yield 82%.

¹H NMR (400 MHz, CDCl₃): δ 7.79 (d, *J* = 7.3 Hz, 1H), 7.32 (d, *J* = 7.8 Hz, 2H), 7.18 (m, 3H), 7.10 (d, *J* = 7.5 Hz, 1H), 6.89 (t, *J* = 7.9 Hz, 1H), 6.58 (m, 1H), 5.68 (d, *J* = 17.7 Hz, 1H), 5.22 (d, *J* = 10.8 Hz, 1H), 3.36 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.5, 147.1, 140.2, 138.9, 136.1, 134.9, 130.1, 129.5, 129.1, 128.9, 125.5, 115.3, 99.1, 37.7.

HRMS (ESI-TOF): calculated for [C₁₆H₁₅INO (M + H)]⁺: 364.0198, found: 364.0199.

4-(Benzyloxy)-*N*-(2-iodophenyl)-*N*-methylbenzamide (3e):



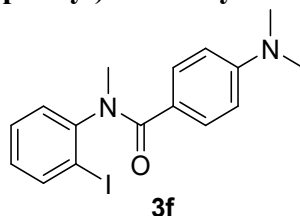
The title compound was prepared according to the general procedure for 6 h, obtained as a yellow solid, 239 mg, yield 90%. Melting Point: 94-95 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.82 (d, *J* = 7.8 Hz, 1H), 7.40 – 7.28 (m, 7H), 7.24 (t, *J* = 7.2 Hz, 1H), 7.10 (d, *J* = 6.7 Hz, 1H), 6.92 (t, *J* = 7.3 Hz, 1H), 6.74 (d, *J* = 7.0 Hz, 2H), 4.97 (s, 2H), 3.36 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.3, 159.9, 147.4, 140.2, 136.4, 130.5, 130.0, 129.5, 128.9, 128.6, 128.1, 128.0, 127.4, 113.8, 99.1, 69.9, 37.8.

HRMS (ESI-TOF): calculated for [C₂₁H₁₉INO₂ (M + H)]⁺: 444.0460, found: 444.0458.

4-(Dimethylamino)-*N*-(2-iodophenyl)-*N*-methylbenzamide (**3f**):



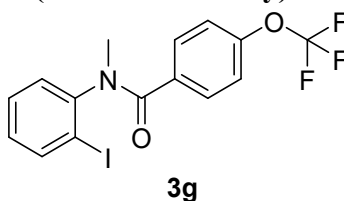
The title compound was prepared according to the general procedure at 40 °C for 10 h, obtained as a yellow solid, 96 mg, yield 42%. Melting Point: 165-166 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.84 (d, *J* = 7.8 Hz, 1H), 7.35 – 7.19 (m, 3H), 7.11 (d, *J* = 6.2 Hz, 1H), 6.91 (t, *J* = 7.2 Hz, 1H), 6.43 (d, *J* = 6.1 Hz, 2H), 3.34 (s, 3H), 2.90 (s, 6H).

¹³C NMR (101 MHz, CDCl₃): δ 170.8, 151.3, 148.1, 140.1, 130.6, 129.9, 129.4, 128.6, 122.2, 110.4, 99.2, 40.0, 38.1.

HRMS (ESI-TOF): calculated for [C₁₆H₁₈IN₂O (M + H)]⁺: 381.0464, found: 381.0461.

N-(2-iodophenyl)-*N*-methyl-4-(trifluoromethoxy)benzamide (**3g**):



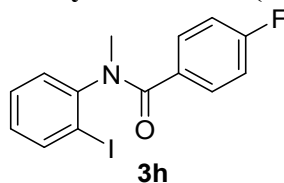
The title compound was prepared according to the general procedure for 1 h, obtained as a yellow oil, 232 mg, yield 92%.

¹H NMR (400 MHz, CDCl₃): δ 7.73 (d, *J* = 7.3 Hz, 1H), 7.33 (d, *J* = 7.6 Hz, 2H), 7.17 (t, *J* = 8.4 Hz, 1H), 7.02 (d, *J* = 7.2 Hz, 1H), 6.91 (d, *J* = 7.5 Hz, 2H), 6.86 (t, *J* = 7.2 Hz, 1H), 3.30 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 169.2, 149.8, 146.4, 140.2, 134.1, 130.1, 129.9, 129.5, 129.3, 120.1 (q, *J* = 258.6 Hz, 124.0, 121.4, 118.8, 116.3), 119.6, 98.9, 37.5. **¹⁹F NMR (377 MHz, CDCl₃)** δ -57.8.

HRMS (ESI-TOF): calculated for [C₁₅H₁₂F₃INO₂ (M + H)]⁺: 421.9865, found: 421.9862.

4-Fluoro-*N*-(2-iodophenyl)-*N*-methylbenzamide (**3h**):



The title compound was prepared according to the general procedure for 2 h, obtained as a yellow solid, 192 mg, yield 90%. Melting Point: 80-81 °C.

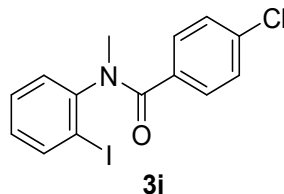
¹H NMR (400 MHz, CDCl₃): δ 7.80 (d, *J* = 7.7 Hz, 1H), 7.37 (t, *J* = 8.0 Hz, 2H), 7.24 (t, *J* = 7.3 Hz, 1H), 7.10 (d, *J* = 7.5 Hz, 1H), 6.92 (t, *J* = 7.3 Hz, 1H), 6.83 (t, *J* = 8.2 Hz, 2H), 3.37 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 169.7, 163.3 (d, *J* = 250.5 Hz, 164.6, 162.1), 146.9, 140.3, 131.8 (d, *J* = 3.2 Hz, 131.8, 131.7), 130.8 (d, *J* = 8.5 Hz, 130.8, 130.7), 130.0, 129.5, 129.2, 114.7 (d, *J* = 21.8 Hz, 114.8, 114.6), 99.0, 37.7. **¹⁹F NMR (377 MHz, CDCl₃)** δ -109.8.

HRMS (ESI-TOF): calculated for [C₁₄H₁₂FINO (M + H)]⁺: 355.9948, found: 355.9946.

The ¹H NMR and ¹³C NMR of **3h** are consistent with the reported spectra^[18].

4-Chloro-*N*-(2-iodophenyl)-*N*-methylbenzamide (**3i**):



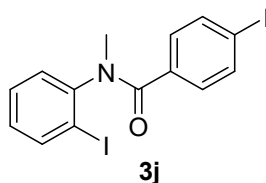
The title compound was prepared according to the general procedure for 1 h, obtained as a yellowish solid, 209 mg, yield 94%. Melting Point: 90-91 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.30 (d, *J* = 7.7 Hz, 2H), 7.24 (d, *J* = 7.5 Hz, 1H), 7.12 (m, 3H), 6.93 (t, *J* = 7.2 Hz, 1H), 3.37 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 169.5, 146.6, 140.3, 135.9, 134.1, 130.0, 129.9, 129.5, 129.3, 127.9, 99.0, 37.6.

HRMS (ESI-TOF): calculated for [C₁₄H₁₂ClINO (M + H)]⁺: 371.9652, found: 371.9647.

4-Iodo-*N*-(2-iodophenyl)-*N*-methylbenzamide (**3j**):



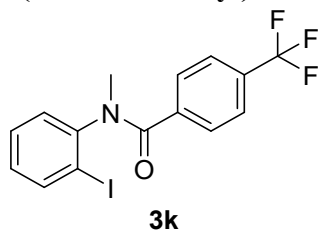
The title compound was prepared according to the general procedure for 2 h, obtained as a yellow solid, 241 mg, yield 87%. Melting Point: 120-121 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.50 (d, *J* = 7.5 Hz, 2H), 7.23 (d, *J* = 7.4 Hz, 1H), 7.13-7.06 (m, 3H), 6.93 (t, *J* = 7.2 Hz, 1H), 3.36 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 169.7, 146.5, 140.2, 136.8, 135.1, 130.0, 129.9, 129.5, 129.3, 99.0, 96.6, 37.6.

HRMS (ESI-TOF): calculated for [C₁₄H₁₂I₂NO (M + H)]⁺: 463.9008, found: 463.9003.

***N*-(2-iodophenyl)-*N*-methyl-4-(trifluoromethyl)benzamide (3k):**



The title compound was prepared according to the general procedure for 10 h, obtained as a yellow oil, 221 mg, yield 91%.

¹H NMR (400 MHz, CDCl₃): δ 7.81 (d, *J* = 7.9 Hz, 1H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 3.40 (s, 3H).

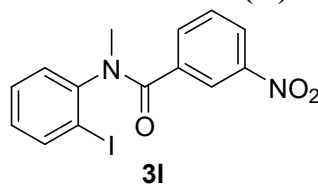
¹³C NMR (101 MHz, CDCl₃): δ 169.4, 146.3, 140.4, 139.2, 131.5 (d, *J* = 32.6 Hz, 131.7, 131.4), 130.1, 129.6 (d, *J* = 3.9 Hz, 129.6, 129.6), 128.7, 124.7 (q, *J* = 3.5 Hz, 124.8, 124.8, 124.7, 124.7), 123.7 (q, *J* = 273.6 Hz, 127.7, 125.0, 122.3, 119.6), 99.0, 37.6.

¹⁹F NMR (377 MHz, CDCl₃): δ -64.1.

HRMS (ESI-TOF): calculated for [C₁₅H₁₂F₃INO (M + H)]⁺: 405.9916, found: 405.9912.

The ¹H NMR and ¹³C NMR of **3k** are consistent with the reported spectra^[18].

***N*-(2-iodophenyl)-*N*-methyl-3-nitrobenzamide (3l):**



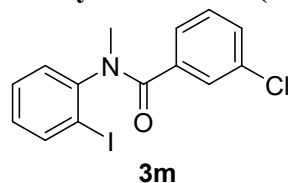
The title compound was prepared according to the general procedure for 6 h, obtained as a white solid, 76 mg, yield 33%. Melting Point: 149-150 °C.

¹H NMR (400 MHz, CDCl₃): δ 8.23 (s, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.80 (d, *J* = 7.9 Hz, 1H), 7.74 (d, *J* = 7.7 Hz, 1H), 7.37 (t, *J* = 8.0 Hz, 1H), 7.29 (t, *J* = 7.9 Hz, 1H), 7.22 (dd, *J* = 7.8, 1.3 Hz, 1H), 6.95 (t, *J* = 7.6 Hz, 1H), 3.41 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 168.1, 147.4, 146.0, 140.5, 137.3, 134.3, 130.0, 129.9, 129.8, 128.9, 124.6, 123.7, 99.1, 37.6.

HRMS (ESI-TOF): calculated for [C₁₄H₁₂IN₂O₃ (M + H)]⁺: 382.9893, found: 382.9892.

3-Chloro-*N*-(2-iodophenyl)-*N*-methylbenzamide (3m):



The title compound was prepared according to the general procedure for 2 h, obtained as a white solid, 187 mg, yield 84%. Melting Point: 123-124 °C.

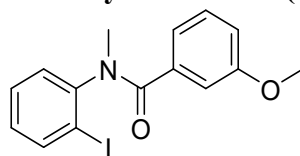
¹H NMR (400 MHz, CDCl₃): δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.42 (s, 1H), 7.24 (d, *J* = 7.4 Hz, 1H), 7.19 (d, *J* = 7.4 Hz, 2H), 7.12 (d, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 7.5 Hz, 1H), 6.93 (t, *J* = 7.2 Hz, 1H), 3.37 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 169.2, 146.5, 140.3, 137.4, 133.8, 130.1, 130.0, 129.6, 129.5, 128.9, 128.7, 126.3, 99.0, 37.6.

HRMS (ESI-TOF): calculated for [C₁₄H₁₂ClINO (M + H)]⁺: 371.9652, found: 371.9651.

The ¹H NMR and ¹³C NMR of **3m** are consistent with the reported spectra^[20].

***N*-(2-iodophenyl)-3-methoxy-*N*-methylbenzamide (**3n**):**



3n

The title compound was prepared according to the general procedure for 3 h, obtained as a white solid, 214 mg, yield 97%. Melting Point: 105-106 °C.

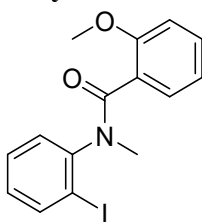
¹H NMR (400 MHz, CDCl₃): δ 7.80 (t, *J* = 7.1 Hz, 1H), 7.18 (d, *J* = 6.3 Hz, 1H), 7.09 – 6.99 (m, 2H), 6.97 – 6.83 (m, 3H), 6.74 (m, 1H), 3.65 (d, *J* = 5.5 Hz, 3H), 3.36 (d, *J* = 3.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.6, 158.8, 147.0, 140.1, 136.8, 130.1, 129.4, 129.1, 128.8, 120.8, 116.5, 113.0, 99.0, 55.3, 37.6.

HRMS (ESI-TOF): calculated for [C₁₅H₁₅INO₂ (M + H)]⁺: 368.0147, found: 368.0141.

The ¹H NMR of **3n** are consistent with the reported spectra^[21].

***N*-(2-iodophenyl)-2-methoxy-*N*-methylbenzamide (**3o**):**



3o

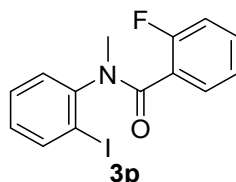
The title compound was prepared according to the general procedure for 10 h, obtained as a yellow solid, 207 mg, yield 94% (a mixture of rotamers). Melting Point: 77-78 °C.

¹H NMR (400 MHz, CDCl₃): major isomer: δ 7.72 (d, *J* = 7.8 Hz, 1H), 7.37 (d, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 8.3 Hz, 1H), 7.13 (t, *J* = 7.5 Hz, 2H), 6.83 (t, *J* = 7.5 Hz, 1H), 6.75 (t, *J* = 7.4 Hz, 1H), 6.66 (d, *J* = 8.3 Hz, 1H), 3.79 (s, 3H), 3.38 (s, 3H); minor isomer: δ 7.94 (d, *J* = 7.9 Hz, 1H), 7.54 – 7.40 (m, 5H), 7.06 (t, *J* = 6.9 Hz, 1H), 6.97 (d, *J* = 8.3 Hz, 1H), 3.92 (s, 3H), 3.11 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 169.1, 169.0 (minor), 155.6 (minor), 155.1, 145.9, 145.8 (minor), 139.9 (minor), 139.6, 130.7 (minor), 130.3, 129.8 (minor), 129.3, 129.2, 129.0 (minor), 128.6, 128.1 (minor), 127.5 126.4 126.1 (minor), 121.0 (minor), 120.0, 111.1 (minor), 110.4, 99.1, 55.7 (minor), 55.2, 38.9 (minor), 36.4.

HRMS (ESI-TOF): calculated for [C₁₅H₁₅INO₂ (M + H)]⁺: 368.0147, found: 368.0144.

2-Fluoro-*N*-(2-iodophenyl)-*N*-methylbenzamide (3p**):**



The title compound was prepared according to the general procedure for 10 h, obtained as a yellow solid, 187 mg, yield 88% (a mixture of rotamers). Melting Point: 129-130 °C.

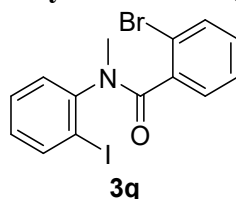
¹H NMR (400 MHz, CDCl₃): major isomer: δ 7.73 (d, *J* = 7.9 Hz, 1H), 7.44 (t, *J* = 7.1 Hz, 1H), 7.31 – 7.13 (m, 3H), 6.95 (t, *J* = 7.5 Hz, 1H), 6.92 – 6.80 (m, 2H), 3.39 (s, 3H); minor isomer: δ 7.95 (d, *J* = 7.8 Hz, 1H), 7.60 (t, *J* = 7.0 Hz, 1H), 7.44 (m, 1H), 7.39 (d, *J* = 7.5 Hz, 1H), 6.92 – 6.80 (m, 3H), 7.09 (t, *J* = 7.3 Hz, 1H), 3.20 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 166.6, 166.4 (minor), 159.3, 157.2 (minor), 156.9, 145.4, 145.3 (minor), 140.1 (minor), 139.8, 131.6 (d, *J* = 8.2 Hz, 131.6, 131.6) (minor), 131.1 (d, *J* = 8.2 Hz, 131.2, 131.1), 129.9 (minor), 129.7, 129.6, 129.2, 128.8 (minor), 128.6 (d, *J* = 3.3 Hz, 128.6, 128.5), 125.0 (d, *J* = 17.2 Hz, 125.1, 124.9) (minor), 124.7 (d, *J* = 3.2 Hz, 124.8, 124.7) (minor), 123.6 (d, *J* = 3.5 Hz, 123.7, 123.6), 115.9 (d, *J* = 21.6 Hz, 116.0, 115.8) (minor), 115.5 (d, *J* = 21.6 Hz, 115.5, 115.3), 99.1, 97.9 (minor), 39.2 (minor), 36.7.

¹⁹F NMR (377 MHz, CDCl₃): δ -112.4.

HRMS (ESI-TOF): calculated for [C₁₄H₁₂FINO (M + H)]⁺: 355.9948, found: 355.9947.

2-Bromo-*N*-(2-iodophenyl)-*N*-methylbenzamide (3q):



The title compound was prepared according to the general procedure for 2 h, obtained as a white solid, 241 mg, yield 97% (a mixture of rotamers). Melting Point: 105-106 °C.

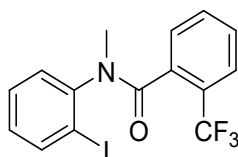
¹H NMR (400 MHz, CDCl₃): major isomer: δ 7.76 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.50 – 7.34 (m, 3H), 7.16 (ddd, *J* = 7.8, 2.8, 1.4 Hz, 1H), 7.11 – 6.97 (m, 2H), 6.86 (ddd, *J* = 7.8, 2.8, 1.4 Hz, 1H), 3.41 (d, *J* = 0.9 Hz, 3H); minor isomer: δ 7.93 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.54 (d, *J* = 5.6 Hz, 1H), 7.50 – 7.34 (m, 2H), 7.29 (m, 1H), 7.11 – 6.97 (m, 2H), 3.11 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 168.6, 145.6, 145.0 (minor), 140.0, 139.9 (minor), 138.2 (minor), 138.1, 132.8 (minor), 132.7, 130.6 (minor), 130.2, 130.0 (minor), 129.7, 129.5, 129.2, 128.8 (minor), 128.0 (minor), 127.9 (minor), 127.2, 126.8, 120.0, 119.2 (minor), 98.8, 39.2 (minor), 36.6.

HRMS (ESI-TOF): calculated for [C₁₄H₁₂BrINO (M + H)]⁺: 415.9147, found: 415.9151.

The ¹H NMR and ¹³C NMR of **3q** are consistent with the reported spectra^[19].

N-(2-iodophenyl)-*N*-methyl-2-(trifluoromethyl)benzamide (3r):



3r

The title compound was prepared according to the general procedure for 2 h, obtained as a white solid, 216 mg, yield 89% (a mixture of rotamers). Melting Point: 141-142 °C.

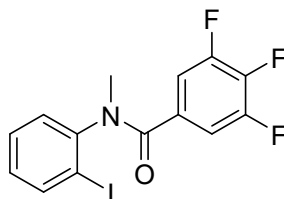
¹H NMR (400 MHz, CDCl₃): major isomer: δ 7.81 (d, *J* = 7.8 Hz, 1H), 7.75 (t, *J* = 7.3 Hz, 1H), 7.57 (d, *J* = 7.2 Hz, 1H), 7.44 (d, *J* = 7.2 Hz, 1H), 7.34 – 7.27 (m, 1H), 7.16 – 7.06 (m, 2H), 6.86 (t, *J* = 7.3 Hz, 1H), 3.42 (s, 3H); minor isomer: δ 7.95 (d, *J* = 7.7 Hz, 1H), 7.69 (t, *J* = 7.2 Hz, 1H), 7.60 – 7.55 (m, 1H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.37 (d, *J* = 7.7 Hz, 1H), 7.34 – 7.27 (m, 2H), 7.16 – 7.06 (m, 1H), 3.06 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 168.1, 168.0 (minor), 145.7, 144.6 (minor), 140.2, 139.9 (minor), 135.0 (d, *J* = 2.1 Hz, 135.1, 135.0) (minor), 134.5 (d, *J* = 2.1 Hz, 134.5, 134.5), 132.3 (minor), 131.3, 130.1 (minor), 129.8, 129.5 (d, *J* = 6.8 Hz, 129.6, 129.5), 129.4, 129.2, 128.6 (minor), 128.1 (minor), 127.8 (minor), 127.5, 127.1 (minor), 127.0, 126.7 (q, *J* = 4.5 Hz, 126.8, 126.7, 126.7, 126.6), 123.9 (q, *J* = 274.7 Hz, 128.0, 125.2, 122.5, 119.8), 99.0, 98.1 (minor), 39.6 (minor), 36.9.

¹⁹F NMR (377 MHz, CDCl₃): δ -58.5, -59.9.

HRMS (ESI-TOF): calculated for [C₁₅H₁₂F₃INO (M + H)]⁺: 405.9916, found: 405.9915.

3,4,5-Trifluoro-*N*-(2-iodophenyl)-*N*-methylbenzamide (3s):



3s

The title compound was prepared according to the general procedure for 3 h, obtained as a yellow solid, 171 mg, yield 73%. Melting Point: 57-58 °C.

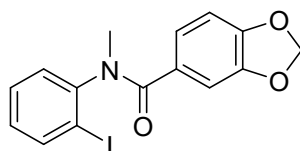
¹H NMR (400 MHz, CDCl₃): δ 7.84 (d, *J* = 7.9 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.06 – 6.95 (m, 3H), 3.36 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 167.2, 150.4 (ddd, *J* = 251.2, 10.0, 3.2 Hz, 151.7, 151.7, 151.6, 151.6, 149.2, 149.2, 149.1, 149.1), 146.1, 140.7 (dt, *J* = 30.1, 15.3 Hz, 142.2, 142.0, 141.9, 139.6, 139.5, 139.3), 140.6, 131.5 (dd, *J* = 11.9, 6.8 Hz, 131.6, 131.5, 131.5, 131.4), 129.9 (d, *J* = 208.7 Hz, 131.0, 128.9), 129.9, 129.7, 113.3 (dd, *J* = 16.6, 6.4 Hz, 113.4, 113.3, 113.2, 113.2), 98.8, 37.8.

¹⁹F NMR (377 MHz, CDCl₃): δ -133.5, -157.0.

HRMS (ESI-TOF): calculated for [C₁₄H₁₀F₃INO (M + H)]⁺: 391.9759, found: 391.9764.

N-(2-iodophenyl)-*N*-methylbenzo[d][1,3]dioxole-5-carboxamide (3t):



3t

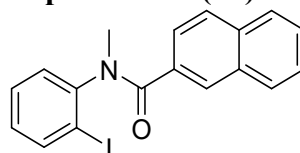
The title compound was prepared according to the general procedure for 3 h, obtained as a white solid, 199 mg, yield 87%. Melting Point: 99-100 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.82 (d, *J* = 6.8 Hz, 1H), 7.25 (s, 1H), 7.10 (d, *J* = 6.2 Hz, 1H), 6.91 (m, 2H), 6.86 (d, *J* = 7.1 Hz, 1H), 6.55 (d, *J* = 6.1 Hz, 1H), 5.88 (s, 2H), 3.34 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.0, 148.9, 147.2, 146.9, 140.2, 129.9, 129.5, 129.4, 129.0, 123.5, 109.2, 107.4, 101.3, 98.9, 37.9.

HRMS (ESI-TOF): calculated for [C₁₅H₁₃INO₃ (M + H)]⁺: 381.9940, found: 381.9937.

***N*-(2-iodophenyl)-*N*-methyl-2-naphthamide (3u):**



3u

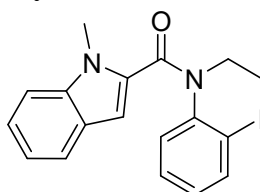
The title compound was prepared according to the general procedure for 3 h, obtained as colorless crystals, 211 mg, yield 91%. Melting Point: 118-120 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.90 (s, 1H), 7.75 (s, 1H), 7.69 (d, *J* = 6.8 Hz, 2H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.47 – 7.38 (m, 3H), 7.14 (s, 2H), 6.82 (s, 1H), 3.42 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.8, 147.0, 140.2, 133.7, 133.0, 132.2, 130.1, 129.4, 129.1, 128.9, 128.7, 127.6, 127.3, 127.1, 126.3, 125.3, 99.1, 37.7.

HRMS (ESI-TOF): calculated for [C₁₈H₁₅INO (M + H)]⁺: 388.0198, found: 388.0199. The ¹H NMR and ¹³C NMR of **3u** are consistent with the reported spectra^[18, 19].

***N*-ethyl-*N*-(2-iodophenyl)-1-methyl-1*H*-indole-2-carboxamide (3v):**



3v

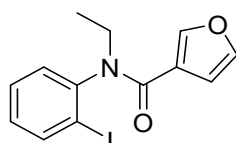
The title compound was prepared according to the general procedure for 12 h, obtained as a brown solid, 225 mg, yield 93%. Melting Point: 135-137 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.77 (d, *J* = 6.6 Hz, 1H), 7.36 (m, 2H), 7.31 (m, 2H), 7.22 (d, *J* = 7.5 Hz, 1H), 7.04 – 6.93 (m, 2H), 5.93 (s, 1H), 4.18 (m, 1H), 4.01 (s, 3H), 3.70 (m, 1H), 1.29 (t, *J* = 7.0 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 163.0, 145.4, 140.3, 138.0, 132.0, 130.2, 129.3, 129.3, 126.1, 123.6, 121.9, 119.9, 109.9, 107.2, 100.7, 44.8, 32.0, 12.9.

HRMS (ESI-TOF): calculated for [C₁₈H₁₈IN₂O (M + H)]⁺: 405.0464, found: 405.0469.

***N*-ethyl-*N*-(2-iodophenyl)furan-3-carboxamide (3w):**



3w

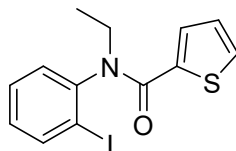
The title compound was prepared according to the general procedure for 4 h, obtained as a brown solid, 115 mg, yield 56%. Melting Point: 63-65 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.96 (d, *J* = 7.9 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 1H), 7.14 (m, 2H), 6.75 (s, 1H), 6.21 (s, 1H), 4.32 (m, 1H), 3.35 (m, 1H), 1.23 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 162.6, 145.2, 144.5, 142.2, 140.5, 131.1, 130.2, 129.5, 122.2, 111.1, 101.1, 44.4, 12.7.

HRMS (ESI-TOF): calculated for [C₁₃H₁₃INO₂ (M + H)]⁺: 341.9991, found: 341.9985.

***N*-ethyl-*N*-(2-iodophenyl)thiophene-2-carboxamide (3x):**



3x

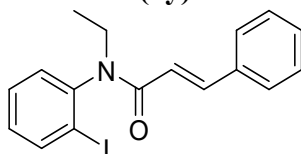
The title compound was prepared according to the general procedure for 4 h, obtained as a yellow solid, 201 mg, yield 94%. Melting Point: 70-72 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.97 (d, *J* = 7.9 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.30 (s, 2H), 7.14 (t, *J* = 7.6 Hz, 1H), 6.81 (m, 2H), 4.35 (m, 1H), 3.37 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 161.8, 144.4, 140.5, 138.1, 132.1, 131.4, 130.9, 130.2, 129.6, 126.8, 101.4, 45.1, 12.6.

HRMS (ESI-TOF): calculated for [C₁₃H₁₃INOS (M + H)]⁺: 357.9763, found: 357.9773.

***N*-ethyl-*N*-(2-iodophenyl)cinnamamide (3y):**



3y

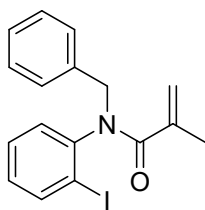
The title compound was prepared according to the general procedure for 10 h, obtained as a yellow oil, 161 mg, yield 71%.

¹H NMR (400 MHz, CDCl₃): δ 8.00 (d, *J* = 7.8 Hz, 1H), 7.71 (d, *J* = 15.5 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 1H), 7.28 (m, 6H), 7.13 (t, *J* = 7.5 Hz, 1H), 6.05 (d, *J* = 15.5 Hz, 1H), 4.27 (m, 1H), 3.36 (m, 1H), 1.21 (t, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 165.4, 144.0, 142.4, 140.4, 135.2, 130.9, 129.9, 129.6, 129.5, 128.7, 127.9, 118.6, 101.0, 43.6, 13.0.

HRMS (ESI-TOF): calculated for [C₁₇H₁₇INO (M + H)]⁺: 378.0355, found: 378.0349.

***N*-benzyl-*N*-(2-iodophenyl)methacrylamide (3z):**



3z

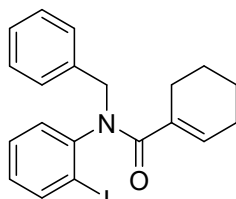
The title compound was prepared according to the general procedure for 4 h, obtained as a yellow oil, 176 mg, yield 78%.

¹H NMR (400 MHz, CDCl₃): δ 7.88 (d, *J* = 7.8 Hz, 1H), 7.23 (m, 5H), 7.14 (t, *J* = 7.4 Hz, 1H), 6.96 (t, *J* = 7.5 Hz, 1H), 6.67 (d, *J* = 7.3 Hz, 1H), 5.69 (d, *J* = 14.2 Hz, 1H), 5.01 (d, *J* = 27.0 Hz, 2H), 4.11 (d, *J* = 14.2 Hz, 1H), 1.85 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 171.4, 144.5, 140.2, 140.2, 136.8, 131.3, 129.4, 129.3, 128.7, 128.4, 127.6, 118.8, 100.0, 51.8, 20.8.

HRMS (ESI-TOF): calculated for [C₁₇H₁₇INO (M + H)]⁺: 378.0355, found: 378.0350. The ¹H NMR and ¹³C NMR of **3z** are consistent with the reported spectra^[22].

***N*-benzyl-*N*-(2-iodophenyl)cyclohex-1-ene-1-carboxamide (**3aa**):**



3aa

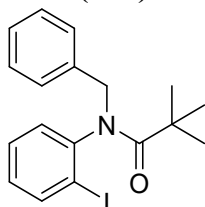
The title compound was prepared according to the general procedure at 40 °C for 1 h, obtained as a yellowish solid, 200 mg, yield 80%. Melting Point: 120-123 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.86 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.26 – 7.16 (m, 5H), 7.13 (td, *J* = 7.8, 1.5 Hz, 1H), 6.92 (td, *J* = 7.8, 1.5 Hz, 1H), 6.68 (d, *J* = 7.7 Hz, 1H), 5.85 (m, 1H), 5.64 (d, *J* = 13.8 Hz, 1H), 4.14 (d, *J* = 14.1 Hz, 1H), 2.12 (t, *J* = 17.8 Hz, 2H), 1.82 (m, 2H), 1.41 (m, 4H).

¹³C NMR (101 MHz, CDCl₃): δ 172.0, 144.9, 140.0, 137.1, 134.2, 132.0, 131.3, 129.3, 129.0, 128.5, 128.3, 127.4, 100.1, 51.9, 26.2, 24.9, 22.1, 21.4.

HRMS (ESI-TOF): calculated for [C₂₀H₂₁INO (M + H)]⁺: 418.0668, found: 418.0678.

***N*-benzyl-*N*-(2-iodophenyl)pivalamide (**3ab**):**



3ab

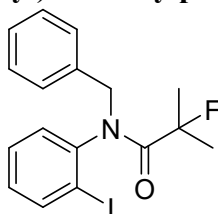
The title compound was prepared according to the general procedure for 1 h, obtained as a yellow solid, 116 mg, yield 49%. Melting Point: 80-82 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.92 (d, *J* = 7.8 Hz, 1H), 7.23 (m, 3H), 7.15 (m, 3H), 6.99 (t, *J* = 7.5 Hz, 1H), 6.72 (d, *J* = 7.7 Hz, 1H), 5.83 (d, *J* = 14.3 Hz, 1H), 3.75 (d, *J* = 14.2 Hz, 1H), 1.06 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 177.3, 145.0, 140.2, 137.4, 131.9, 129.6, 129.3, 128.3, 128.3, 127.4, 102.1, 54.4, 41.2, 29.2.

HRMS (ESI-TOF): calculated for $[C_{18}H_{21}INO (M + H)]^+$: 394.0668, found: 394.0662. The 1H NMR and ^{13}C NMR of **3ab** are consistent with the reported spectra^[23].

***N*-benzyl-2-fluoro-*N*-(2-iodophenyl)-2-methylpropanamide (**3ac**):**



3ac

The title compound was prepared according to the general procedure for 6 h, obtained as a yellow solid, 148 mg, yield 62%. Melting Point: 79-81 °C.

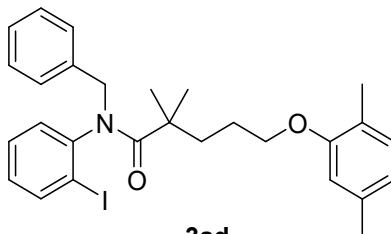
1H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, $J = 6.5$ Hz, 1H), 7.30 – 7.03 (m, 6H), 6.96 (d, $J = 5.4$ Hz, 1H), 6.62 (d, $J = 6.4$ Hz, 1H), 5.76 (d, $J = 13.9$ Hz, 1H), 3.83 (d, $J = 13.9$ Hz, 1H), 1.68 (d, $J = 21.4$ Hz, 3H), 1.55 (d, $J = 21.0$ Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 172.1 (d, $J = 20.2$ Hz, 172.2, 172.0), 144.6, 144.6, 139.6, 136.7, 130.0 (d, $J = 4.7$ Hz, 130.0, 129.9), 129.5, 129.1, 128.4, 128.2, 127.7, 99.8 (d, $J = 5.3$ Hz, 99.8, 99.7), 96.7 (d, $J = 188.7$ Hz, 97.7, 95.8), 54.0, 26.7 (dd, $J = 127.5, 24.2$ Hz, 27.5, 27.3, 26.2, 26.0).

^{19}F NMR (377 MHz, $CDCl_3$): δ -141.5.

HRMS (ESI-TOF): calculated for $[C_{17}H_{18}FINO (M + H)]^+$: 398.0417, found: 398.0415.

***N*-benzyl-5-(2,5-dimethylphenoxy)-*N*-(2-iodophenyl)-2,2-dimethylpentanamide (**3ad**):**



3ad

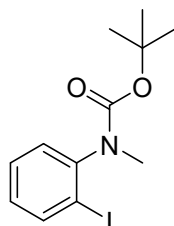
The title compound was prepared according to the general procedure at 40 °C for 6 h, obtained as a yellowish solid, 214 mg, yield 66%. Melting Point: 84-86 °C.

1H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, $J = 7.8$ Hz, 1H), 7.23 – 7.10 (m, 6H), 6.99 (d, $J = 6.1$ Hz, 2H), 6.71 (d, $J = 7.7$ Hz, 1H), 6.65 (d, $J = 7.3$ Hz, 1H), 6.61 (s, 1H), 5.85 (d, $J = 14.1$ Hz, 1H), 3.90 (d, $J = 5.8$ Hz, 2H), 3.75 (d, $J = 14.1$ Hz, 1H), 2.30 (s, 3H), 2.14 (s, 3H), 1.94 – 1.71 (m, 3H), 1.47 (t, $J = 11.3$ Hz, 1H), 1.02 (s, 3H), 0.93 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 176.1, 157.1, 144.9, 140.2, 137.4, 136.5, 131.9, 130.3, 129.6, 128.3, 128.3, 127.5, 123.6, 120.7, 112.2, 102.2, 68.3, 54.5, 44.5, 40.1, 27.4, 26.5, 25.4, 21.5, 15.9.

HRMS (ESI-TOF): calculated for $[C_{28}H_{33}INO_2 (M + H)]^+$: 542.1556, found: 542.1553.

***Tert*-butyl (2-iodophenyl)(methyl)carbamate (**3ba**):**



3ba

The title compound was prepared according to the general procedure for 12 h, obtained as a yellow solid, 140 mg, yield 70% (a mixture of rotamers). Melting Point: 55-56 °C. Melting Point: 51-52 °C.

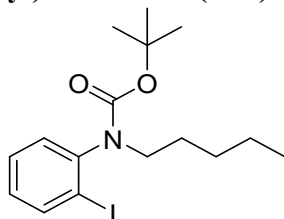
¹H NMR (400 MHz, CDCl₃): δ 7.85 (d, *J* = 7.4 Hz, 1H), 7.35 (t, *J* = 7.0 Hz, 1H), 7.21 (d, *J* = 7.5 Hz, 1H), 6.98 (t, *J* = 7.1 Hz, 1H), 3.14 (s, 3H), 1.54 (minor)/1.36 (major) (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 154.4 (minor), 154.2, 146.1 (minor), 145.9, 139.7 (minor), 139.3, 129.5 (minor), 129.2, 129.1 (minor), 128.9 (minor), 128.6, 128.5, 99.5, 80.5 (minor), 80.1, 37.4 (minor), 36.4, 28.5 (minor), 28.3.

HRMS (ESI-TOF): calculated for [C₁₂H₁₆INNaO₂ (M + Na)]⁺: 356.0123, found: 356.0127.

The ¹H NMR and ¹³C NMR of **3ba** are consistent with the reported spectra^[24].

***Tert*-butyl (2-iodophenyl)(pentyl)carbamate (**3bb**):**



3bb

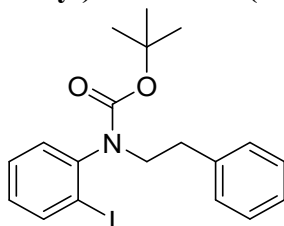
The title compound was prepared according to the general procedure at 40 °C for 15 h, obtained as a yellow oil, 142 mg, yield 61% (a mixture of rotamers).

¹H NMR (400 MHz, CDCl₃): δ 7.86 (d, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.25 – 7.10 (m, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 3.83 – 3.69 (m, 1H), 3.29 – 3.07 (m, 1H), 1.60 – 1.49 (m, 1H), 1.54 (minor)/1.34 (major) (br s, 9H), 1.32 – 1.20 (m, 4H), 0.87 (t, *J* = 6.6 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 154.4 (minor), 154.1, 145.0 (minor), 144.8, 139.8 (minor), 139.5, 130.3 (minor), 129.8, 129.1 (minor), 128.8, 128.7 (minor), 128.5, 100.6, 80.5 (minor), 80.0, 50.3 (minor), 49.4, 29.2, 28.6, 28.4 (minor), 28.3, 28.1 (minor), 22.5, 22.4 (minor), 14.1.

HRMS (ESI-TOF): calculated for [C₁₆H₂₄INNaO₂ (M + Na)]⁺: 412.0749, found: 412.0755.

***Tert*-butyl (2-iodophenyl)(phenethyl)carbamate (**3bc**):**



3bc

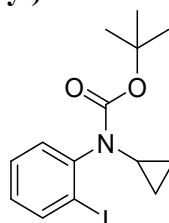
The title compound was prepared according to the general procedure at 40 °C for 15 h, obtained as a yellowish oil, 170 mg, yield 67% (a mixture of rotamers).

¹H NMR (400 MHz, CDCl₃): δ 7.88 (minor)/ 7.86 (major) (br d, *J* = 8.0 Hz 1H), 7.36 – 7.23 (br m, 3H), 7.22 – 7.14 (br m, 3H), 7.12 – 6.93 (br m, 2H), 4.13 – 3.94 (br m, 1H), 3.49 – 3.28 (br m, 1H), 2.94 (br t, *J* = 7.8 Hz, 2H), 1.55 (minor)/1.36 (major) (br s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 154.2 (minor), 153.9, 145.0 (minor), 144.9, 139.8 (minor), 139.5, 139.0, 130.3 (minor), 129.8, 129.2 (minor), 128.9, 128.9 (minor), 128.8 (minor), 128.6, 128.6, 128.4, 128.3 (minor), 126.4 (minor), 126.3, 100.5, 80.8 (minor), 80.2, 52.3 (minor), 51.2, 35.5 (minor), 34.8, 28.5 (minor), 28.3.

HRMS (ESI-TOF): calculated for [C₁₉H₂₂INNaO₂ (M + Na)]⁺: 446.0593, found: 446.0589.

***Tert*-butyl cyclopropyl(2-iodophenyl)carbamate (3bd):**



3bd

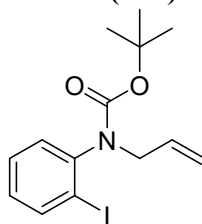
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as a yellow solid, 185 mg, yield 86% (a mixture of rotamers). Melting Point: 88-90 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.84 (d, *J* = 7.7 Hz, 1H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.08 (s, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 3.09 (br s, 1H), 1.53 (minor)/1.36 (major) (br s, 9H), 0.81 – 0.61 (br m, 3H), 0.58 – 0.44 (br m, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 154.9, 144.9, 139.2, 129.2, 128.9, 128.4, 100.8, 80.5, 30.7, 28.3, 8.0, 6.8.

HRMS (ESI-TOF): calculated for [C₁₄H₁₉INO₂ (M + H)]⁺: 360.0460, found: 360.0459.

***Tert*-butyl allyl(2-iodophenyl)carbamate (3be):**



3be

The title compound was prepared according to the general procedure at 40 °C for 8 h, obtained as a yellowish solid, 142 mg, yield 66% (a mixture of rotamers). Melting Point: 70-72 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.85 (br d, *J* = 7.2 Hz, 1H), 7.31 (t, *J* = 6.8 Hz, 1H), 7.24 – 7.08 (br m, 1H), 6.98 (br t, *J* = 6.4 Hz, 1H), 5.94 (td, *J* = 16.0, 6.4 Hz, 1H), 5.18 – 4.96 (br m, 2H), 4.57 – 4.34 (br m, 1H), 3.83 – 3.61 (br m, 1H), 1.53 (minor)/1.35 (major) (br s, 9H).

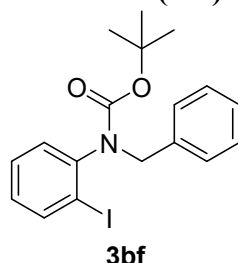
¹³C NMR (101 MHz, CDCl₃): δ 154.0 (minor), 153.9, 144.8 (minor), 144.3, 139.7 (minor), 139.4, 133.9 (minor), 133.7, 130.3 (minor), 130.0, 129.1 (minor),

129.0 (minor), 128.7, 128.7, 117.9, 117.3 (minor), 100.6, 80.8 (minor), 80.3, 53.3 (minor), 52.0, 28.5 (minor), 28.3.

HRMS (ESI-TOF): calculated for $[C_{14}H_{19}INO_2 (M + H)]^+$: 360.0460, found: 360.0459.

The 1H NMR and ^{13}C NMR of **3be** are consistent with the reported spectra^[25].

***Tert*-butyl benzyl(2-iodophenyl)carbamate (**3bf**):**



The title compound was prepared according to the general procedure at 40 °C for 15 h, obtained as a yellowish oil, 216 mg, yield 88% (a mixture of rotamers).

Scale-up: when **1bf** (1.0 g, 4.83 mmol) was employed in this reaction (40 °C for 6 h), 1.76 g of **3bf** was obtained, 89% yield.

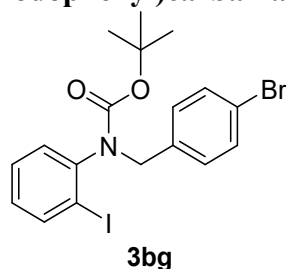
1H NMR (400 MHz, $CDCl_3$): δ 7.87 (minor)/ 7.84 (major) (br d, $J = 7.6$ Hz 1H), 7.34–7.12 (br m, 6 H), 6.99–6.71 (br m, 2 H), 5.27 (major)/5.16 (minor) (br d, $J = 14.8$ Hz), 4.14 (minor)/4.10 (major) (br d, $J = 14.8$ Hz), 1.55 (minor)/1.37 (major) (br s, 9 H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 154.4, 154.2 (minor), 144.3 (minor), 144.2, 139.8 (minor), 139.4, 138.1 (minor), 137.8, 130.7 (minor), 130.3, 129.1, 129.1 (minor), 128.7, 128.6, 128.4, 127.5, 100.3, 81.2 (minor), 80.4, 54.1 (minor), 52.7, 28.6 (minor), 28.3.

HRMS (ESI-TOF): calculated for $[C_{18}H_{20}INNaO_2 (M + Na)]^+$: 432.0436, found: 432.0436.

The 1H NMR and ^{13}C NMR of **3bf** are consistent with the reported spectra^[26].

***Tert*-butyl (4-bromobenzyl)(2-iodophenyl)carbamate (**3bg**):**



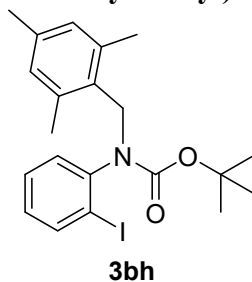
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as a yellow oil, 261 mg, yield 89% (a mixture of rotamers).

1H NMR (400 MHz, $CDCl_3$): δ 7.88 (minor)/ 7.84 (major) (br d, $J = 7.6$ Hz 1H), 7.46 – 7.30 (br m, 2H), 7.24 – 7.07 (br m, 3H), 6.94 (br t, $J = 6.3$ Hz, 1H), 6.75 (br d, $J = 7.1$ Hz, 1H), 5.17 (major)/ 5.08 (minor) (br d, $J = 14.8$ Hz 1H), 4.08 (br d, $J = 14.8$ Hz 1H), 1.54 (minor)/1.36 (major) (br s, 9 H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 154.2, 153.9 (minor), 144.1 (minor), 144.0, 139.9 (minor), 139.5, 137.0 (minor), 136.8, 131.5, 130.8, 130.5 (minor), 130.1, 129.1 (minor), 128.8, 128.7, 121.5, 100.2, 81.3 (minor), 80.6, 53.4 (minor), 52.1, 28.5 (minor), 28.3.

HRMS (ESI-TOF): calculated for $[C_{18}H_{19}BrINNaO_2 (M + Na)]^+$: 509.9542, found: 509.9539.

***Tert*-butyl (2-iodophenyl)(2,4,6-trimethylbenzyl)carbamate (3bh):**



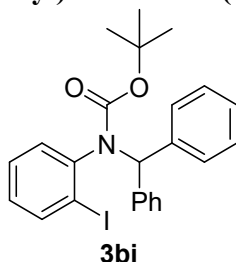
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as a yellowish solid, 225 mg, yield 83% (a mixture of rotamers). Melting Point: 134-136 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.82 (minor)/ 7.79 (major) (br d, *J* = 7.6 Hz 1H), 6.99 (br t, *J* = 7.1 Hz, 1H), 6.87 (br t, *J* = 7.2 Hz, 1H), 6.71 (br s, 2H), 6.29 (br d, *J* = 7.6 Hz, 1H), 5.24 (major)/ 5.12 (minor) (br d, *J* = 14.4 Hz 1H), 4.47 (minor)/4.42 (major) (br d, *J* = 14.4 Hz 1H), 2.21 (minor)/2.02 (major) (br s, 9H), 1.60 (minor)/1.36 (major) (br s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 154.0 (minor), 153.9, 142.6, 139.3 (minor), 138.9, 138.4, 138.2 (minor), 137.1, 131.0, 130.2, 128.9, 128.6, 128.3, 100.7, 80.9 (minor), 80.1, 45.9 (minor), 44.4, 28.6 (minor), 28.3, 21.0, 19.6.

HRMS (ESI-TOF): calculated for [C₂₁H₂₆INNaO₂ (M + Na)]⁺: 474.0906, found: 474.0910.

***Tert*-butyl benzhydryl(2-iodophenyl)carbamate (3bi):**



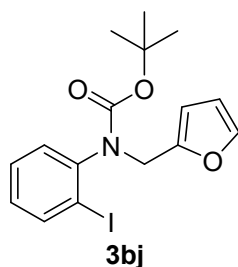
The title compound was prepared according to the general procedure for 10 h, obtained as a yellowish solid, 175 mg, yield 60% (a mixture of rotamers). Melting Point: 93-95 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.62 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.40 – 7.24 (m, 5H), 7.19 (d, *J* = 7.8 Hz, 1H), 7.15 – 7.03 (m, 6H), 6.77 (td, *J* = 7.7, 1.6 Hz, 1H), 6.63 (s, 1H), 1.38 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 154.0, 143.0, 142.1 (minor), 141.8, 139.4, 137.7, 131.3, 130.4 (minor), 128.6 (minor), 128.3, 128.2, 127.6, 127.4 (minor), 127.3 (minor), 126.8, 103.9, 80.9, 66.5, 28.4 (minor), 28.3.

HRMS (ESI-TOF): calculated for [C₂₄H₂₄INNaO₂ (M + Na)]⁺: 508.0749, found: 508.0754.

***Tert*-butyl (furan-2-ylmethyl)(2-iodophenyl)carbamate (3bj):**



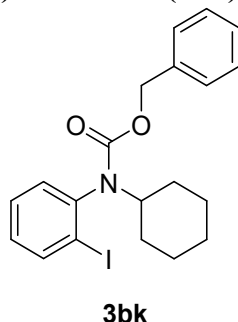
The title compound was prepared according to the general procedure at 50 °C for 10 h, obtained as a yellow oil, 74 mg, yield 31% (a mixture of rotamers).

¹H NMR (400 MHz, CDCl₃): δ 7.87 (minor)/ 7.84 (major) (br d, *J* = 7.6 Hz 1H), 7.35 (br s, 1H), 7.31–7.19 (br m, 1H), 7.11–6.85 (br m, 2H), 6.30 (minor)/ 6.26 (major) (br s, 1H), 6.13 (br s, 1H), 5.12 (major)/5.02 (minor) (br d, *J* = 15.6 Hz), 4.26 (major)/4.22 (minor) (br d, *J* = 16.0 Hz), 1.55 (minor)/1.36 (major) (br s, 9 H).

¹³C NMR (101 MHz, CDCl₃): δ 153.9, 151.4 (major), 151.0, 144.4 (major), 143.9, 142.2, 142.1 (major), 139.6 (major), 139.3, 130.5 (major), 130.0, 129.2 (major), 128.8, 128.8, 110.4, 109.1, 108.5 (major), 100.3, 81.2 (major), 80.6, 46.7 (major), 45.1, 28.5 (major), 28.3.

HRMS (ESI-TOF): calculated for [C₁₆H₁₈INO₃ (M + Na)]⁺: 422.0229, found: 422.0226.

Benzyl cyclohexyl(2-iodophenyl)carbamate (3bk):



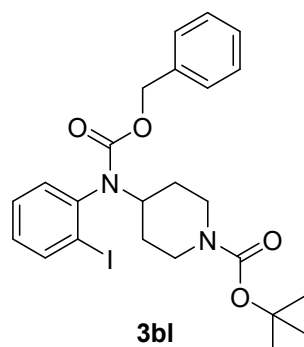
The title compound was prepared according to the general procedure at 40 °C for 10 h, obtained as a yellowish solid, 170 mg, yield 65% (a mixture of rotamers). Melting Point: 115-118 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.88 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.34 (td, *J* = 7.7, 1.3 Hz, 2H), 7.27 – 7.08 (m, 5H), 7.00 (td, *J* = 7.7, 1.6 Hz, 1H), 5.09 (s, 2H), 4.13 (s, 1H), 2.20 (d, *J* = 7.3 Hz, 1H), 1.98 (d, *J* = 12.0 Hz, 1H), 1.82 – 1.74 (m, 1H), 1.73 – 1.65 (m, 1H), 1.60 (d, *J* = 15.8 Hz, 1H), 1.49 – 1.29 (m, 3H), 1.06 – 0.82 (m, 2H).

¹³C NMR (101 MHz, CDCl₃): δ 154.4, 142.2, 139.7, 136.7, 130.1, 129.0, 128.8, 128.3, 127.7, 127.6, 103.5, 67.2, 58.5, 32.9 (isomer) / 30.5, 26.0 / 25.9 (isomer), 25.6.

HRMS (ESI-TOF): calculated for [C₂₀H₂₃INO₂ (M + H)]⁺: 436.0773, found: 436.0767.

***Tert*-butyl 4-(((benzyloxy)carbonyl) (2-iodophenyl)amino)piperidine-1-carboxylate (3bl):**



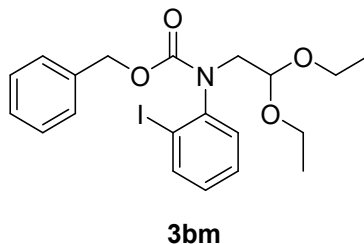
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as a yellow solid, 145 mg, yield 45% (a mixture of rotamers). Melting Point: 105-107 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.89 (d, *J* = 7.7 Hz, 1H), 7.35 (t, *J* = 6.8 Hz, 2H), 7.26 – 7.09 (m, 5H), 7.03 (t, *J* = 7.4 Hz, 1H), 5.10 (s, 2H), 4.30 (m, 1H), 4.13 (m, 2H), 2.76 (m, 2H), 2.14 (m, 1H), 1.93 (m, 1H), 1.58 (m, 1H), 1.41 (s, 9H), 1.27 (m, 1H), 1.10 (m, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 154.5, 154.4, 141.5, 139.8, 136.4, 130.1, 129.3, 129.0, 128.2, 127.7, 127.6, 103.3, 79.6, 67.3, 56.5, 43.2, 31.6 / 29.6 (isomer), 28.4.

HRMS (ESI-TOF): calculated for [C₂₄H₂₉I_N₂NaO₄ (M + Na)]⁺: 559.1070, found: 559.1066.

Benzyl (2,2-diethoxyethyl)(2-iodophenyl)carbamate (3bm):



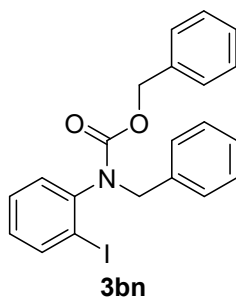
The title compound was prepared according to the general procedure at 40 °C for 10 h, obtained as a yellowish oil, 155 mg, yield 55% (a mixture of rotamers).

¹H NMR (400 MHz, CDCl₃): δ 7.91 – 7.82 (m, 1H), 7.46 – 7.14 (m, 7H), 6.99 (ddd, *J* = 8.9, 6.5, 2.6 Hz, 1H), 5.32 – 5.03 (m, 2H), 4.87 – 4.65 (m, 1H), 4.16 – 4.00 (m, 1H), 3.73 – 3.38 (m, 4H), 3.33 – 3.16 (m, 1H), 1.21 – 1.09 (m, 6H).

¹³C NMR (101 MHz, CDCl₃): δ 155.1, 154.9 (minor), 144.6 (minor), 144.3, 139.6 (minor), 139.3, 136.4, 136.4 (minor), 130.9 (minor), 130.6, 129.2 (minor), 129.1, 129.0 (minor), 128.8, 128.5 (minor), 128.4 (minor), 128.2, 128.2 (minor), 127.7, 127.4 (minor), 127.4, 126.6 (minor), 100.8 – 99.3 (m, 100.6, 100.1, 99.8, 99.5), 67.8 (minor), 67.4, 62.3, 61.8, 52.1 (minor), 52.0, 15.3 (d, *J* = 9.6 Hz, 15.3, 15.2), 15.2 (d, *J* = 8.3 Hz, 15.3, 15.2) (minor).

HRMS (ESI-TOF): calculated for [C₂₀H₂₄I_NNaO₄ (M + Na)]⁺: 492.0648, found: 492.0648.

Benzyl benzyl(2-iodophenyl)carbamate (3bn):



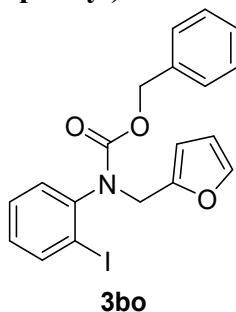
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as a yellow oil, 207 mg, yield 78% (a mixture of rotamers).

¹H NMR (400 MHz, CDCl₃): δ 7.88 (minor)/ 7.86 (major) (br d, *J* = 8.0 Hz 1H), 7.42–7.12 (br m, 11 H), 6.99–6.85 (br m, 1 H), 6.76 (br d, *J* = 7.8 Hz, 1H), 5.33 (br d, *J* = 14.7 Hz, 1H), 5.28 – 5.07 (br m, 2H), 4.25–4.10 (br m, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 155.3, 143.3, 139.9 (minor), 139.6, 137.2 (minor), 137.1, 136.5, 136.4 (minor), 130.6 (minor), 130.3, 129.3 (minor), 129.2, 129.0 (minor), 128.9 (minor), 128.8, 128.6 (minor), 128.5 (minor), 128.4, 128.3, 127.7, 127.7, 127.6 (minor), 127.5, 100.1, 99.8 (minor), 67.8 (minor), 67.5, 53.8 (minor), 53.5.

HRMS (ESI-TOF): calculated for [C₂₁H₁₉INO₂ (M + H)]⁺: 444.0460, found: 444.0459.

Benzyl (furan-2-ylmethyl)(2-iodophenyl)carbamate (3bo):



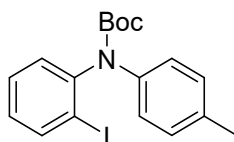
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as reddish-brown oil, 120 mg, yield 46% (a mixture of rotamers).

¹H NMR (400 MHz, CDCl₃): δ 7.88 (minor)/ 7.85 (major) (br d, *J* = 8.0 Hz 1H), 7.48 – 7.32 (br m, 2H), 7.32 – 7.14 (br m, 5H), 7.06 – 6.89 (br m, 2H), 6.28 – 6.24 (br m, 1H), 6.15 (major)/ 6.05 (minor) (br d, *J* = 2.8 Hz 1H), 5.34 – 5.07 (br m, 3H), 4.34 – 4.25 (br m, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 154.9, 154.7 (minor), 150.6 (minor), 150.4, 143.8 (minor), 143.1, 142.4, 142.3 (minor), 139.7 (minor), 139.5, 136.5, 130.4 (minor), 130.1, 129.5 (minor), 129.4, 129.3 (minor), 129.0, 128.5 (minor), 128.3, 128.2 (minor), 127.8, 127.5, 110.4, 109.5, 109.0 (minor), 100.2, 99.9 (minor), 67.9 (minor), 67.6, 46.4 (minor), 46.0.

HRMS (ESI-TOF): calculated for [C₁₉H₁₇INO₃ (M + H)]⁺: 434.0253, found: 434.0251.

***Tert*-butyl (2-iodophenyl)(*p*-tolyl)carbamate (3ca1):**



3ca1

The title compound was prepared according to the general procedure at 40 °C for 4 h, obtained as a yellow oil, 223 mg, yield 91%.

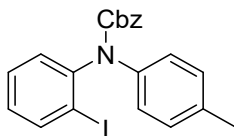
Scale-up: when **1ca1** (5.0 g, 24.2 mmol) was employed in this reaction (40 °C for 4 h), 9.21 g of **3ca1** was obtained, 93% yield.

¹H NMR (400 MHz, CDCl₃): δ 7.89 (d, *J* = 7.8 Hz, 1H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.26 (d, *J* = 6.7 Hz, 1H), 7.20 (d, *J* = 7.6 Hz, 2H), 7.07 (d, *J* = 7.8 Hz, 2H), 6.98 (t, *J* = 7.4 Hz, 1H), 2.29 (s, 3H), 1.45 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 152.8, 145.2, 139.8, 139.3, 134.7, 130.1, 129.3, 129.1, 128.7, 125.0, 100.7, 81.3, 28.3, 20.9.

HRMS (ESI-TOF): calculated for [C₁₈H₂₀INNaO₂ (M + Na)]⁺: 432.0436, found: 432.0436.

Benzyl (2-iodophenyl)(*p*-tolyl)carbamate (3ca2):



3ca2

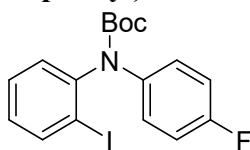
The title compound was prepared according to the general procedure at 40 °C for 2 h, obtained as a white solid, 260 mg, yield 98%. Melting Point: 102-104 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.90 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.37 – 7.20 (m, 9H), 7.12 – 7.07 (m, 2H), 7.00 (td, *J* = 8.0, 1.6 Hz, 1H), 5.22 (d, *J* = 4.3 Hz, 2H), 2.30 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 153.9, 144.5, 140.0, 138.9, 136.2, 135.3, 130.0, 129.4, 129.3, 129.0, 128.3, 127.9, 127.8, 125.0, 100.3, 67.7, 20.9.

HRMS (ESI-TOF): calculated for [C₂₁H₁₉INO₂ (M + H)]⁺: 444.0460, found: 444.0462.

***Tert*-butyl (4-fluorophenyl)(2-iodophenyl)carbamate (3cb):**



3cb

The title compound was prepared according to the general procedure at 40 °C for 3 h, obtained as a brown solid, 245 mg, yield 99%. Melting Point: 83-85 °C.

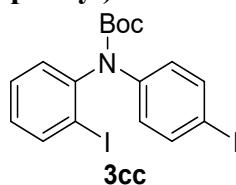
¹H NMR (400 MHz, CDCl₃): δ 7.90 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.36 (td, *J* = 7.7, 1.4 Hz, 1H), 7.33 – 7.28 (m, 2H), 7.27 – 7.25 (m, 1H), 7.03 – 6.93 (m, 3H), 1.45 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 160.0 (d, *J* = 244.9 Hz, 161.2, 158.8), 152.8, 144.9, 140.0, 137.8, 130.1, 129.4, 128.9, 126.9, 115.3 (d, *J* = 22.5 Hz, 115.4, 115.2), 100.5, 81.7, 28.3.

¹⁹F NMR (377 MHz, CDCl₃): δ -117.3.

HRMS (ESI-TOF): calculated for [C₁₇H₁₇FINNaO₂ (M + Na)]⁺: 436.0186, found: 436.0183.

***Tert*-butyl (2-iodophenyl)(4-iodophenyl)carbamate (3cc):**



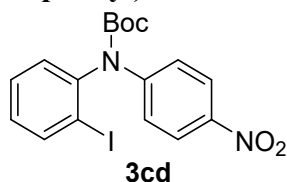
The title compound was prepared according to the general procedure at 40 °C for 3 h, obtained as a yellow solid, 294 mg, yield 94%. Melting Point: 92-94 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.90 (d, *J* = 7.6 Hz, 1H), 7.56 (d, *J* = 7.8 Hz, 2H), 7.37 (t, *J* = 7.1 Hz, 1H), 7.24 (d, *J* = 8.8 Hz, 1H), 7.12 – 6.95 (m, 3H), 1.44 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 152.4, 144.4, 141.5, 140.0, 137.5, 130.2, 129.5, 129.1, 126.4, 100.6, 88.9, 81.9, 28.2.

HRMS (ESI-TOF): calculated for [C₁₇H₁₇I₂NNaO₂ (M + Na)]⁺: 543.9246, found: 543.9247.

***Tert*-butyl (2-iodophenyl)(4-nitrophenyl)carbamate (3cd):**



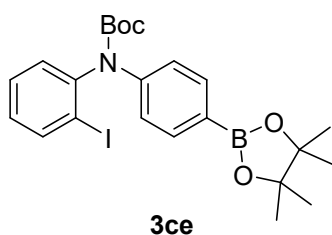
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as a yellow solid, 187 mg, yield 71%. Melting Point: 80-85 °C.

¹H NMR (400 MHz, CDCl₃): δ 8.12 (d, *J* = 8.7 Hz, 2H), 7.95 (d, *J* = 7.8 Hz, 1H), 7.48 – 7.35 (m, 3H), 7.27 (d, *J* = 7.3 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 1.44 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 152.0, 147.3, 143.5, 143.4, 140.3, 130.2, 129.8, 129.8, 124.3, 122.8, 100.4, 82.9, 28.1.

HRMS (ESI-TOF): calculated for [C₁₇H₁₇IN₂NaO₄ (M + Na)]⁺: 463.0131, found: 463.0131.

***Tert*-butyl (2-iodophenyl) (4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) phenyl) carbamate (3ce):**



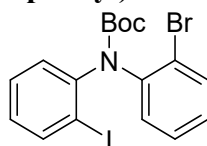
The title compound was prepared according to the general procedure at 40 °C for 4 h, obtained as gray solid, 216 mg, yield 69%. Melting Point: 140-142 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.90 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.71 (d, *J* = 8.6 Hz, 2H), 7.36 (td, *J* = 7.7, 1.4 Hz, 1H), 7.28 – 7.22 (m, 3H), 7.01 (td, *J* = 7.8, 1.6 Hz, 1H), 1.45 (s, 9H), 1.32 (s, 12H).

¹³C NMR (101 MHz, CDCl₃): δ 152.5, 144.8, 144.4, 139.9, 135.2, 130.3, 129.4, 128.9, 123.5, 100.8, 83.8, 81.7, 28.3, 24.9.

HRMS (ESI-TOF): calculated for [C₂₃H₂₉BINNaO₄ (M + Na)]⁺: 544.1132, found: 544.1133.

***Tert*-butyl (2-bromophenyl)(2-iodophenyl)carbamate (3cf):**



3cf

The title compound was prepared according to the general procedure at 40 °C for 5 h, obtained as a white solid, 278 mg, yield 98% (a mixture of rotamers). Melting Point: 140-141 °C.

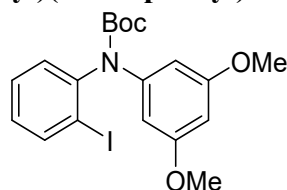
Scale-up: when **1cf** (1.0 g, 3.69 mmol) was employed in this reaction (40 °C for 3 h), 1.71 g of **3cf** was obtained, 98% yield.

¹H NMR (400 MHz, CDCl₃): δ 7.90 (d, *J* = 5.0 Hz, 1H), 7.64 (t, *J* = 6.5 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.30 – 7.18 (m, 2H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.93 (t, *J* = 7.5 Hz, 1H), 1.48 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 151.8 / 151.8, 145.6 / 145.4, 142.2 / 142.0, 140.1 / 139.8, 133.7 / 133.4, 129.8, 129.5 / 129.3, 129.2, 128.8, 128.6 / 128.5, 128.5 / 128.4, 123.4 / 123.2, 100.0 / 99.8, 81.7 / 81.7, 28.3.

HRMS (ESI-TOF): calculated for [C₁₇H₁₇BrINNaO₂ (M + Na)]⁺: 495.9385, found: 495.9379.

***Tert*-butyl (3,5-dimethoxyphenyl)(2-iodophenyl)carbamate (3cg):**



3cg

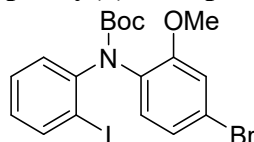
The title compound was prepared according to the general procedure at 40 °C for 9 h, obtained as a white solid, 262 mg, yield 96%. Melting Point: 102-105 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.89 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.34 (td, *J* = 7.6, 1.3 Hz, 1H), 7.27 – 7.24 (m, 1H), 6.99 (td, *J* = 7.7, 1.6 Hz, 1H), 6.51 (d, *J* = 2.2 Hz, 2H), 6.24 (t, *J* = 2.2 Hz, 1H), 3.73 (s, 6H), 1.45 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 160.5, 152.5, 144.9, 143.5, 139.8, 130.0, 129.4, 128.9, 103.5, 100.6, 96.9, 81.5, 55.4, 28.3.

HRMS (ESI-TOF): calculated for [C₁₉H₂₂INNaO₄ (M + Na)]⁺: 478.0491, found: 478.0492.

***Tert*-butyl (4-bromo-2-methoxyphenyl)(2-iodophenyl)carbamate (3ch):**



3ch

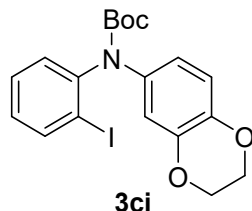
The title compound was prepared according to the general procedure at 40 °C for 4 h, obtained as a white solid, 278 mg, yield 92%. Melting Point: 147-152 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.86 (d, *J* = 7.9 Hz, 1H), 7.32 – 7.20 (m, 3H), 7.07 (s, 1H), 7.00 (dd, *J* = 8.3, 1.5 Hz, 1H), 6.94 – 6.88 (m, 1H), 3.89 (s, 3H), 1.43 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 155.6, 152.8, 145.6, 139.6, 131.4, 129.3, 128.9, 128.4, 123.9, 121.2, 116.0, 115.2, 100.2, 81.0, 55.9, 28.2.

HRMS (ESI-TOF): calculated for [C₁₈H₁₉BrINNaO₃ (M + Na)]⁺: 525.9491, found: 525.9490.

***Tert*-butyl (2,3-dihydrobenzo[b][1,4]dioxin-6-yl)(2-iodophenyl)carbamate (3ci):**



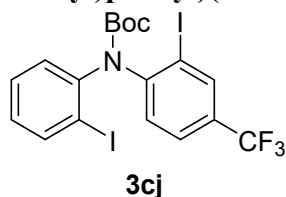
The title compound was prepared according to the general procedure at 40 °C for 5 h, obtained as a yellow solid, 245 mg, yield 90%. Melting Point: 90-93 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.88 (d, *J* = 7.8 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.25 (s, 1H), 6.97 (t, *J* = 7.4 Hz, 1H), 6.86 (d, *J* = 10.3 Hz, 2H), 6.76 (d, *J* = 8.5 Hz, 1H), 4.21 (s, 4H), 1.44 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 152.9, 145.2, 143.1, 141.3, 139.8, 135.4, 130.0, 129.3, 128.7, 119.0, 116.8, 114.8, 100.5, 81.4, 64.4, 64.3, 28.3.

HRMS (ESI-TOF): calculated for [C₁₉H₂₀INNaO₄ (M + Na)]⁺: 476.0335, found: 476.0337.

***Tert*-butyl (2-iodo-4-(trifluoromethyl)phenyl)(2-iodophenyl)carbamate (3cj):**



The title compound was prepared according to the general procedure at 40 °C for 10 h, obtained as a yellow oil, 269 mg, yield 76% (a mixture of rotamers).

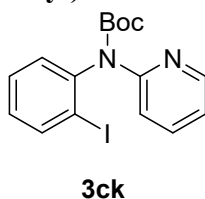
¹H NMR (400 MHz, CDCl₃): δ 8.15 (s, 1H), 7.93 (d, *J* = 7.8 Hz, 1H), 7.53 (m, 2H), 7.43 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.29 (td, *J* = 7.7, 1.4 Hz, 1H), 6.99 (td, *J* = 7.8, 1.6 Hz, 1H), 1.51 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 151.0, 148.7, 145.0, 140.3 (d, *J* = 31.1 Hz, 140.4, 140.1), 137.1 (d, *J* = 36.2 Hz, 137.2, 136.9), 130.6, 130.3, 129.6 (minor), 129.5, 129.4 (minor), 129.2, 128.7, 126.5 (q, *J* = 3.5 Hz, 126.5, 126.5, 126.5, 126.4), 125.7 (q, *J* = 3.5 Hz, 125.7, 125.7, 125.7, 125.6) (minor), 123.8, 122.8 (q, *J* = 273.7 Hz, 126.8, 124.1, 121.4, 118.7), 100.7 / 99.8 / 99.6 / 99.5, 82.5, 82.3 (minor), 28.4, 28.2 (minor).

¹⁹F NMR (377 MHz, CDCl₃): δ -62.6, -62.7.

HRMS (ESI-TOF): calculated for [C₁₈H₁₆F₃I₂NNaO₂ (M + Na)]⁺: 611.9120, found: 611.9127.

***Tert*-butyl (2-iodophenyl)(pyridin-2-yl)carbamate (3ck):**



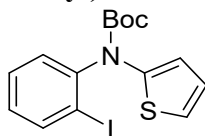
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as a yellow solid, 109 mg, yield 46%. Melting Point: 80-83 °C.

¹H NMR (400 MHz, CDCl₃): δ 8.27 – 8.23 (m, 1H), 7.92 – 7.86 (m, 2H), 7.68 (ddd, *J* = 8.4, 7.3, 2.0 Hz, 1H), 7.43 – 7.33 (m, 2H), 7.05 – 6.96 (m, 2H), 1.44 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 154.0, 152.7, 147.9, 143.8, 139.4, 137.2, 130.9, 128.9, 128.9, 119.6, 118.9, 100.7, 81.9, 28.2.

HRMS (ESI-TOF): calculated for [C₁₆H₁₈IN₂O₂ (M + H)]⁺: 397.0413, found: 397.0406.

***Tert*-butyl (2-iodophenyl)(thiophen-2-yl)carbamate (3cl):**



3cl

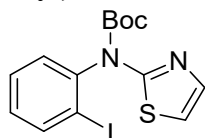
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as black solid, 192 mg, yield 80%. Melting Point: 111-115 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.93 (d, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.3 Hz, 1H), 7.36 (d, *J* = 7.0 Hz, 1H), 7.09 (t, *J* = 7.3 Hz, 1H), 6.90 (d, *J* = 4.6 Hz, 1H), 6.73 (m, 1H), 5.99 (m, 1H), 1.43 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 151.6, 144.2, 143.6, 140.1, 129.7, 129.6, 124.2, 118.5, 113.7, 99.6, 82.4, 28.2.

HRMS (ESI-TOF): calculated for [C₁₅H₁₇INO₂S (M + H)]⁺: 402.0025, found: 402.0022.

***Tert*-butyl (2-iodophenyl)(thiazol-2-yl)carbamate (3cm):**



3cm

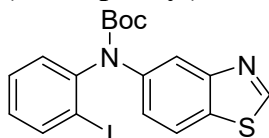
The title compound was prepared according to the general procedure at 50 °C for 10 h, obtained as orange solid, 111 mg, yield 46%. Melting Point: 137-139 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.93 (d, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.35 (d, *J* = 4.5 Hz, 2H), 7.13 (t, *J* = 7.4 Hz, 1H), 6.98 (s, 1H), 1.45 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 161.2, 151.7, 142.1, 141.3, 139.7, 138.2, 129.9, 129.4, 113.9, 99.4, 83.7, 28.1.

HRMS (ESI-TOF): calculated for [C₁₄H₁₆IN₂O₂S (M + H)]⁺: 402.9977, found: 402.9974.

***Tert*-butyl benzo[d]thiazol-5-yl(2-iodophenyl)carbamate (3cn):**



3cn

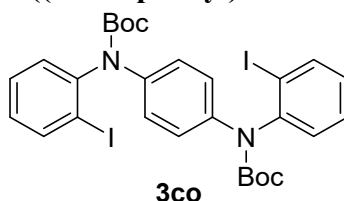
The title compound was prepared according to the general procedure at 40 °C for 10 h, obtained as a yellow oil, 176 mg, yield 65%.

¹H NMR (400 MHz, CDCl₃): δ 8.95 (s, 1H), 7.95 (d, *J* = 2.0 Hz, 1H), 7.92 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.85 (d, *J* = 8.7 Hz, 1H), 7.62 (d, *J* = 8.3 Hz, 1H), 7.35 (dtd, *J* = 9.7, 7.9, 1.6 Hz, 2H), 7.01 (td, *J* = 7.9, 1.8 Hz, 1H), 1.47 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 154.9, 153.7, 152.8, 144.9, 140.6, 140.0, 130.3, 130.2, 129.5, 129.0, 123.8, 121.3, 119.6, 100.6, 81.9, 28.3.

HRMS (ESI-TOF): calculated for [C₁₈H₁₈IN₂O₂S (M + H)]⁺: 453.0134, found: 453.0131.

Di-*tert*-butyl 1,4-phenylenebis((2-iodophenyl)carbamate) (3co):



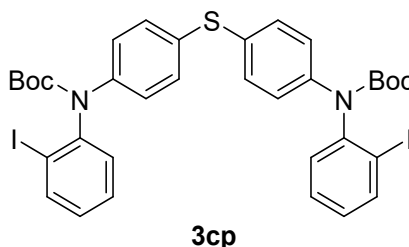
The title compound was prepared according to the general procedure (5.0 equiv of NaH and 5.0 equiv of **2a** were used) at 40 °C for 12 h, obtained as a yellow solid, 158 mg, yield 37%. Melting Point: 195-200 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.88 (dd, *J* = 7.9, 1.3 Hz, 2H), 7.33 (td, *J* = 7.7, 1.4 Hz, 2H), 7.25 – 7.19 (m, 6H), 6.98 (td, *J* = 7.8, 1.6 Hz, 2H), 1.44 (s, 18H).

¹³C NMR (101 MHz, CDCl₃): δ 152.8, 144.9, 139.8, 138.5, 130.2, 129.3, 128.8, 125.0, 100.7, 81.6, 28.3.

HRMS (ESI-TOF): calculated for [C₂₈H₃₀I₂N₂NaO₄ (M + Na)]⁺: 735.0193, found: 735.0189.

***Tert*-butyl (4-((4-((*tert*-butoxycarbonyl) (2-iodophenyl)amino)phenyl)thio)phenyl) (phenyl)carbamate (3cp):**



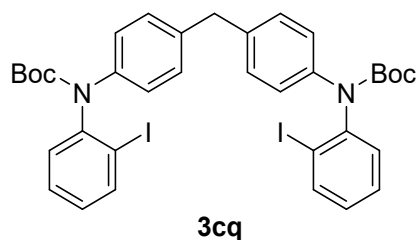
The title compound was prepared according to the general procedure (5.0 equiv of NaH and 5.0 equiv of **2a** were used) at 40 °C for 6 h, obtained as a white solid, 438 mg, yield 89%. Melting Point: 184-187 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.90 (dd, *J* = 7.9, 1.3 Hz, 2H), 7.36 (td, *J* = 7.7, 1.4 Hz, 2H), 7.25 (d, *J* = 1.6 Hz, 1H), 7.23 (d, *J* = 1.6 Hz, 1H), 7.22 (m, 8H), 7.01 (td, *J* = 7.8, 1.6 Hz, 2H), 1.44 (s, 18H).

¹³C NMR (101 MHz, CDCl₃): δ 152.6, 144.6, 140.8, 139.9, 131.2, 130.2, 129.4, 129.0, 125.3, 100.7, 81.8, 28.3.

HRMS (ESI-TOF): calculated for [C₃₄H₃₄I₂N₂NaO₄S (M + Na)]⁺: 843.0226, found: 843.0226.

Di-*tert*-butyl (methylenebis(4,1-phenylene))bis((2-iodophenyl)carbamate) (3cq):



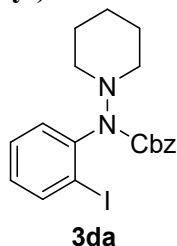
The title compound was prepared according to the general procedure (5.0 equiv of NaH and 5.0 equiv of **2a** were used) at 40 °C for 6 h, obtained as a yellowish oil, 452 mg, yield 94%.

¹H NMR (400 MHz, CDCl₃): δ 7.88 (dd, *J* = 7.9, 1.3 Hz, 2H), 7.33 (td, *J* = 7.7, 1.3 Hz, 2H), 7.25 – 7.17 (m, 6H), 7.05 (d, *J* = 8.5 Hz, 4H), 6.97 (td, *J* = 7.8, 1.6 Hz, 2H), 3.86 (s, 2H), 1.44 (s, 18H).

¹³C NMR (101 MHz, CDCl₃): δ 152.8, 145.1, 139.9, 139.8, 137.7, 130.1, 129.3, 129.0, 128.7, 125.0, 100.7, 81.5, 40.8, 28.3.

HRMS (ESI-TOF): calculated for [C₃₅H₃₆I₂N₂NaO₄ (M + Na)]⁺: 825.0662, found: 825.0651.

Benzyl (2-iodophenyl)(piperidin-1-yl)carbamate (3da):



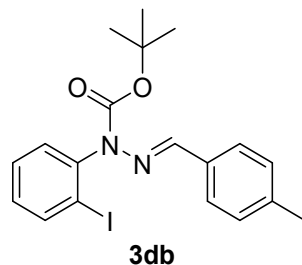
The title compound was prepared according to the general procedure at 40 °C for 12 h, obtained as a white solid, 204 mg, yield 78%. Melting Point: 98-100 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.87 (d, *J* = 7.8 Hz, 1H), 7.37 – 7.10 (m, 7H), 6.98 (t, *J* = 7.4 Hz, 1H), 5.16 (s, 2H), 3.30 (m, 4H), 1.61 (m, 4H), 1.37 (m, 2H).

¹³C NMR (101 MHz, CDCl₃): δ 153.6, 144.3, 139.8, 136.4, 128.9, 128.8, 128.6, 128.4, 128.3, 127.8, 99.8, 67.3, 53.4, 26.6, 23.5.

HRMS (ESI-TOF): calculated for [C₁₉H₂₂IN₂O₂ (M + H)]⁺: 437.0726, found: 437.0724.

***Tert*-butyl (*E*)-1-(2-iodophenyl)-2-(4-methylbenzylidene)hydrazine-1-carboxylate (3db):**



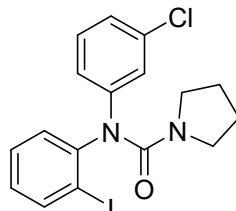
The title compound was prepared according to the general procedure at 40 °C for 2 h, obtained as a reddish brown solid, 84 mg, yield 32%. Melting Point: 150-153 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.98 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 7.8 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.22 (d, *J* = 7.7 Hz, 1H), 7.17 – 7.09 (m, 3H), 7.03 (s, 1H), 2.33 (s, 3H), 1.49 (s, 9H).

^{13}C NMR (101 MHz, CDCl_3): δ 142.1, 140.3, 140.1, 139.9, 131.7, 130.6, 130.5, 129.9, 129.8, 129.2, 127.4, 100.4, 82.1, 28.2, 21.5.

HRMS (ESI-TOF): calculated for $[\text{C}_{19}\text{H}_{22}\text{IN}_2\text{O}_2 (\text{M} + \text{H})]^+$: 437.0726, found: 437.0720.

***N*-(3-chlorophenyl)-*N*-(2-iodophenyl)pyrrolidine-1-carboxamide (3dc):**



3dc

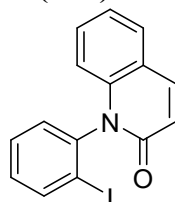
The title compound was prepared according to the general procedure for 6 h, obtained as orange solid, 115 mg, yield 45% (a mixture of rotamers). Melting Point: 112-115 °C.

^1H NMR (400 MHz, CDCl_3): δ 7.87 (d, $J = 8.0$ Hz, 1H), 7.33 (t, $J = 7.7$ Hz, 1H), 7.20 (t, $J = 8.0$ Hz, 1H), 7.17 – 7.05 (m, 2H), 6.98 – 6.85 (m, 3H), 3.28 (m, 4H), 1.85 – 1.73 (m, 4H).

^{13}C NMR (101 MHz, CDCl_3): δ 156.9, 152.4 (minor), 146.2, 145.3, 140.5 (minor), 140.1, 134.7, 134.2 (minor), 130.0, 129.7, 129.7, 128.2, 124.6, 124.5, 122.7, 122.2 (minor), 118.9 (minor), 116.9 (minor), 99.6, 47.9, 25.4.

HRMS (ESI-TOF): calculated for $[\text{C}_{17}\text{H}_{17}\text{ClIN}_2\text{O} (\text{M} + \text{H})]^+$: 427.0074, found: 427.0075.

1-(2-Iodophenyl)quinolin-2(1*H*)-one (3dd):



3dd

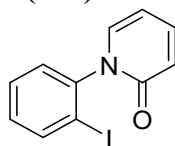
The title compound was prepared according to the general procedure for 6 h, obtained as a yellow solid, 62 mg, yield 30%. Melting Point: 195-200 °C.

^1H NMR (400 MHz, CDCl_3): δ 8.06 (d, $J = 7.9$ Hz, 1H), 7.82 (d, $J = 9.6$ Hz, 1H), 7.62 (d, $J = 7.6$ Hz, 1H), 7.58 (t, $J = 7.8$ Hz, 1H), 7.40 – 7.31 (m, 2H), 7.26 – 7.19 (m, 2H), 6.79 (d, $J = 9.5$ Hz, 1H), 6.49 (d, $J = 8.4$ Hz, 1H).

^{13}C NMR (101 MHz, CDCl_3): δ 161.4, 140.6, 140.6, 140.3, 140.0, 130.6, 130.5, 130.2, 130.0, 128.6, 122.7, 122.3, 120.4, 115.4, 99.2.

HRMS (ESI-TOF): calculated for $[\text{C}_{15}\text{H}_{11}\text{INO} (\text{M} + \text{H})]^+$: 347.9885, found: 347.9880.

1-(2-Iodophenyl)pyridin-2(1*H*)-one (3de):



3de

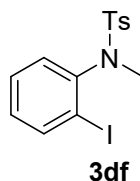
The title compound was prepared according to the general procedure for 15 h, obtained as a brown solid, 73 mg, yield 41%. Melting Point: 78-80 °C.

¹H NMR (400 MHz, CDCl₃): δ 8.17 (ddd, *J* = 5.0, 1.9, 0.6 Hz, 1H), 7.88 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.72 (ddd, *J* = 8.3, 7.2, 2.0 Hz, 1H), 7.39 (ddd, *J* = 8.1, 7.5, 1.6 Hz, 1H), 7.15 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.04 – 6.94 (m, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 163.0, 154.0, 147.7, 139.8, 139.6, 129.6, 126.7, 123.0, 118.7, 111.7, 90.9.

HRMS (ESI-TOF): calculated for [C₁₁H₉INO (M + H)]⁺: 297.9729, found: 297.9729.

***N*-(2-iodophenyl)-*N*,4-dimethylbenzenesulfonamide (3df):**



The title compound was prepared according to the general procedure (a mixture of 6 mL of THF and 1.2 mL of DMA was use as the solvent) for 12 h, obtained as a white solid, 187 mg, yield 97%.

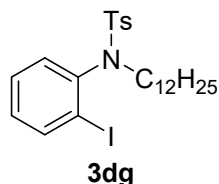
¹H NMR (400 MHz, CDCl₃): δ 7.90 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 7.27 (td, *J* = 7.6, 1.5 Hz, 1H), 7.02 (td, *J* = 7.7, 1.6 Hz, 1H), 6.96 (dd, *J* = 7.9, 1.6 Hz, 1H), 3.13 (s, 3H), 2.45 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 143.9, 140.4, 135.7, 130.0, 129.7, 129.2, 128.9, 128.3, 101.9, 38.9, 21.7.

HRMS (ESI-TOF): calculated for [C₁₄H₁₅INO₂S (M + H)]⁺: 387.9868, found: 387.9859.

The ¹H NMR and ¹³C NMR of **3df** are consistent with the reported spectra^[27].

***N*-dodecyl-*N*-(2-iodophenyl)-4-methylbenzenesulfonamide (3dg):**



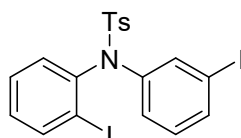
The title compound was prepared according to the general procedure (a mixture of 6 mL of THF and 1.2 mL of DMA was use as the solvent) for 12 h, obtained as a colorless oil, 230 mg, yield 85%.

¹H NMR (400 MHz, CDCl₃): δ 7.92 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.64 (d, *J* = 8.1 Hz, 2H), 7.32 – 7.25 (m, 3H), 7.02 (td, *J* = 7.6, 1.6 Hz, 1H), 6.92 (dd, *J* = 7.9, 1.6 Hz, 1H), 3.70 – 3.54 (m, 1H), 3.42 – 3.28 (m, 1H), 2.44 (s, 3H), 1.58 – 1.46 (m, 1H), 1.43 – 1.35 (m, 1H), 1.32 – 1.14 (m, 18H), 0.88 (t, *J* = 6.8 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 143.7, 141.9, 140.6, 136.4, 130.4, 129.9, 129.6, 128.9, 128.3, 103.5, 52.1, 32.0, 29.7, 29.6, 29.6, 29.5, 29.3, 27.0, 22.8, 21.7, 14.2.

HRMS (ESI-TOF): calculated for [C₂₅H₃₇INO₂S (M + H)]⁺: 542.1590, found: 542.1581.

***N*-(2-iodophenyl)-*N*-(3-iodophenyl)-4-methylbenzenesulfonamide (3dh):**



3dh

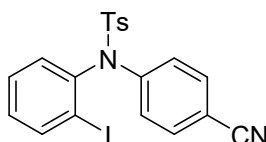
The title compound was prepared according to the general procedure (a mixture of 2.4 mL of THF and 2.4 mL of DMA was use as the solvent) for 12 h, obtained as white solid, 150 mg, yield 52%. Melting Point: 132 – 134 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.91 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.70 (t, *J* = 1.9 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.43 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.36 (td, *J* = 7.6, 1.5 Hz, 1H), 7.32 – 7.27 (m, 3H), 7.10 – 6.99 (m, 2H), 2.45 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 144.4, 142.3, 141.2, 141.1, 136.7, 136.4, 136.0, 131.0, 130.5, 130.3, 129.8, 129.2, 128.4, 127.2, 102.7, 93.7, 21.8.

HRMS (ESI-TOF): calculated for [C₁₉H₁₅I₂NNaO₂S (M + Na)]⁺: 597.8811, found: 597.8808.

***N*-(4-cyanophenyl)-*N*-(2-iodophenyl)-4-methylbenzenesulfonamide (3di):**



3di

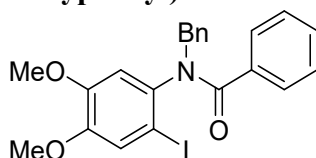
The title compound was prepared according to the general procedure (a mixture of 2.4 mL of THF and 2.4 mL of DMA was use as the solvent) for 12 h, obtained as a white solid, 119 mg, yield 50%. Melting Point: 133 – 135 °C

¹H NMR (400 MHz, CDCl₃): δ 7.93 (d, *J* = 8.0 Hz, 1H), 7.64 (d, *J* = 8.0 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.48 – 7.37 (m, 3H), 7.36 – 7.27 (m, 3H), 7.12 (t, *J* = 7.7 Hz, 1H), 2.44 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 144.9, 144.4, 141.5, 141.3, 136.6, 133.0, 131.6, 130.9, 130.0, 129.4, 128.2, 125.5, 118.5, 109.2, 102.3, 21.8.

HRMS (ESI-TOF): calculated for [C₂₀H₁₆IN₂O₂S (M + H)]⁺: 474.9977, found: 474.9984.

***N*-benzyl-*N*-(2-iodo-4,5-dimethoxyphenyl)benzamide (3ef):**



3ef

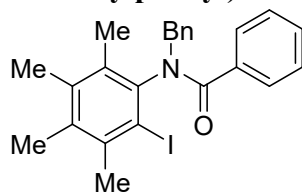
The title compound was prepared according to the general procedure at 40 °C for 10 h, obtained as a yellow oil, 238 mg, yield 84%.

¹H NMR (400 MHz, CDCl₃): δ 7.39 (d, *J* = 7.2 Hz, 2H), 7.32 – 7.23 (m, 5H), 7.21 (d, *J* = 7.2 Hz, 1H), 7.15 (t, *J* = 7.4 Hz, 2H), 7.09 (s, 1H), 5.98 (s, 1H), 5.86 (d, *J* = 14.0 Hz, 1H), 4.15 (d, *J* = 14.0 Hz, 1H), 3.78 (s, 3H), 3.37 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.6, 148.8, 148.7, 137.3, 137.1, 136.1, 129.8, 129.7, 128.5, 128.1, 127.7, 127.6, 120.9, 114.9, 88.2, 56.1, 55.8, 52.3.

HRMS (ESI-TOF): calculated for [C₂₂H₂₁INO₃ (M + H)]⁺: 474.0566, found: 474.0563.

***N*-benzyl-*N*-(2-iodo-3,4,5,6-tetramethylphenyl)benzamide (3eg):**



3eg

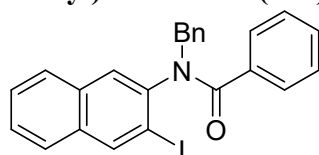
The title compound was prepared according to the general procedure at 40 °C for 10 h, obtained as a yellowish solid, 211 mg, yield 75%. Melting Point: 150-152 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.39 – 7.35 (m, 2H), 7.28 – 7.15 (m, 6H), 7.11 – 7.06 (m, 2H), 5.76 (d, *J* = 13.7 Hz, 1H), 4.23 (d, *J* = 13.7 Hz, 1H), 2.55 (s, 3H), 2.23 (s, 3H), 1.88 (s, 3H), 1.31 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 170.1, 140.5, 138.8, 136.5, 136.5, 136.3, 135.4, 135.0, 130.8, 129.6, 128.1, 127.8, 127.7, 127.3, 107.1, 53.9, 28.0, 18.5, 17.6, 16.8.

HRMS (ESI-TOF): calculated for [C₂₄H₂₅INO (M + H)]⁺: 470.0981, found: 470.0988.

***N*-benzyl-*N*-(3-iodonaphthalen-2-yl)benzamide (3eh):**



3eh

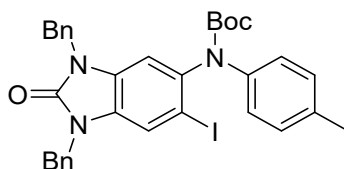
The title compound was prepared according to the general procedure at 40 °C for 1.5 h, obtained as reddish-brown oil, 247 mg, yield 89%.

¹H NMR (400 MHz, CDCl₃): δ 8.34 (s, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.46 – 7.37 (m, 5H), 7.32 – 7.26 (m, 5H), 7.14 – 7.03 (m, 4H), 5.91 (d, *J* = 14.3 Hz, 1H), 4.27 (d, *J* = 14.3 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 170.6, 140.7, 139.9, 136.9, 136.0, 133.6, 132.6, 130.6, 129.7, 128.5, 128.4, 127.9, 127.7, 127.7, 127.4, 127.1, 126.5, 97.1, 53.1.

HRMS (ESI-TOF): calculated for [C₂₄H₁₉INO (M + H)]⁺: 464.0511, found: 464.0507.

***Tert*-butyl (1,3-dibenzyl-6-iodo-2-oxo-2,3-dihydro-1*H*-benzo[d]imidazol-5-yl)(*p*-tolyl)carbamate (3ei):**



3ei

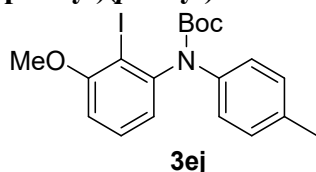
The title compound was prepared according to the general procedure (1.5 equiv of **2g** was used) at 45 °C for 10 h, obtained as a white solid, 271 mg, yield 70%. Melting Point: 144-146 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.38 – 7.22 (m, 11H), 7.07 (d, *J* = 8.3 Hz, 2H), 7.01 (d, *J* = 8.2 Hz, 2H), 6.79 (d, *J* = 1.9 Hz, 1H), 5.13 – 4.97 (m, 4H), 2.27 (s, 3H), 1.36 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 154.5, 153.1, 139.1, 138.4, 135.7, 135.6, 134.6, 130.1, 129.6, 129.0, 128.9, 128.0, 127.9, 127.5, 127.5, 124.9, 118.0, 110.2, 90.4, 81.3, 45.2, 28.2, 20.9.

HRMS (ESI-TOF): calculated for [C₃₃H₃₃IN₃O₃ (M + H)]⁺: 646.1567, found: 646.1559.

***Tert*-butyl (2-iodo-3-methoxyphenyl)(*p*-tolyl)carbamate (3ej):**



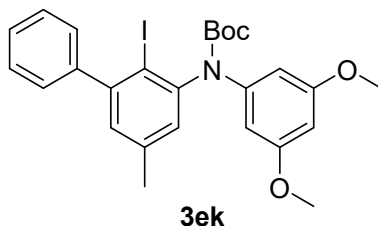
The title compound was prepared according to the general procedure at 60 °C for 12 h, obtained as a yellow oil, 176 mg, yield 67%.

¹H NMR (400 MHz, CDCl₃): δ 7.28 (t, *J* = 8.1 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.06 (d, *J* = 8.2 Hz, 2H), 6.90 (dd, *J* = 7.9, 1.2 Hz, 1H), 6.73 (dd, *J* = 8.3, 1.0 Hz, 1H), 3.90 (s, 3H), 2.28 (s, 3H), 1.44 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 159.5, 152.9, 146.7, 139.3, 134.6, 129.7, 129.1, 124.9, 122.6, 109.4, 93.5, 81.3, 56.7, 28.3, 20.9.

HRMS (ESI-TOF): calculated for [C₁₉H₂₂INNaO₃ (M + Na)]⁺: 462.0542, found: 462.0543.

***Tert*-butyl(3,5-dimethoxyphenyl)(2-iodo-5-methyl-[1,1'-biphenyl]-3-yl)carbamate (3ek):**



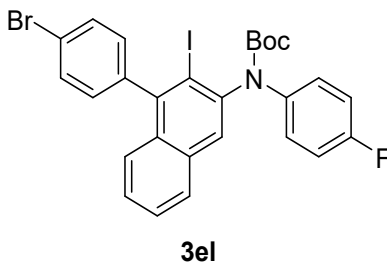
The title compound was prepared according to the general procedure at 50 °C for 6 h, obtained as a yellow solid, 213 mg, yield 65%. Melting Point: 120-123 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.44 – 7.35 (m, 3H), 7.32 (d, *J* = 6.8 Hz, 2H), 7.04 (s, 1H), 7.02 (s, 1H), 6.58 (s, 2H), 6.26 (s, 1H), 3.76 (s, 6H), 2.30 (s, 3H), 1.48 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 160.5, 152.7, 148.7, 145.3, 144.9, 143.7, 139.0, 129.7, 129.4, 129.2, 127.9, 127.7, 103.6, 101.8, 96.7, 81.5, 55.4, 28.4, 20.8.

HRMS (ESI-TOF): calculated for [C₂₆H₂₉INO₄ (M + H)]⁺: 546.1141, found: 546.1132.

***Tert*-butyl(4-(4-bromophenyl)-3-iodonaphthalen-2-yl)(4-fluorophenyl)carbamate (3el):**



The title compound was prepared according to the general procedure at 50 °C for 4 h, obtained as a yellow solid, 329 mg, yield 89%. Melting Point: 140-145 °C.

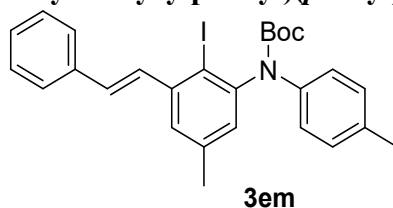
¹H NMR (400 MHz, CDCl₃): δ 7.84 – 7.79 (m, 2H), 7.71 – 7.65 (m, 2H), 7.50 (ddd, *J* = 8.1, 6.2, 1.8 Hz, 1H), 7.44 – 7.39 (m, 2H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.14 (d, *J* = 8.6 Hz, 2H), 6.99 (t, *J* = 8.7 Hz, 2H), 1.46 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 161.3, 158.8, 153.1, 146.2, 142.7, 141.6, 138.3, 133.5, 132.0, 131.8, 131.8, 128.3, 128.1, 127.5, 127.1, 126.9, 122.4, 115.4 (d, *J* = 22.5 Hz, 115.5, 115.2), 105.6, 81.9, 28.4.

¹⁹F NMR (377 MHz, CDCl₃): δ -117.3.

HRMS (ESI-TOF): calculated for [C₂₇H₂₃BrFINO₂ (M + H)]⁺: 617.9941, found: 617.9933.

***Tert*-butyl (*E*)-(2-iodo-5-methyl-3-styrylphenyl)(*p*-tolyl)carbamate (**3em**):**



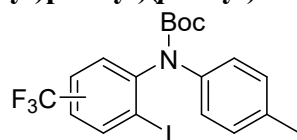
The title compound was prepared according to the general procedure at 50 °C for 3 h, obtained as a yellow solid, 161 mg, yield 51%. Melting Point: 133-135 °C.

¹H NMR (400 MHz, CDCl₃): δ 7.55 (d, *J* = 6.9 Hz, 2H), 7.45 – 7.32 (m, 4H), 7.29 (d, *J* = 7.2 Hz, 1H), 7.27 – 7.20 (m, 2H), 7.08 (d, *J* = 6.6 Hz, 2H), 6.99 (s, 1H), 6.93 (d, *J* = 15.9 Hz, 1H), 2.31 (s, 3H), 2.29 (s, 3H), 1.47 (s, 9H).

¹³C NMR (101 MHz, CDCl₃): δ 153.0, 145.7, 142.4, 139.4, 139.1, 137.0, 134.6, 133.4, 131.8, 129.7, 129.1, 128.8, 128.1, 126.9, 125.9, 124.8, 103.6, 81.4, 28.4, 20.9, 20.9.

HRMS (ESI-TOF): calculated for [C₂₇H₂₉INO₂ (M + H)]⁺: 526.1243, found: 526.1243.

***Tert*-butyl (2-iodo-4-(trifluoromethyl)phenyl)(*p*-tolyl)carbamate (**3en**) and *Tert*-butyl (2-iodo-5-(trifluoromethyl)phenyl)(*p*-tolyl)carbamate (**3en'**):**



The title compound was prepared according to the general procedure at 40 °C for 6 h, obtained as a yellow oil, 238 mg, yield 83%.

Major isomer **3en**: **¹H NMR (400 MHz, CDCl₃):** δ 8.14 (s, 1H), 7.59 (d, *J* = 8.1 Hz, 1H), 7.34 (d, *J* = 8.2 Hz, 1H), 7.19 – 7.14 (m, 2H), 7.12 – 7.06 (m, 2H), 2.30 (s, 3H), 1.45 (s, 9H).

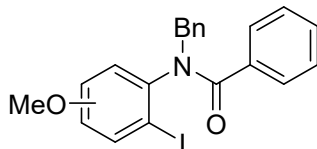
Minor isomer **3en'**: **¹H NMR (400 MHz, CDCl₃):** δ 8.03 (d, *J* = 8.2 Hz, 1H), 7.47 (s, 1H), 7.22 (d, *J* = 8.5 Hz, 1H), 7.19 – 7.14 (m, 2H), 7.12 – 7.06 (m, 2H), 2.30 (s, 3H), 1.45 (s, 9H).

Mixture of **3en** and **3en'** **¹³C NMR (101 MHz, CDCl₃):** δ 152.5, 152.4, 148.7, 146.1, 140.7, 138.8, 138.7, 136.9 (q, *J* = 3.8 Hz, 137.0, 136.9, 136.9, 136.9), 135.5, 135.5, 132.2, 131.8, 130.7, 130.3, 130.3, 129.5, 129.4, 129.4, 126.8 (q, *J* = 3.5 Hz, 126.8, 126.8, 126.8, 126.7), 126.4 (q, *J* = 3.4 Hz, 126.4, 126.4, 126.4, 126.3), 125.3, 125.2, 125.1 (q, *J* = 3.7 Hz, 125.2, 125.1, 125.1, 125.0), 124.8, 124.1, 122.1, 121.4, 105.3, 100.5, 82.0, 82.0, 28.3, 28.3, 21.0.

¹⁹F NMR (377 MHz, CDCl₃): δ -62.5, -62.8.

HRMS (ESI-TOF): calculated for [C₁₉H₁₉F₃INNaO₂ (M + Na)]⁺: 500.0310, found: 500.0320.

N-benzyl-*N*-(2-iodo-4-methoxyphenyl)benzamide (**3eo**) and *N*-benzyl-*N*-(2-iodo-5-methoxyphenyl)benzamide (**3eo'**):



3eo and **3eo'**, 1:1

The title compound was prepared according to the general procedure for 12 h, obtained as a yellow oil, 221 mg, yield 83%.

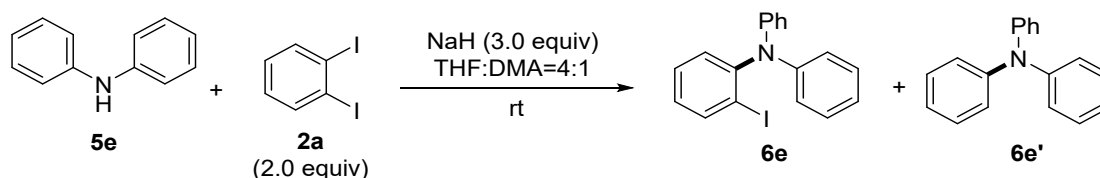
Isomer **3eo**: ¹H NMR (400 MHz, CDCl₃): δ 7.38 (t, *J* = 8.6 Hz, 2H), 7.33 – 7.26 (m, 5H), 7.25 – 7.11 (m, 4H), 6.57 – 6.48 (m, 2H), 4.22 (t, *J* = 14.8 Hz, 2H), 3.68 (s, 3H).

Isomer **3eo'**: ¹H NMR (400 MHz, CDCl₃): δ 7.59 (d, *J* = 8.6 Hz, 1H), 7.38 (t, *J* = 8.6 Hz, 2H), 7.33 – 7.26 (m, 5H), 7.25 – 7.11 (m, 3H), 6.46 (d, *J* = 8.7 Hz, 1H), 6.14 (s, 1H), 5.81 (d, *J* = 14.1 Hz, 2H), 3.44 (s, 3H).

Mixture of **3eo** and **3eo'** ¹³C NMR (101 MHz, CDCl₃): δ 170.7, 170.3, 159.9, 158.7, 145.3, 140.1, 137.4, 137.0, 137.0, 136.2, 136.0, 132.1, 129.8, 129.6, 129.6, 128.5, 128.5, 128.3, 128.2, 127.7, 127.6, 124.6, 117.8, 115.9, 114.4, 100.3, 88.3, 55.6, 55.4, 52.6, 52.4.

HRMS (ESI-TOF): calculated for [C₂₁H₁₉INO₂ (M + H)]⁺: 444.0460, found: 444.0457.

5. Table S2. Optimization of the reaction conditions for amines^a

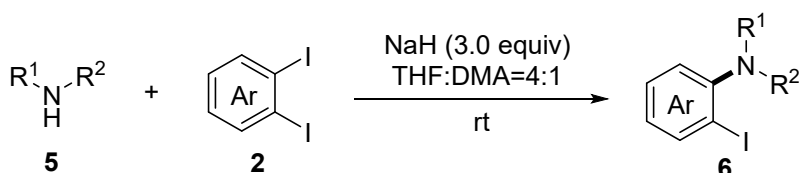


Entry	Deviation from standard conditions	Yield of 6e (%) ^b	Yield of 6e' (%) ^b
1	none	72	8
2	KH, LiH or CaH ₂ instead of NaH	0	0
3	THF instead of THF/DMA (4:1)	54	18
4	DMF instead of THF/DMA (4:1)	trace	24
5	Toluene instead of THF/DMA (4:1)	0	0
6	CH ₃ CN instead of THF/DMA (4:1)	0	0
7	DMA instead of THF/DMA (4:1)	43	23

8	2.0 equiv of NaH was used	54	trace
9	5.0 equiv of NaH was used	57	20

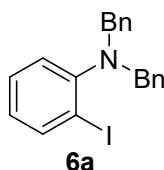
^aReaction conditions: NaH (1.8 mmol, 3.0 equiv) and **5e** (0.6 mmol, 1.0 equiv) in THF+DMA (1.0+0.5 mL) was stirred for 5 min before **2a** (1.2 mmol, 2.0 equiv, in 1.0 mL THF) was added, and the reaction was stirred at rt for 1 h. ^bIsolated yield by flash column chromatography

6. The reaction of *N*-arylation of amines



General procedure: NaH (1.8 mmol, 3.0 equiv) was weighed in a vial at room temperature, and 1.0 mL of anhydrous THF was added. Secondary amine **5** (0.6 mmol, 1.0 equiv, dissolved in 0.5 mL DMA) was added dropwise. After stirring for 5 min, *o*-diiodoarene **2** (1.2 mmol, 2.0 equiv, dissolved in 1.0 mL THF) was added. After the reaction was completed monitored by TLC, the reaction solution was slowly added to ice water to quench the reaction, extracted with ethyl acetate for 3 times, and the combined organic layers were dried over Na₂SO₄, concentrated under vacuum. The pure product **6** was obtained by column chromatography on silica gel.

N, *N*-dibenzyl-2-iodoaniline (**6a**)



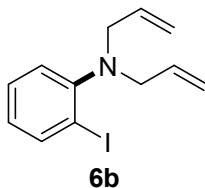
Following the general procedure, the title compound was obtained as a colorless oil, 139 mg, yield 58%.

¹H NMR (400 MHz, CDCl₃): δ 7.87 (d, *J* = 7.7 Hz, 1H), 7.33 (d, *J* = 7.0 Hz, 4H), 7.27 (t, *J* = 7.0 Hz, 4H), 7.22 (d, *J* = 8.2 Hz, 2H), 7.15 (d, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 6.75 (t, *J* = 7.4 Hz, 1H), 4.12 (s, 4H).

¹³C NMR (101 MHz, CDCl₃): δ 151.6, 140.2, 137.8, 129.0, 128.6, 128.3, 127.2, 125.9, 124.7, 99.9, 57.1.

HRMS (ESI-TOF): calculated for [C₂₀H₁₉IN (M + H)]⁺: 400.0562, found: 400.0564. The ¹H NMR and ¹³C NMR of **6a** are consistent with the reported spectra^[16, 28].

N, *N*-diallyl-2-iodoaniline (**6b**).



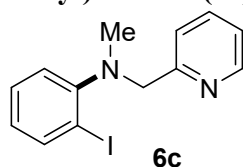
Following the general procedure, the title compound was obtained as a pale-yellow oil, 86 mg, yield 48%.

¹H NMR (400 MHz, CDCl₃): δ 7.86 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.30 – 7.25 (m, 1H), 7.02 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.79 (td, *J* = 7.8, 1.5 Hz, 1H), 5.83 (ddt, *J* = 16.4, 10.2, 6.2 Hz, 2H), 5.14 (m, 4H), 3.63 (dt, *J* = 6.2, 1.2 Hz, 4H).

¹³C NMR (101 MHz, CDCl₃): δ 152.0, 140.1, 135.0, 128.6, 125.7, 124.3, 117.9, 100.5, 56.3.

HRMS (ESI-TOF): calculated for [C₁₂H₁₅IN (M + H)]⁺: 300.0249, found: 300.0254. The ¹H NMR and ¹³C NMR of **6b** are consistent with the reported spectra^[29, 30].

iodo-*N*-methyl-*N*-(pyridin-2-ylmethyl) aniline (**6c**)



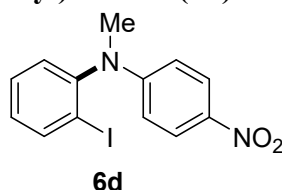
Following the general procedure, the title compound was obtained as a colorless oil, 122 mg, yield 63%.

¹H NMR (400 MHz, CDCl₃): δ 8.64 (d, *J* = 1.3 Hz, 1H), 8.54 – 8.47 (m, 1H), 7.86 (m, 2H), 7.33 – 7.28 (m, 1H), 7.26 (dd, *J* = 7.6, 4.7 Hz, 1H), 7.10 (d, *J* = 7.9 Hz, 1H), 6.88 – 6.77 (m, 1H), 4.12 (s, 2H), 2.62 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 153.5, 150.1, 148.7, 140.2, 136.5, 133.7, 129.2, 126.0, 123.4, 122.4, 98.8, 58.4, 41.8.

HRMS (ESI-TOF): calculated for [C₁₃H₁₄IN₂ (M + H)]⁺: 325.0202, found: 325.0205.

2-iodo-*N*-methyl-*N*-(4-nitrophenyl) aniline (**6d**)



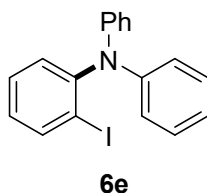
Following the general procedure, the title compound was obtained as a colorless oil, 121 mg, yield 57%.

¹H NMR (400 MHz, CDCl₃): δ 8.07 (d, *J* = 9.0 Hz, 2H), 7.99 (d, *J* = 7.9 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.26 (d, *J* = 7.5 Hz, 1H), 7.12 (t, *J* = 7.6 Hz, 1H), 6.44 (d, *J* = 8.1 Hz, 2H), 3.33 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 153.0, 148.0, 140.8, 138.4, 130.7, 129.7, 126.0, 111.6, 99.6, 39.4.

HRMS (ESI-TOF): calculated for [C₁₃H₁₂IN₂O₂ (M + H)]⁺: 354.9943, found: 354.9946.

2-iodo-*N*, *N*-diphenylaniline (**6e**)



Following the general procedure, the title compound was obtained as a white solid, 161 mg, yield 72%. Mp.216~218°C.

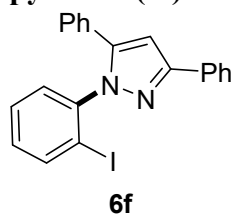
Scale-up: when **5e** (10 mmol) was employed in this reaction (rt for 5 h), 2.12 g of **6e** was obtained, 57% yield.

¹H NMR (400 MHz, CDCl₃) δ 7.91 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.34 (td, *J* = 7.9, 1.3 Hz, 1H), 7.20 (m, 5H), 6.95 (m, 7H).

¹³C NMR (101 MHz, CDCl₃) δ 149.0, 147.0, 141.1, 131.5, 129.9, 129.2, 127.7, 122.2, 122.1, 100.3.

HRMS (ESI-TOF): calculated for [C₁₈H₁₅IN (M + H)]⁺: 372.0249, found: 372.0251. The ¹H NMR and ¹³C NMR of **6e** are consistent with the reported spectra^[30].

(2-iodophenyl)-3,5-diphenyl-1*H*-pyrazole (6f**).**



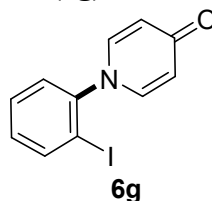
Following the general procedure, the title compound was obtained as a white solid, 114 mg, yield 45%. Mp.79~81°C.

¹H NMR (400 MHz, CDCl₃) δ 8.00 – 7.95 (m, 2H), 7.92 (d, *J* = 7.6 Hz, 1H), 7.51 – 7.36 (m, 5H), 7.29 (m, 5H), 7.13 (ddd, *J* = 8.8, 6.7, 2.5 Hz, 1H), 6.92 (s, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 152.2, 145.6, 143.2, 140.0, 133.1, 130.6, 130.0, 129.8, 129.1, 128.7, 128.5, 128.4, 128.1, 126.0, 104.0, 98.0.

HRMS (ESI-TOF): calculated for [C₂₁H₁₆IN₂ (M + H)]⁺: 423.0358, found: 423.0360.

1-(2-iodophenyl) pyridin-4(1*H*)-one (6g**).**



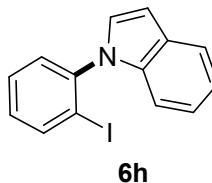
Following the general procedure, the title compound was obtained as a white solid, 96 mg, yield 54%. Mp.85~88°C.

¹H NMR (400 MHz, CDCl₃) δ 8.37 (d, *J* = 6.0 Hz, 2H), 8.00 (d, *J* = 7.9 Hz, 1H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.10 (td, *J* = 7.5, 0.9 Hz, 1H), 6.91 (d, *J* = 6.0 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 149.7, 148.6, 140.9, 136.1, 135.3, 131.1, 129.7, 121.2, 107.7.

HRMS (ESI-TOF): calculated for [C₁₁H₉INO (M + H)]⁺: 297.9729, found: 297.9729.

1-(2-iodophenyl)-1*H*-indole (6h**).**



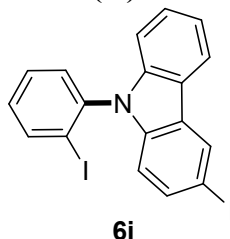
Following the general procedure, the title compound was obtained as a white solid, 90 mg, yield 47%. Mp.65~68°C.

¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 7.9 Hz, 1H), 7.72 (d, *J* = 6.0 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 4.5 Hz, 4H), 7.07 (d, *J* = 7.8 Hz, 1H), 6.72 (d, *J* = 2.2 Hz, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 142.2, 140.2, 136.8, 130.0, 129.5, 129.3, 128.7, 128.5, 122.4, 121.1, 120.4, 110.8, 103.2, 97.8.

HRMS (ESI-TOF): calculated for $[\text{C}_{14}\text{H}_{11}\text{IN} (\text{M} + \text{H})]^+$: 319.9936, found: 319.9939. The ^1H NMR and ^{13}C NMR of **6h** are consistent with the reported spectra^[31].

3-iodo-9-(2-iodophenyl)-9H-carbazole (**6i**)



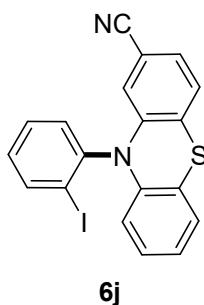
Following the general procedure, the title compound was obtained as a white solid, 270 mg, yield 91%. Mp. 102~104°C.

^1H NMR (400 MHz, CDCl_3) δ 8.47 (s, 1H), 8.16 – 8.06 (m, 2H), 7.65 (dd, $J = 8.5, 1.6$ Hz, 1H), 7.57 (td, $J = 7.6, 1.3$ Hz, 1H), 7.46 – 7.38 (m, 2H), 7.36 – 7.27 (m, 2H), 7.02 (d, $J = 8.2$ Hz, 1H), 6.82 (d, $J = 4$ Hz, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 140.9, 140.7, 139.99, 139.96, 134.4, 130.8, 130.7, 130.0, 129.4, 126.9, 125.9, 122.1, 120.7, 120.6, 112.4, 110.4, 99.2, 82.9.

HRMS (ESI-TOF): calculated for $[\text{C}_{18}\text{H}_{12}\text{I}_2\text{N} (\text{M} + \text{H})]^+$: 495.9059, found: 495.9059.

10-(2-iodophenyl)-10H-phenothiazine-4-carbonitrile (**6j**).



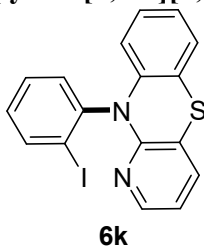
Following the general procedure, the title compound was obtained as a white solid, 238 mg, yield 93%. Mp. 190~193°C.

^1H NMR (400 MHz, CDCl_3) δ 8.15 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.64 (td, $J = 7.7, 1.4$ Hz, 1H), 7.43 (dd, $J = 7.8, 1.5$ Hz, 1H), 7.29 – 7.26 (m, 1H), 7.01 (m, 2H), 6.96 – 6.90 (m, 1H), 6.87 – 6.80 (m, 2H), 6.08 (d, $J = 1.4$ Hz, 1H), 5.99 – 5.92 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 142.5, 142.1, 141.4, 140.8, 132.6, 131.1, 130.8, 127.6, 126.9, 126.7, 126.1, 123.6, 118.9, 117.9, 117.2, 115.8, 110.2, 101.9.

HRMS (ESI-TOF): calculated for $[\text{C}_{19}\text{H}_{12}\text{IN}_2\text{S} (\text{M} + \text{H})]^+$: 426.9766, found: 426.9767.

10-(2-iodophenyl)-10H-benzo[b]pyrido[2,3-e][1,4]thiazine (**6k**).



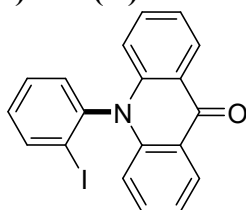
Following the general procedure, the title compound was obtained as a white solid, 135 mg, yield 56%. Mp.92~94°C.

¹H NMR (400 MHz, CDCl₃) δ 8.06 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.75 (dd, *J* = 4.9, 1.6 Hz, 1H), 7.57 (td, *J* = 7.6, 1.4 Hz, 1H), 7.47 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.24 – 7.11 (m, 2H), 7.02 – 6.94 (m, 1H), 6.91 – 6.78 (m, 2H), 6.68 (dd, *J* = 7.5, 4.9 Hz, 1H), 6.04 – 5.89 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 152.7, 145.0, 142.30, 141.7, 140.7, 133.9, 133.2, 129.8, 129.7, 127.3, 126.5, 123.3, 118.7, 118.4, 116.4, 115.3, 102.1.

HRMS (ESI-TOF): calculated for [C₁₇H₁₂IN₂S (M + H)]⁺: 402.9766, found: 402.9773.

10-(2-iodophenyl) acridin-9(10*H*)-one (6l).



6l

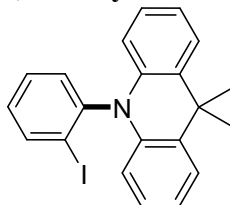
Following the general procedure, the title compound was obtained as white solid, 159 mg, yield 67%. Mp.255~258°C.

¹H NMR (400 MHz, CDCl₃) δ 8.61 (d, *J* = 8.0 Hz, 2H), 8.18 (d, *J* = 7.9 Hz, 1H), 7.69 (t, *J* = 7.6 Hz, 1H), 7.60 – 7.50 (m, 2H), 7.44 (d, *J* = 6.9 Hz, 1H), 7.35 (dd, *J* = 12.2, 4.6 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 2H), 6.62 (d, *J* = 8.6 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 178.3, 142.0, 141.6, 141.3, 133.7, 131.5, 131.3, 131.1, 127.7, 122.1, 122.0, 116.3, 100.7.

HRMS (ESI-TOF): calculated for [C₁₉H₁₃INO (M + H)]⁺: 398.0042, found: 398.0050.

10-(2-iodophenyl)-9,9-dimethyl-9,10-dihydroacridine (6m)



6m

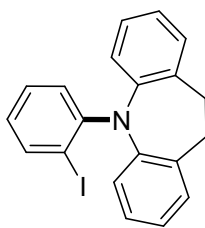
Following the general procedure, the title compound was obtained as a white solid, 185 mg, yield 75%. Mp.180~183°C.

¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 7.9 Hz, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.56 (dd, *J* = 7.2, 1.7 Hz, 2H), 7.43 (d, *J* = 7.7 Hz, 1H), 7.29 (d, *J* = 8.6 Hz, 1H), 7.13 – 6.95 (m, 4H), 6.23 – 6.08 (m, 2H), 1.89 (s, 3H), 1.75 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 143.4, 141.5, 139.2, 132.9, 130.9, 130.0, 129.8, 126.7, 126.0, 121.0, 113.6, 102.9, 36.0, 34.6, 31.10.

HRMS (ESI-TOF): calculated for [C₂₁H₁₉IN (M + H)]⁺: 412.0562, found: 412.0565.

5-(2-iodophenyl)-10,11-dihydro-5*H*-dibenzo [b, f] azepine (6n).



6n

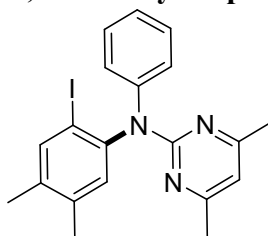
Following the general procedure, the title compound was obtained as a white solid, 81 mg, yield 34%. Mp.140~142°C.

¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 7.9 Hz, 1H), 7.62 – 7.45 (m, 2H), 7.11 (m, 3H), 6.92 (t, *J* = 7.4 Hz, 2H), 6.81 (t, *J* = 7.2 Hz, 2H), 6.43 (d, *J* = 8.4 Hz, 2H), 3.26 (s, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 148.4, 144.4, 141.8, 133.4, 133.0, 130.6, 129.9, 128.7, 126.3, 121.2, 120.5, 101.9, 37.2.

HRMS (ESI-TOF): calculated for [C₂₀H₁₇IN (M + H)]⁺: 398.0406, found: 398.0408.

***N*-(2-iodo-4,5-dimethylphenyl)-4,6-dimethyl-*N*-phenylpyrimidin-2-amine (6o)**



6o

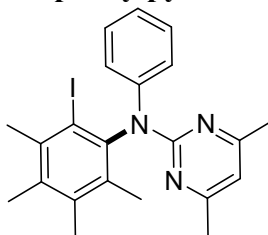
Following the general procedure, the title compound was obtained as a white solid, 200 mg, yield 78%. Mp.143~147°C.

¹H NMR (400 MHz, CDCl₃) δ 7.65 (s, 1H), 7.36 – 7.22 (m, 4H), 7.12 – 7.03 (m, 1H), 6.99 (s, 1H), 6.44 (s, 1H), 2.24 (s, 6H), 2.21 (s, 3H), 2.13 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 167.4, 161.4, 145.2, 144.2, 140.5, 138.3, 136.8, 131.7, 128.4, 125.7, 124.2, 112.0, 97.2, 24.2, 19.6, 19.1.

HRMS (ESI-TOF): calculated for [C₂₀H₂₁IN₃ (M + H)]⁺: 430.0780, found: 430.0782.

***N*-(2-iodophenyl)-4,6-dimethyl-*N*-phenylpyrimidin-2-amine (6p)**



6p

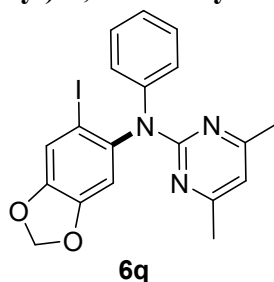
Following the general procedure, the title compound was obtained as a white solid, 178 mg, yield 74%. Mp.102~104°C.

¹H NMR (400 MHz, CDCl₃) δ 7.31 (d, *J* = 8 Hz, 2H), 7.23 – 7.16 (m, 2H), 6.98 (q, *J* = 7.6 Hz, 1H), 6.44 (s, 1H), 2.53 (s, 3H), 2.32 (s, 3H), 2.24 (s, 6H), 2.18 (s, 3H), 2.07 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 167.3, 160.9, 142.9, 142.6, 138.2, 136.4, 135.0, 134.6, 128.0, 123.7, 122.9, 112.1, 108.3, 27.7, 24.2, 18.5, 17.1, 17.0.

HRMS (ESI-TOF): calculated for [C₂₂H₂₅IN₃ (M + H)]⁺: 458.1093, found: 458.1093.

***N*-(6-iodobenzo[*d*][1,3]dioxol-5-yl)-4,6-dimethyl-*N*-phenylpyrimidin-2-amine (6q).**



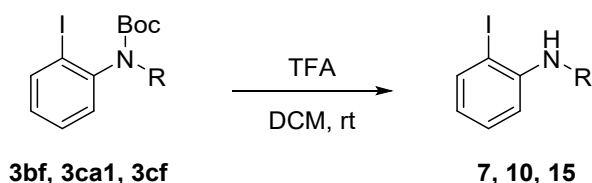
Following the general procedure, the title compound was obtained as a white solid, 155 mg, yield 58%. Mp.132~134°C.

¹H NMR (400 MHz, CDCl₃) δ 7.38 – 7.26 (m, 5H), 7.15 – 7.07 (m, 1H), 6.77 (s, 1H), 6.49 (s, 1H), 5.99 (s, 2H), 2.29 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 167.4, 161.4, 149.1, 146.9, 143.8, 141.3, 128.9, 128.5, 125.4, 124.3, 118.3, 112.2, 111.3, 102.2, 89.9, 24.2.

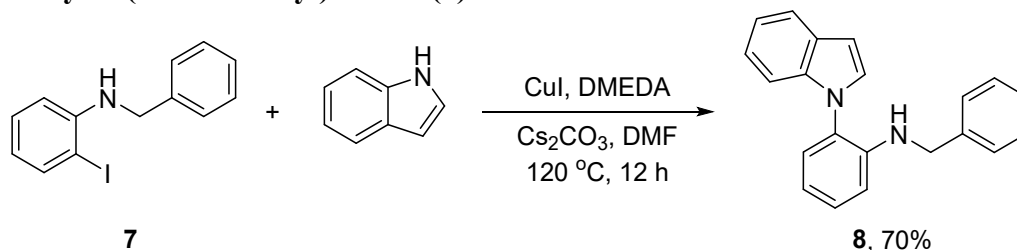
HRMS (ESI-TOF): calculated for [C₁₉H₁₇IN₃O₂ (M + H)]⁺: 446.0365, found: 446.0367.

7. Synthetic applications



General procedure for preparation of **7**, **10** and **15**: To a solution of **3bf**, **3ca1** or **3cf** (1.0 equiv) in DCM (0.33 M) was added CF₃CO₂H (3.0 equiv) at room temperature. After being stirred at room temperature for 1-2 h, the reaction mixture was cooled to 0 °C and carefully neutralized (pH 7-8) with sat. aq. NaHCO₃. And then, the mixture was poured into water and the product was extracted with CH₂Cl₂ for three times, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by column chromatography on silica gel to give the corresponding product in 89-96% yields.

N-benzyl-2-(1*H*-indol-1-yl)aniline (**8**)^[32]:



To a solution of *N*-benzyl-2-iodoaniline **7** (86.5 mg, 0.28 mmol, 1.4 equiv) and DMEDA (*N,N'*-dimethylethylenediamine, 25 mg, 0.28 mmol, 1.4 equiv) in anhydrous DMF (0.5 mL) was added CuI (12 mg, 0.06 mmol, 30 mol%), Cs₂CO₃ (130 mg, 0.4 mmol, 2.0 equiv) and indole (23.4 mg, 0.2 mmol, 1.0 equiv). The reaction mixture was stirred at 120 °C under nitrogen for 12 hours. The reaction was cooled to rt and filtered through diatomite, and the filtrate was added water (10 mL), extracted with EtOAc for three times. The combined organic layer was washed with water for two times to remove DMF, and dried over anhydrous Na₂SO₄. The solvent was removed under

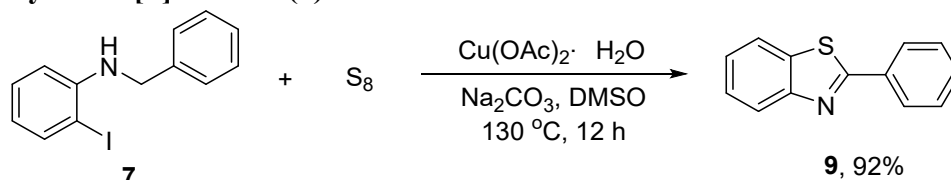
vacuum and the residue was purified by column chromatography to afford the product **8** as a yellow oil, 42 mg, yield 70%.

¹H NMR (400 MHz, CDCl₃): δ 7.72 – 7.66 (m, 1H), 7.30 – 7.25 (m, 3H), 7.24 – 7.14 (m, 8H), 6.77 (ddd, *J* = 14.6, 7.9, 1.2 Hz, 2H), 6.70 (d, *J* = 3.1 Hz, 1H), 4.30 (s, 2H), 4.08 (br, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 144.5, 139.0, 136.7, 132.6, 129.5, 128.8, 128.7, 128.6, 127.2, 127.0, 124.7, 122.3, 121.0, 120.3, 117.0, 111.9, 110.9, 103.5, 47.6.

HRMS (ESI-TOF): calculated for [C₂₁H₁₉N₂ (M + H)]⁺: 299.1548, found: 299.1550.

2-Phenylbenzo[d]thiazole (**9**)^[33]:



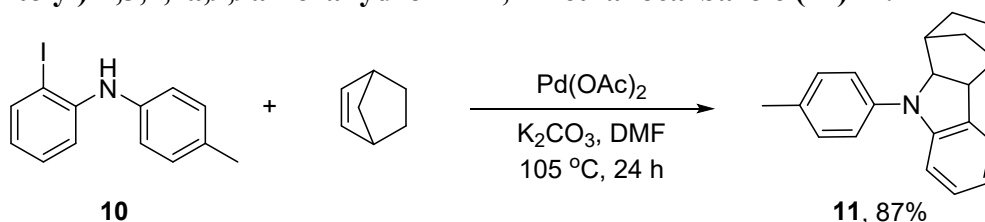
To a solution of *N*-benzyl-2-iodoaniline **7** (123.7 mg, 0.4 mmol, 1.0 equiv) and S₈ (51.2 mg, 1.6 mmol, 4.0 equiv) in anhydrous DMSO (3.0 mL) was added Cu(OAc)₂·H₂O (16 mg, 0.08 mmol, 20 mol%) and Na₂CO₃ (85 mg, 0.8 mmol, 2.0 equiv). Then the reaction mixture was stirred at 130 °C under nitrogen for 12 hours. The reaction was cooled to rt and filtered through diatomite, and the filtrate was added water (10 mL), extracted with EtOAc for three times. The combined organic layer was washed with water to remove DMSO, and dried over anhydrous Na₂SO₄. The solvent was removed under vacuum and the residue was purified by column chromatography to afford the product **9** as a yellow solid, 78 mg, yield 92%. Melting Point: 109–112 °C.

¹H NMR (400 MHz, CDCl₃): δ 8.09 (m, 3H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.51 – 7.44 (m, 4H), 7.37 (dd, *J* = 11.2, 4.0 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 168.1, 154.2, 135.1, 133.7, 131.0, 129.1, 127.6, 126.4, 125.2, 123.3, 121.7.

HRMS (ESI-TOF): calculated for [C₁₃H₁₀NS (M + H)]⁺: 212.0534, found: 212.0536. The ¹H NMR and ¹³C NMR of **6a** are consistent with the reported spectra^[33].

9-(*p*-tolyl)-2,3,4,4a,9,9a-hexahydro-1*H*-1,4-methanocarbazole (**11**)^[34]:



To a solution of 2-iodo-*N*-(*p*-tolyl)aniline **10** (93 mg, 0.3 mmol, 1.0 equiv) and norbornene (28.5 mg, 0.3 mmol, 1.0 equiv) in anhydrous DMF (2.5 mL) was added Pd(OAc)₂ (2.7 mg, 0.012 mmol, 4 mol%) and K₂CO₃ (99.5 mg, 0.72 mmol, 2.4 equiv). The reaction mixture was stirred at 105 °C under nitrogen for 24 hours. Then the reaction was quenched with water, extracted with EtOAc for three times. The combined organic layer was washed with water to remove DMF, and dried over anhydrous Na₂SO₄. The solvent was removed under vacuum and the residue was purified by column chromatography to afford the product **11** as a yellow oil, 72 mg, yield 87%.

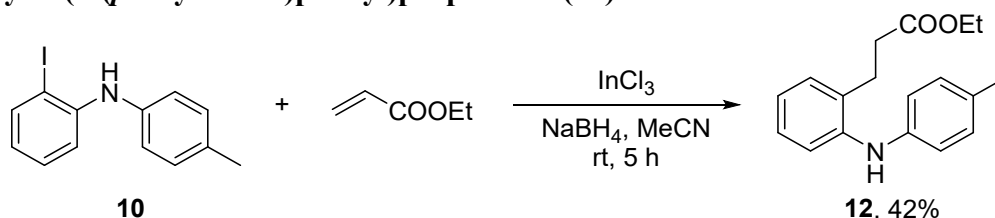
¹H NMR (400 MHz, CDCl₃): δ 7.21 – 7.16 (m, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 7.2 Hz, 1H), 6.98 (t, *J* = 7.7 Hz, 1H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.64 (t, *J* = 7.3 Hz, 1H), 4.20 (d, *J* = 8.3 Hz, 1H), 3.28 (d, *J* = 8.3 Hz, 1H), 2.44 (d, *J* = 1.8 Hz, 1H), 2.33

(m, 1H), 2.31 (s, 3H), 1.58 – 1.48 (m, 3H), 1.37 (t, $J = 8.9$ Hz, 1H), 1.28 – 1.18 (m, 1H), 1.09 (dd, $J = 10.3, 1.4$ Hz, 1H).

^{13}C NMR (101 MHz, CDCl_3): δ 149.4, 141.2, 133.4, 131.3, 129.8, 127.3, 124.9, 120.1, 118.0, 107.7, 71.3, 50.5, 43.5, 41.1, 32.4, 28.6, 25.3, 20.8.

HRMS (ESI-TOF): calculated for $[\text{C}_{20}\text{H}_{22}\text{N} (\text{M} + \text{H})]^+$: 276.1752, found: 276.1756.

Ethyl 3-(2-(*p*-tolylamino)phenyl)propanoate (**12**)^[35]:



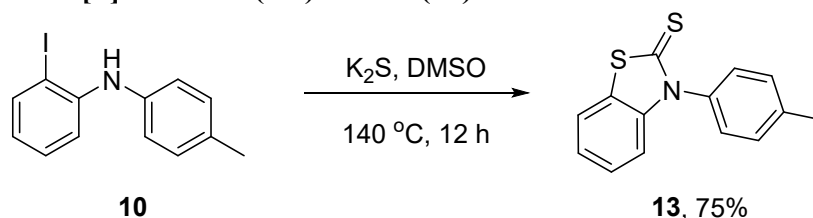
To a solution of 2-iodo-*N*-(*p*-tolyl)aniline **10** (93 mg, 0.3 mmol, 1.0 equiv) and ethyl acrylate (150 mg, 1.5 mmol, 5.0 equiv) in anhydrous MeCN (1.2 mL) was added InCl_3 (7 mg, 0.03 mmol, 0.1 equiv) and NaBH_4 (14 mg, 0.36 mmol, 1.2 equiv). The reaction mixture was stirred at room temperature for 5 hours. The reaction mixture was quenched with water, extracted with EtOAc and dried over anhydrous Na_2SO_4 . The solvents were removed under vacuum and the residue was purified by column chromatography to afford the product **12** as a yellow oil, 35.7 mg, yield 42%.

^1H NMR (400 MHz, CDCl_3): 7.95 (dd, $J = 7.8, 1.4$ Hz, 1H), 7.39 (td, $J = 7.8, 1.5$ Hz, 1H), 7.19 (dd, $J = 7.9, 1.6$ Hz, 1H), 7.00 (m, 3H), 6.49 – 6.43 (m, 2H), 4.11 (q, $J = 7.1$ Hz, 2H), 3.98 – 3.88 (m, 2H), 2.74 – 2.68 (m, 2H), 2.24 (s, 3H), 1.23 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3): δ 172.0, 148.6, 145.2, 140.6, 130.8, 129.9, 129.8, 128.3, 127.4, 114.0, 102.3, 60.7, 47.5, 32.8, 20.4, 14.2.

HRMS (ESI-TOF): calculated for $[\text{C}_{18}\text{H}_{22}\text{NO}_2 (\text{M} + \text{H})]^+$: 284.1651, found: 284.1655.

3-(*P*-tolyl)benzo[*d*]thiazole-2(3*H*)-thione (**13**)^[36]:



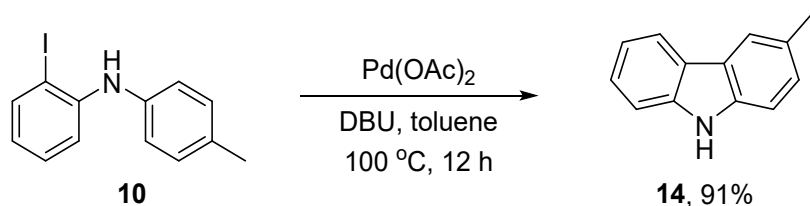
To a solution of 2-iodo-*N*-(*p*-tolyl)aniline **10** (93 mg, 0.3 mmol, 1.0 equiv) in DMSO (2.0 mL) was added K_2S (132.3 mg, 1.2 mmol, 4.0 equiv). The reaction mixture was stirred at 140 °C under nitrogen for 12 hours. Water was added to quench the reaction, extracted with EtOAc and dried over anhydrous Na_2SO_4 . The solvents were removed under vacuum and the residue was purified by column chromatography to afford the product **13** as a yellow solid, 58 mg, yield 75%. Melting Point: 102-105 °C.

^1H NMR (400 MHz, CDCl_3): δ 7.46 (dd, $J = 7.3, 1.6$ Hz, 1H), 7.37 (d, $J = 8.1$ Hz, 2H), 7.28 (d, $J = 8.2$ Hz, 2H), 7.19 (pd, $J = 7.5, 3.8$ Hz, 2H), 6.82 – 6.75 (m, 1H), 2.44 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3): δ 169.9, 139.5, 138.5, 132.2, 130.7, 127.7, 126.3, 123.6, 122.6, 119.2, 111.9, 21.4.

HRMS (ESI-TOF): calculated for $[\text{C}_{14}\text{H}_{12}\text{NS}_2 (\text{M} + \text{H})]^+$: 258.0411, found: 258.0413.

3-Methyl-9*H*-carbazole (**14**)^[37]:



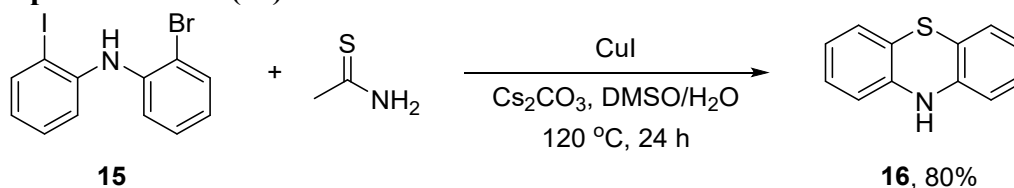
To a solution of 2-iodo-*N*-(*p*-tolyl) aniline **10** (61.8 mg, 0.2 mmol, 1.0 equiv) in anhydrous toluene (1.0 mL) was added Pd(OAc)₂ (11 mg, 0.05 mmol, 25 mol%) and DBU (182.6 mg, 1.2 mmol, 6.0 equiv). The reaction was stirred at 100 °C under nitrogen for 12 hours before water was added to quench the reaction. The mixture was extracted with EtOAc for three times. The combined organic layer was dried over anhydrous Na₂SO₄. The solvent was removed under vacuum and the residue was purified by column chromatography to afford the product **14** as a white solid, 33 mg, yield 91%. Melting Point: 205-208 °C.

¹H NMR (400 MHz, CDCl₃): δ 8.03 (dd, *J* = 7.8, 0.6 Hz, 1H), 7.91 (br, 1H), 7.87 (d, *J* = 0.7 Hz, 1H), 7.42 – 7.34 (m, 2H), 7.30 (d, *J* = 8.2 Hz, 1H), 7.24 – 7.17 (m, 2H), 2.52 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 139.9, 137.8, 128.8, 127.3, 125.7, 123.6, 123.3, 120.3, 120.3, 119.3, 110.6, 110.3, 21.5.

HRMS (ESI-TOF): calculated for [C₁₃H₁₂N (M + H)]⁺: 182.0970, found: 182.0977. The ¹H NMR and ¹³C NMR of **6a** are consistent with the reported spectra^[37].

10*H*-phenothiazine (**16**)^[38]:



A flask was charged with CuI (19 mg, 0.1 mmol, 10 mol%), CH₃CSNH₂ (225 mg, 3.0 mmol, 3.0 equiv), Cs₂CO₃ (978 mg, 3.0 mmol, 3.0 equiv). 2-Bromo-*N*-(2-iodophenyl)aniline **15** (374 mg, 1.0 mmol, 1.0 equiv) in DMSO/H₂O (0.5 mL/0.25 mL) was added. The reaction mixture was stirred at 120 °C under nitrogen for 24 hours. The reaction was cooled to rt and filtered through diatomite, and the filtrate was added water (10 mL), extracted with EtOAc for three times. The combined organic layer was washed with water to remove DMSO, and dried over anhydrous Na₂SO₄. The solvent was removed under vacuum and the residue was purified by column chromatography to afford the product **16** as a yellowish solid, 159 mg, yield 80%. Melting Point: 180-184 °C.

¹H NMR (400 MHz, CDCl₃): δ 6.97 (m, 4H), 6.81 (t, *J* = 7.5 Hz, 2H), 6.53 (d, *J* = 7.9 Hz, 2H), 5.80 (br, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 141.7, 127.4, 126.9, 122.7, 118.4, 114.5.

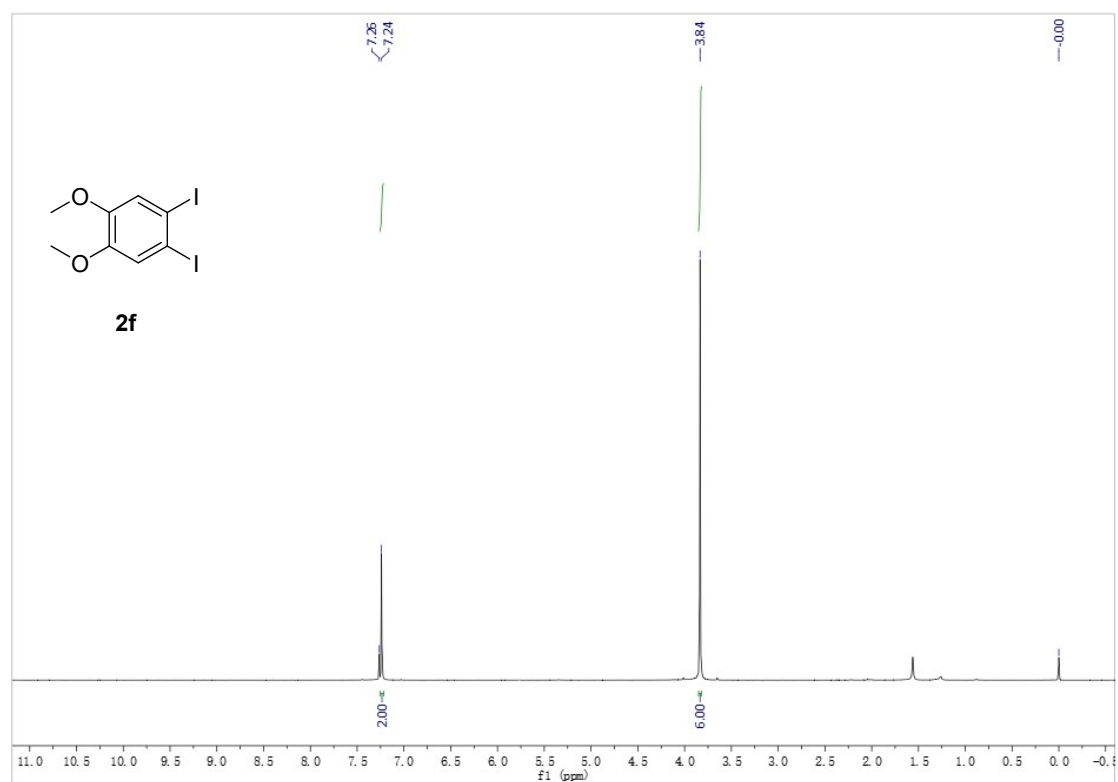
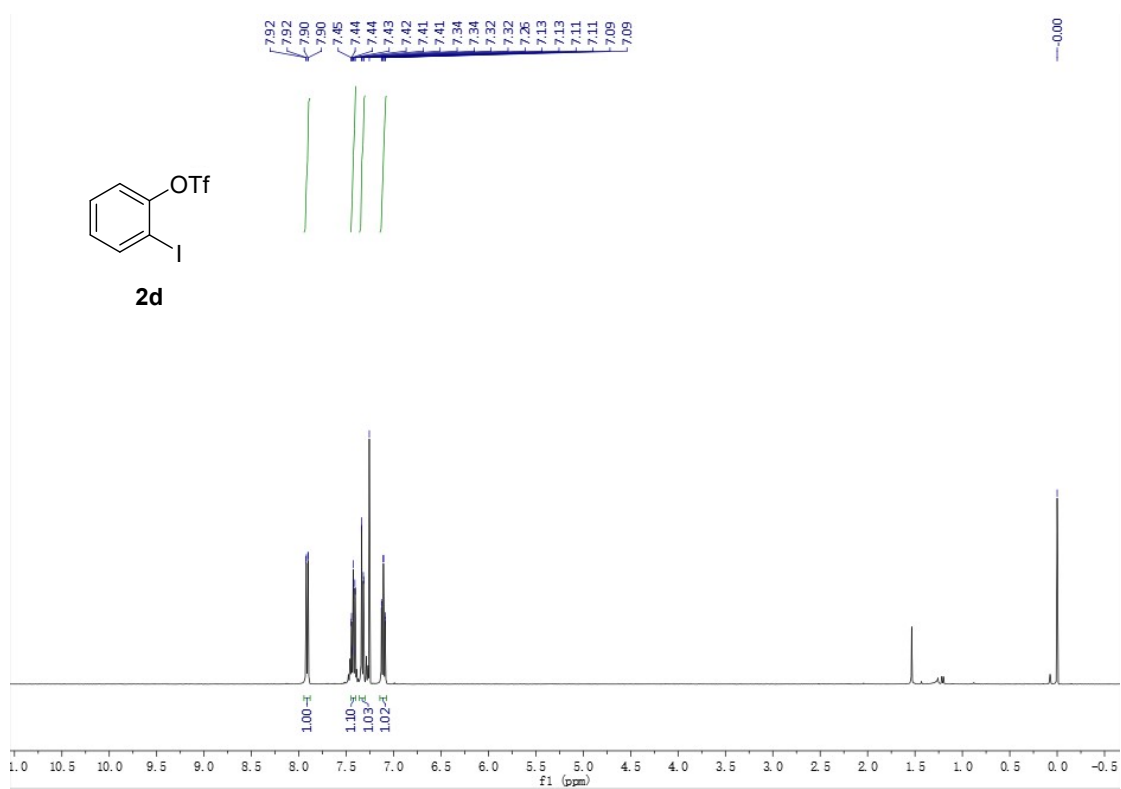
HRMS (ESI-TOF): calculated for [C₁₂H₁₀NS (M + H)]⁺: 200.0534, found: 200.0531.

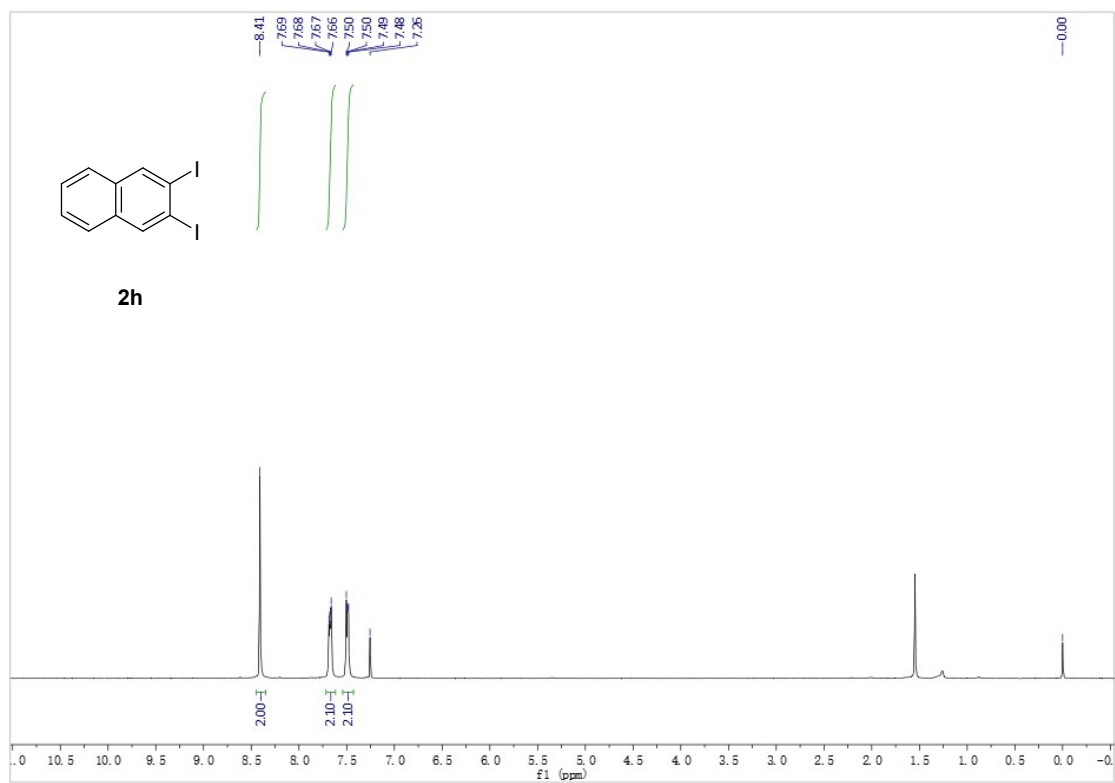
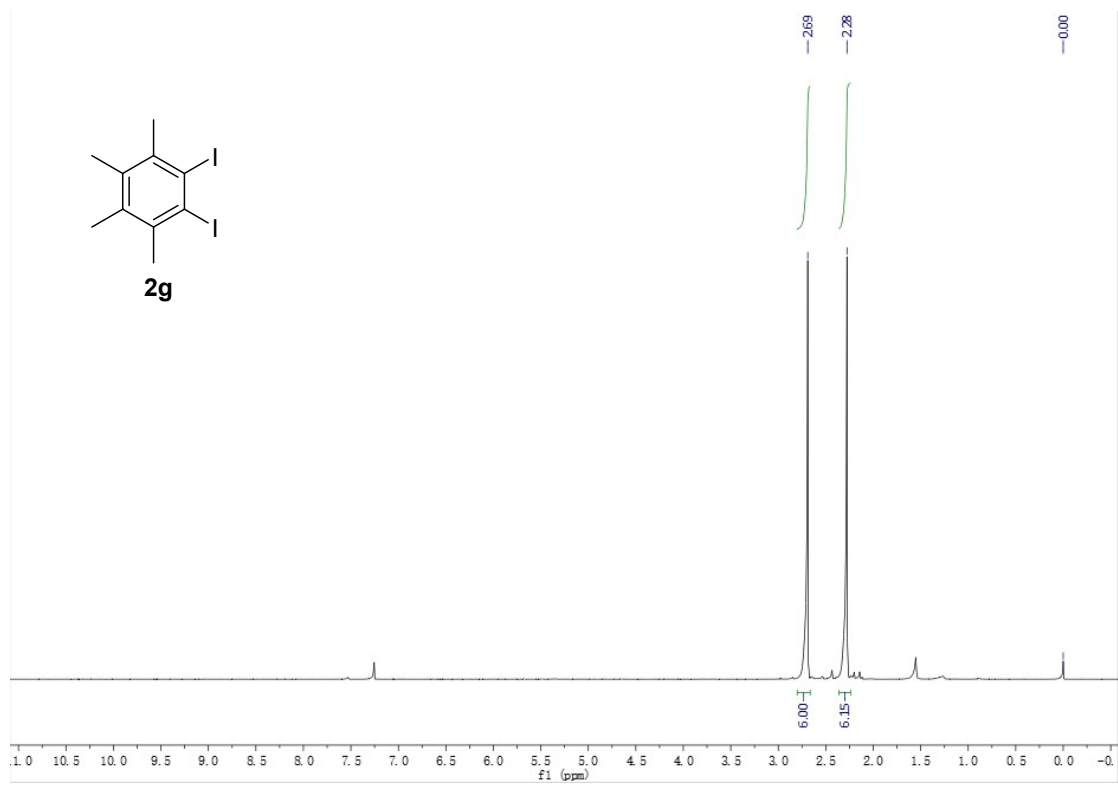
8. Supplementary Reference

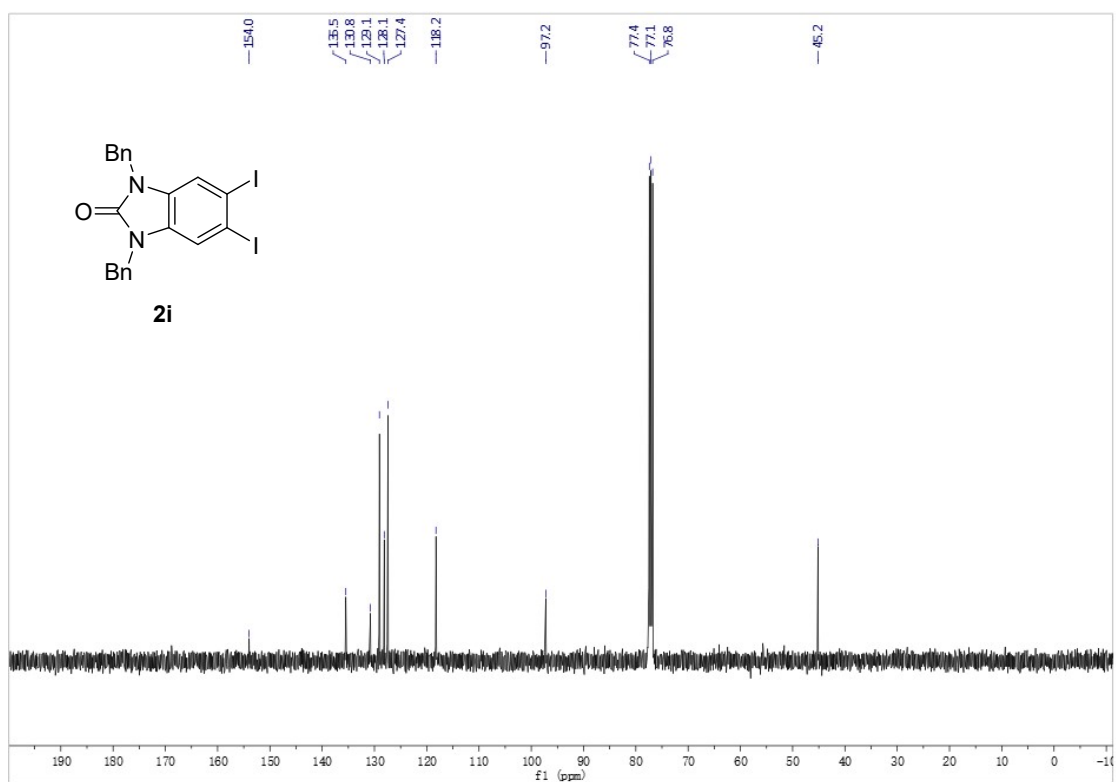
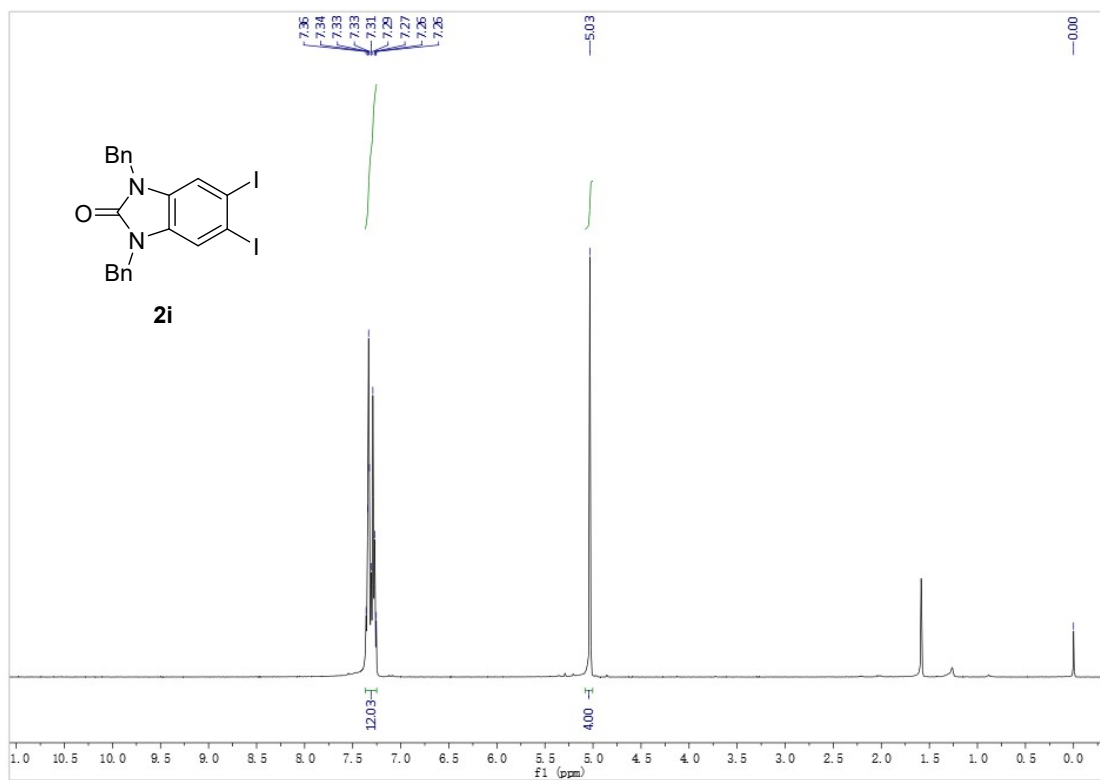
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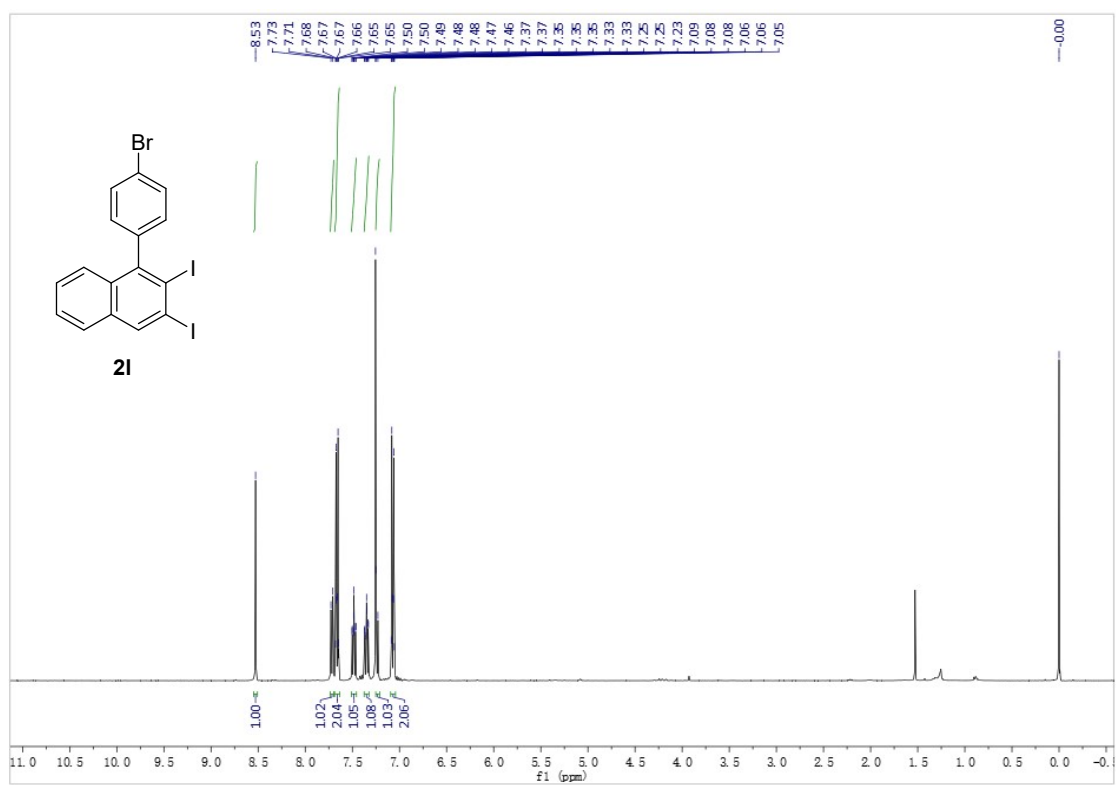
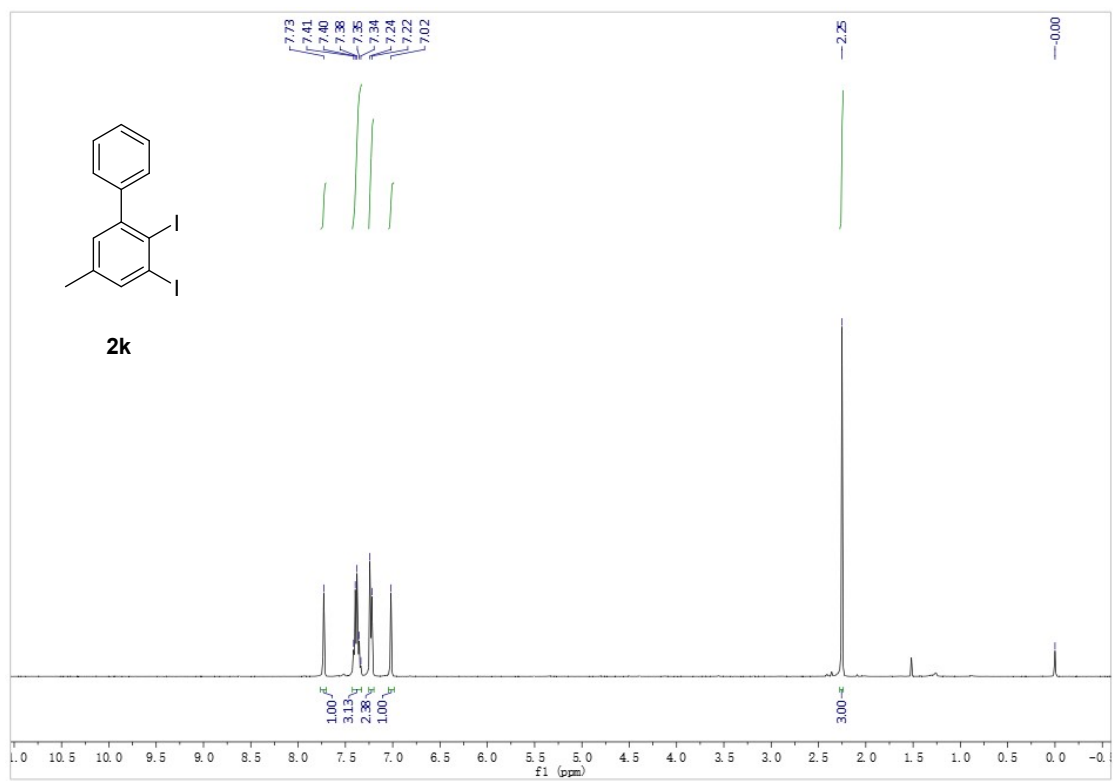
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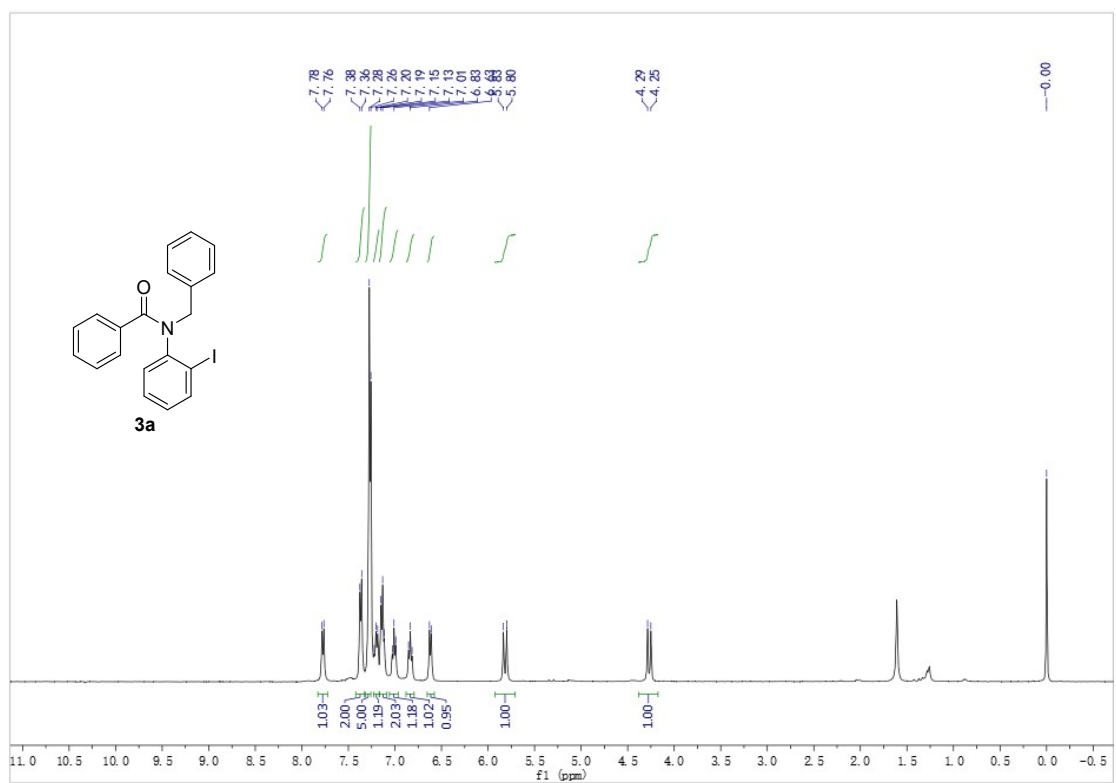
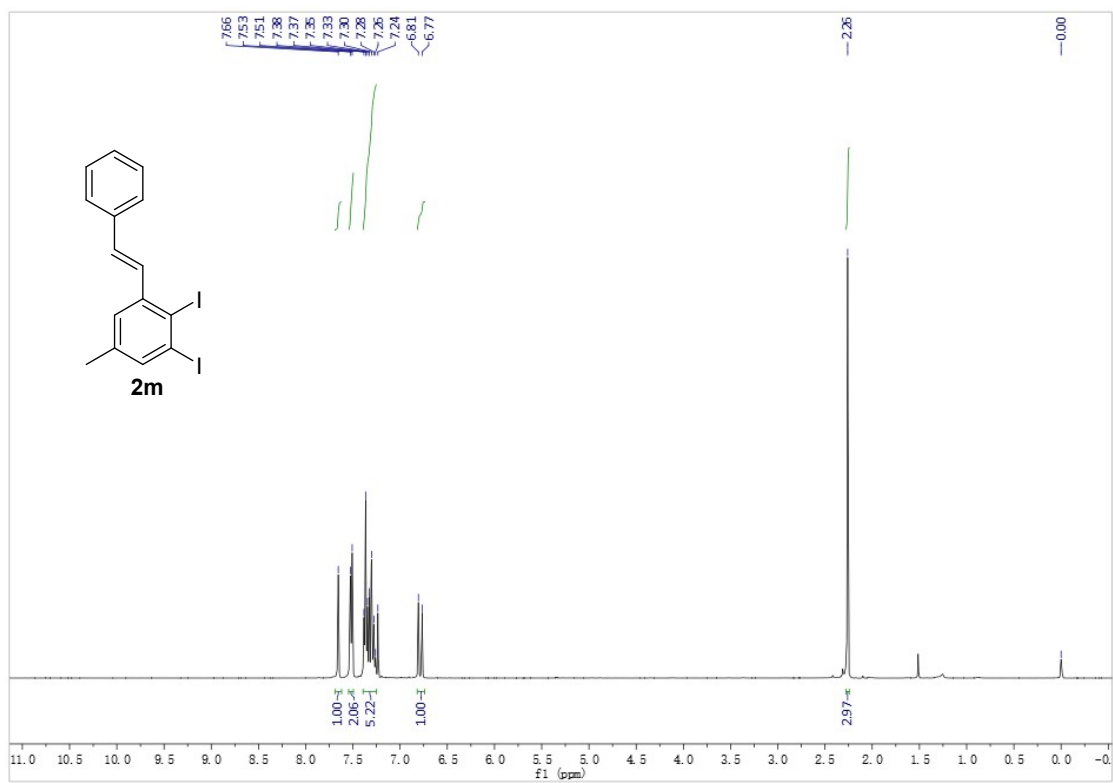
9. NMR spectra



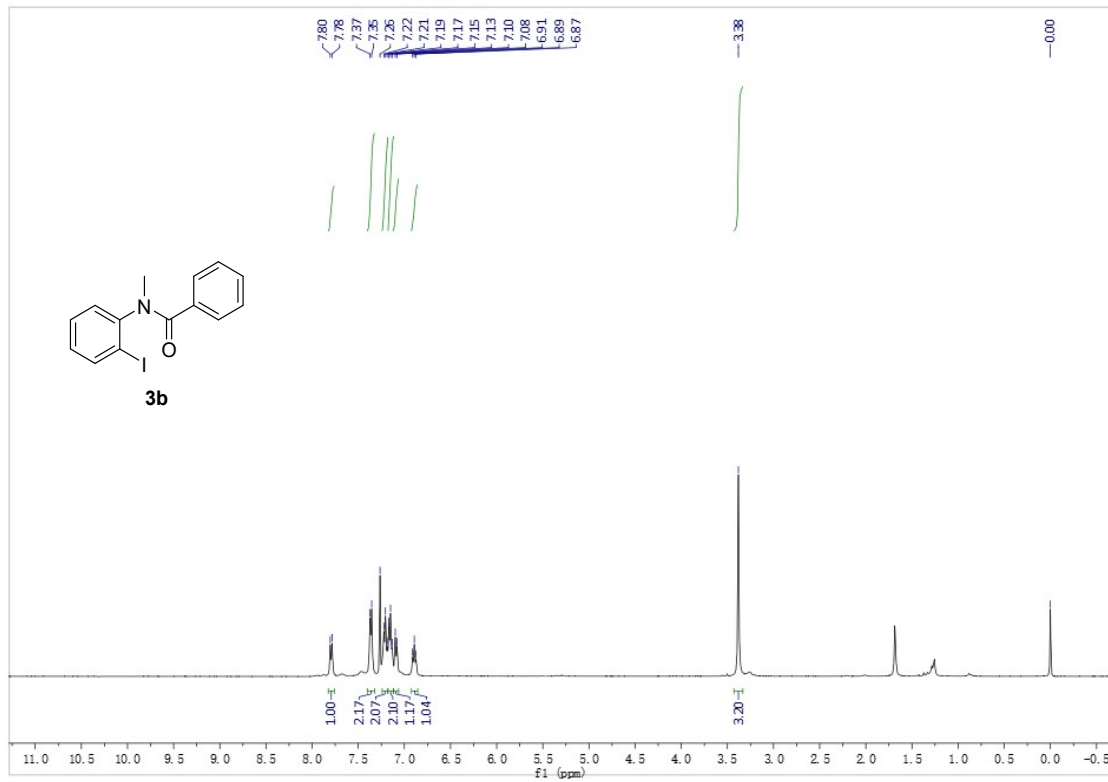
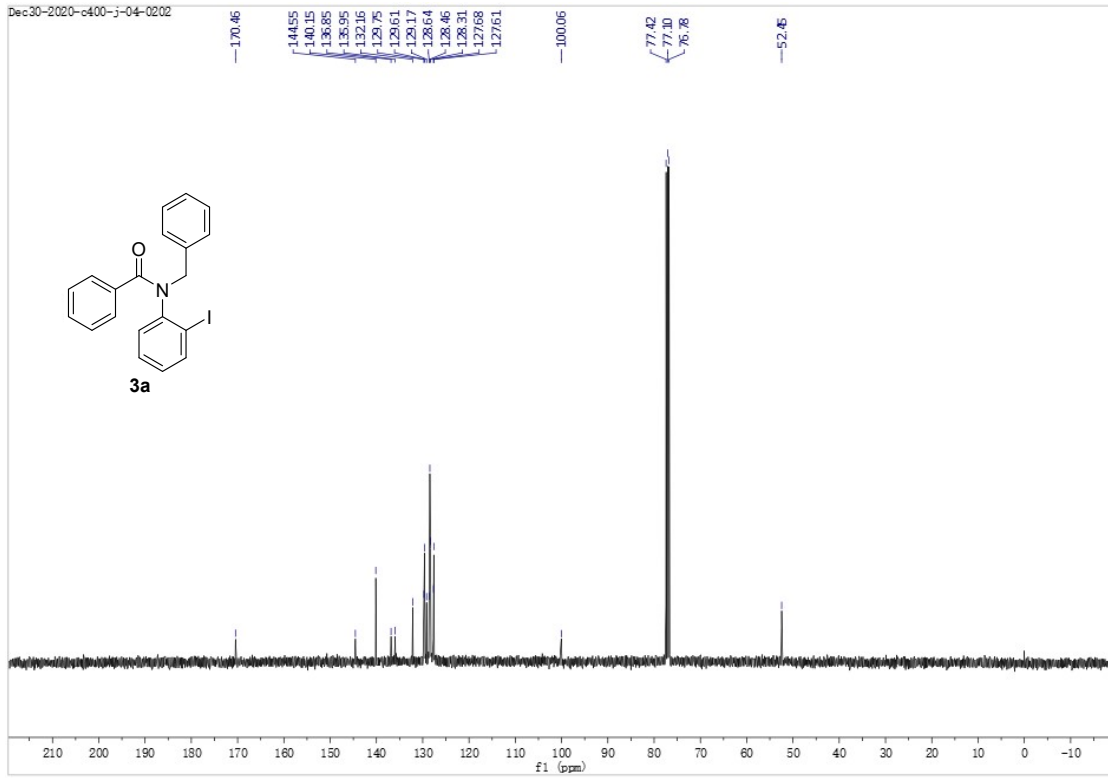


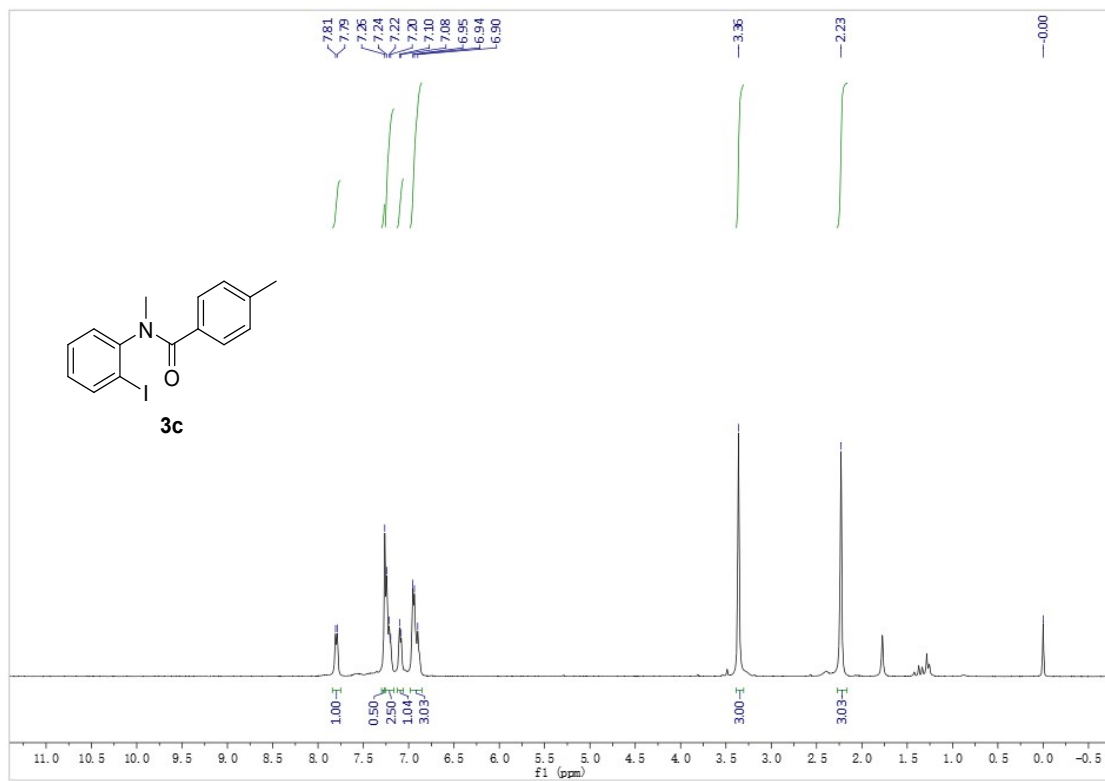
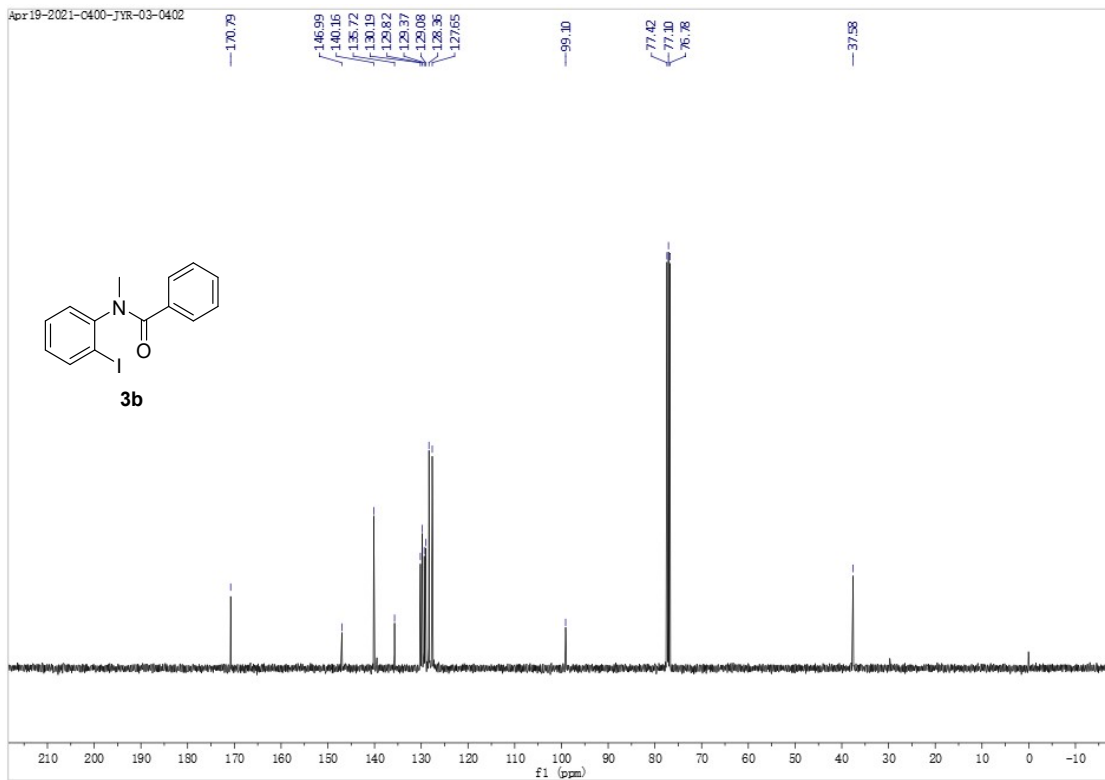


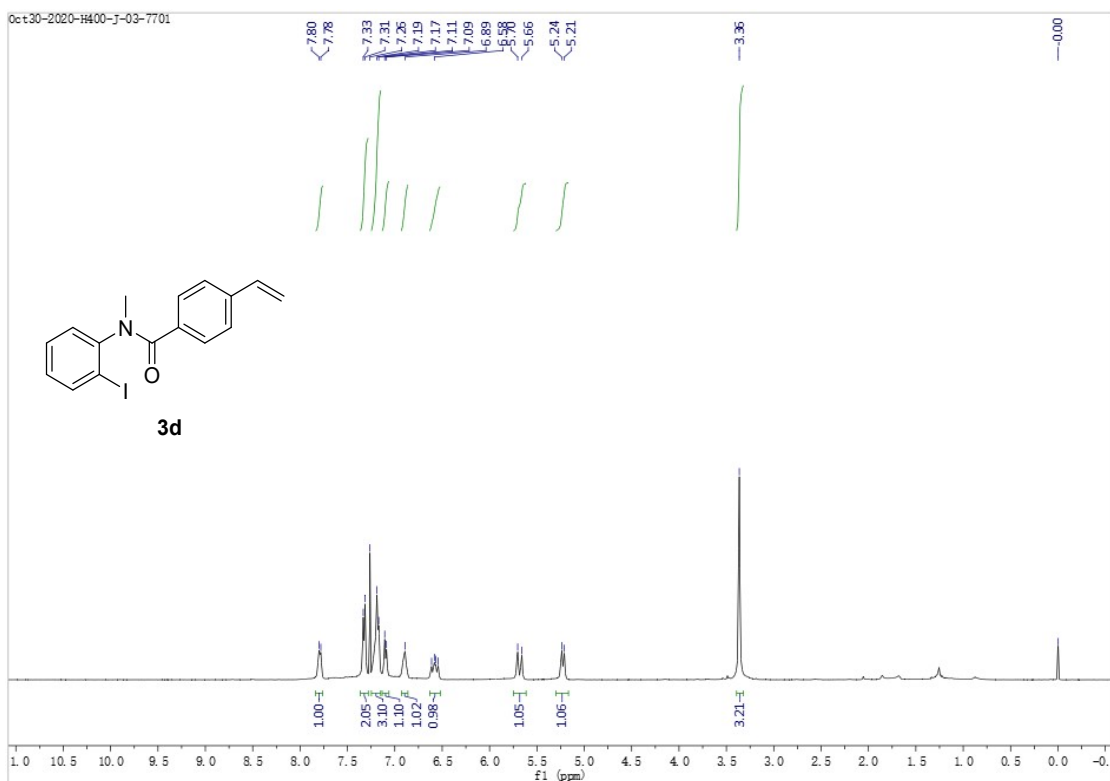
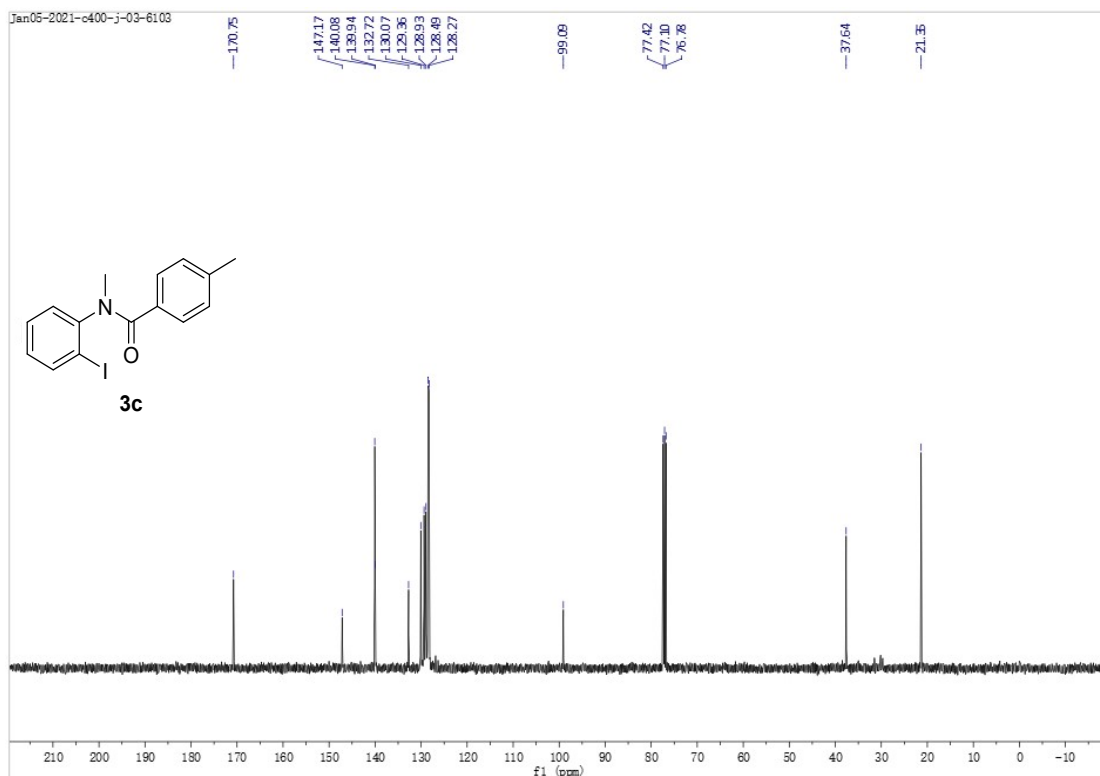


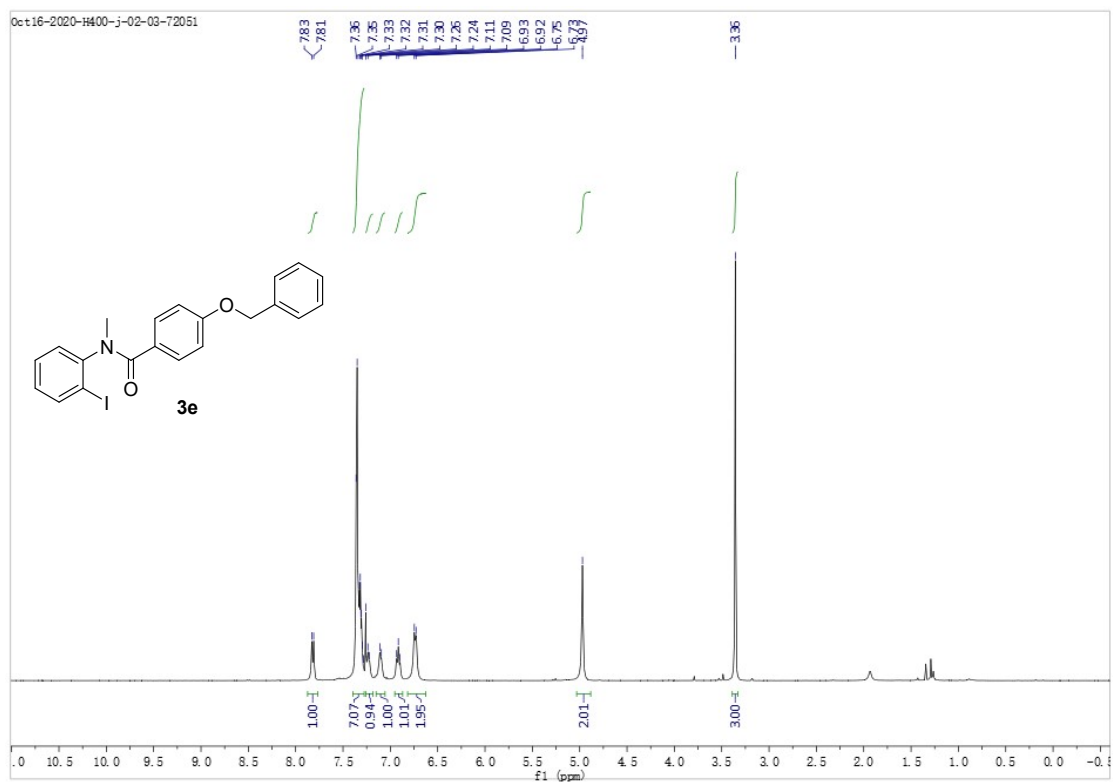
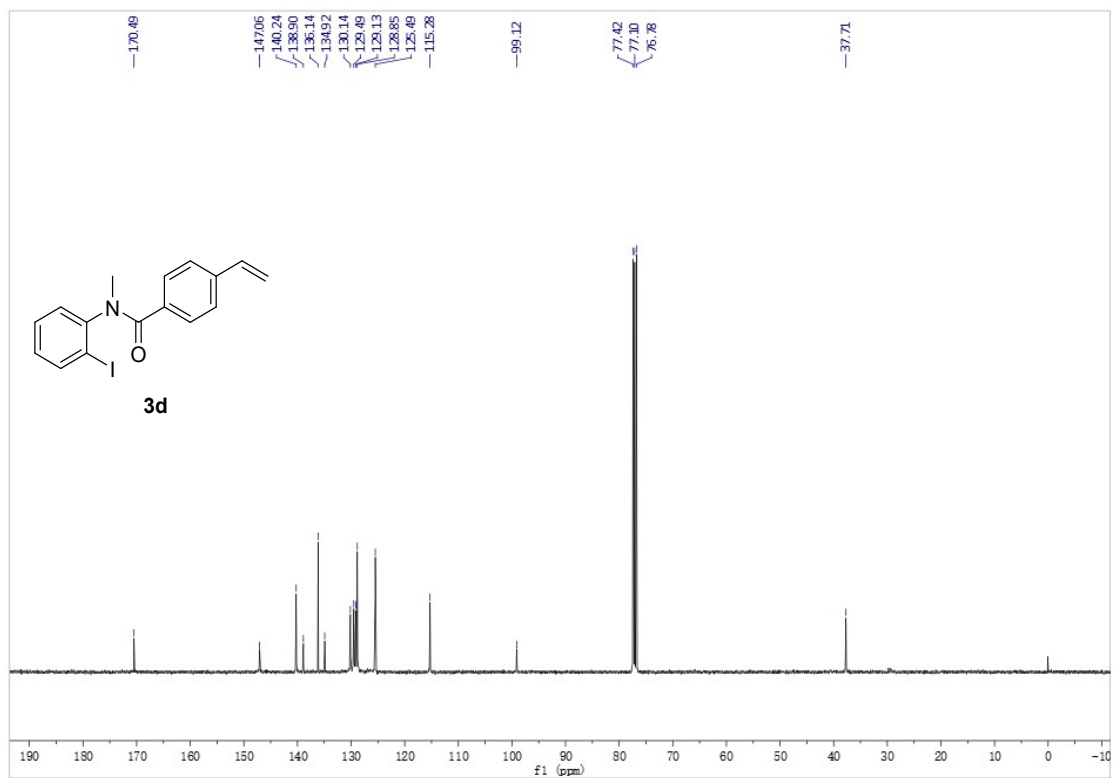


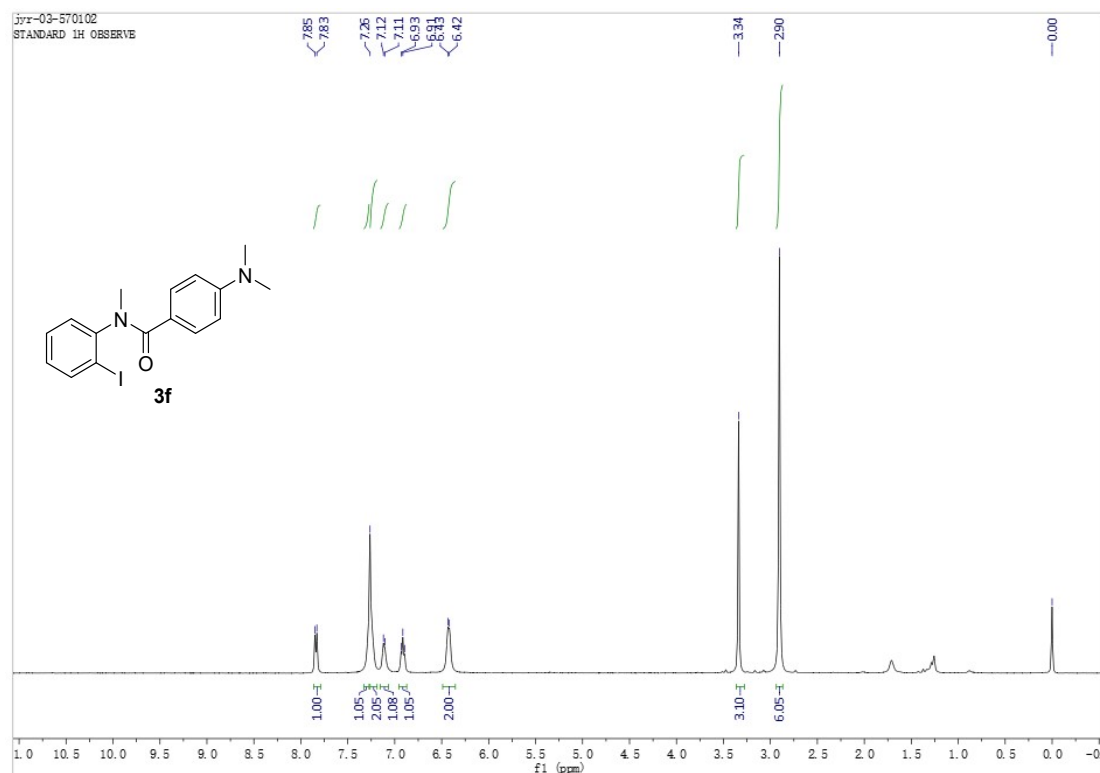
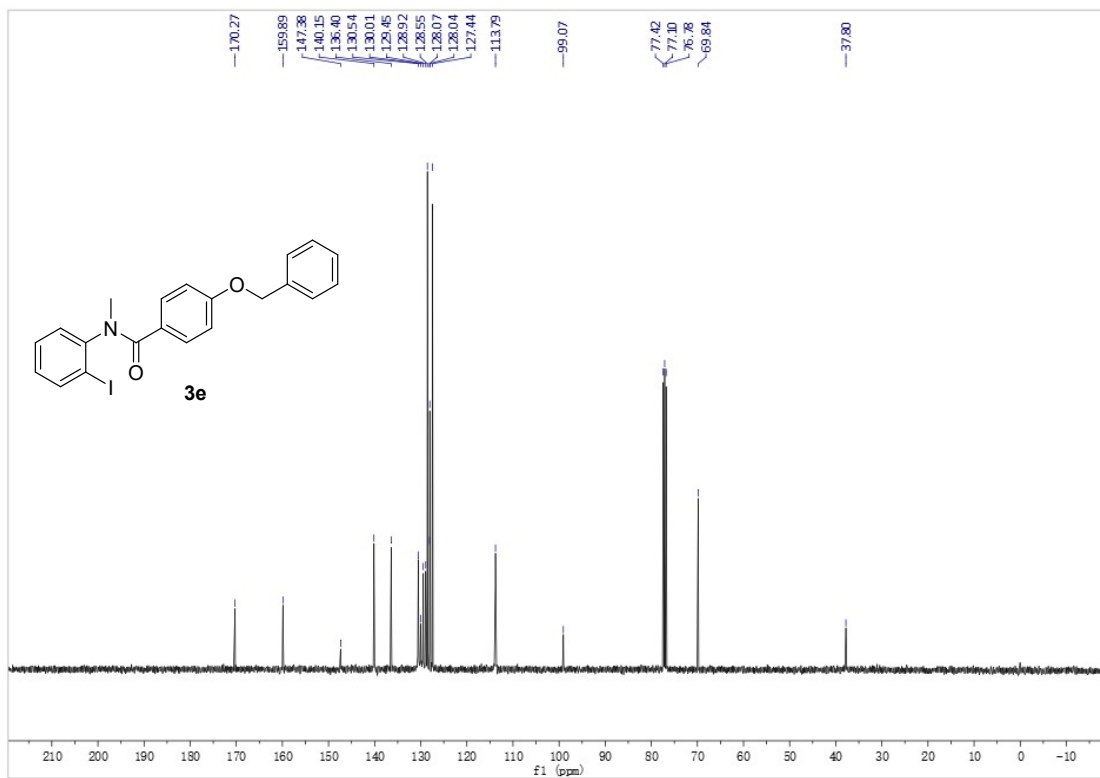
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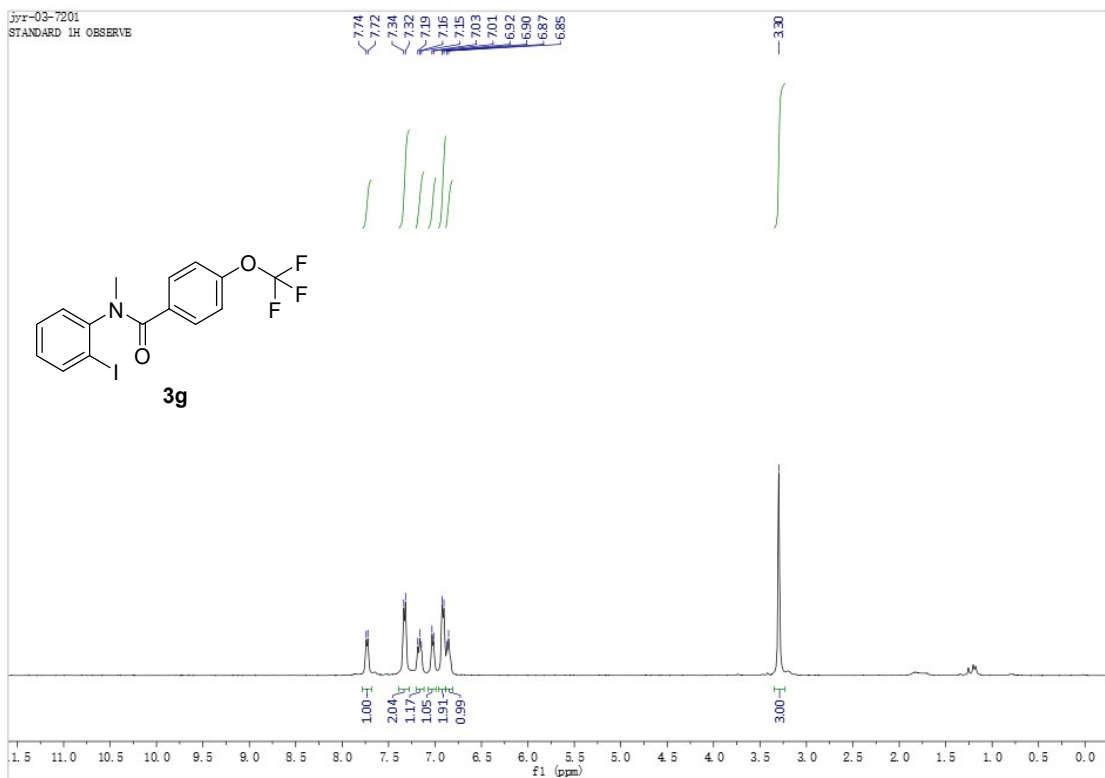
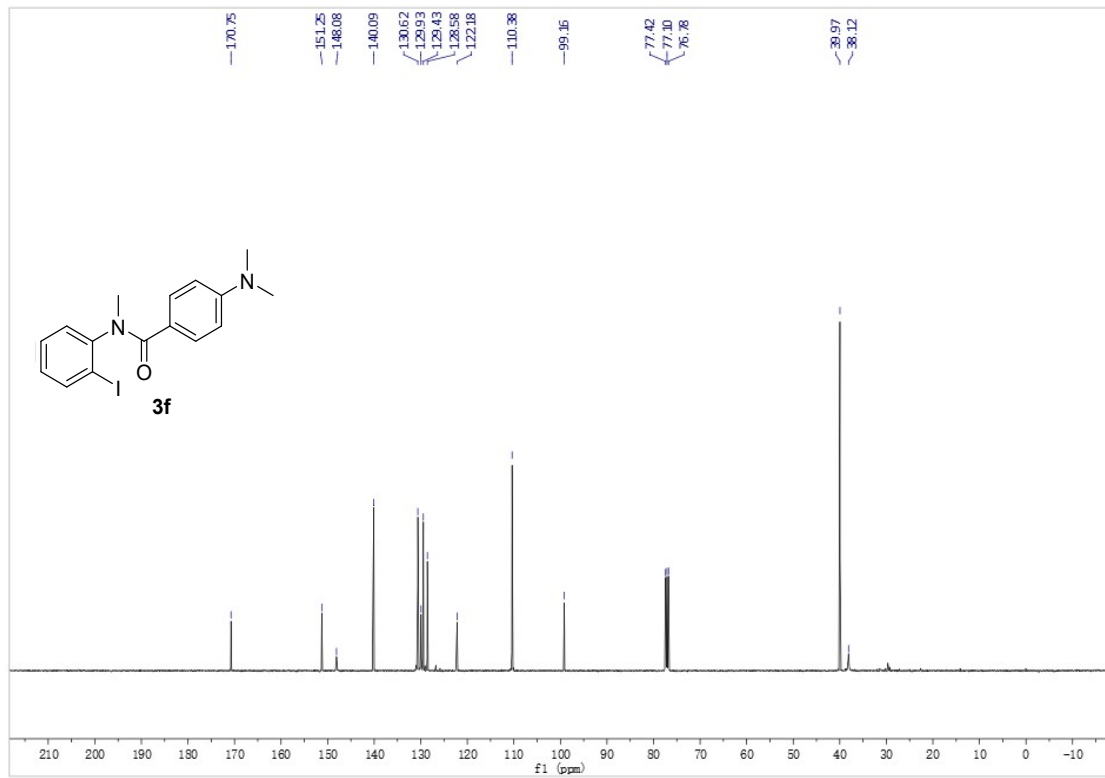


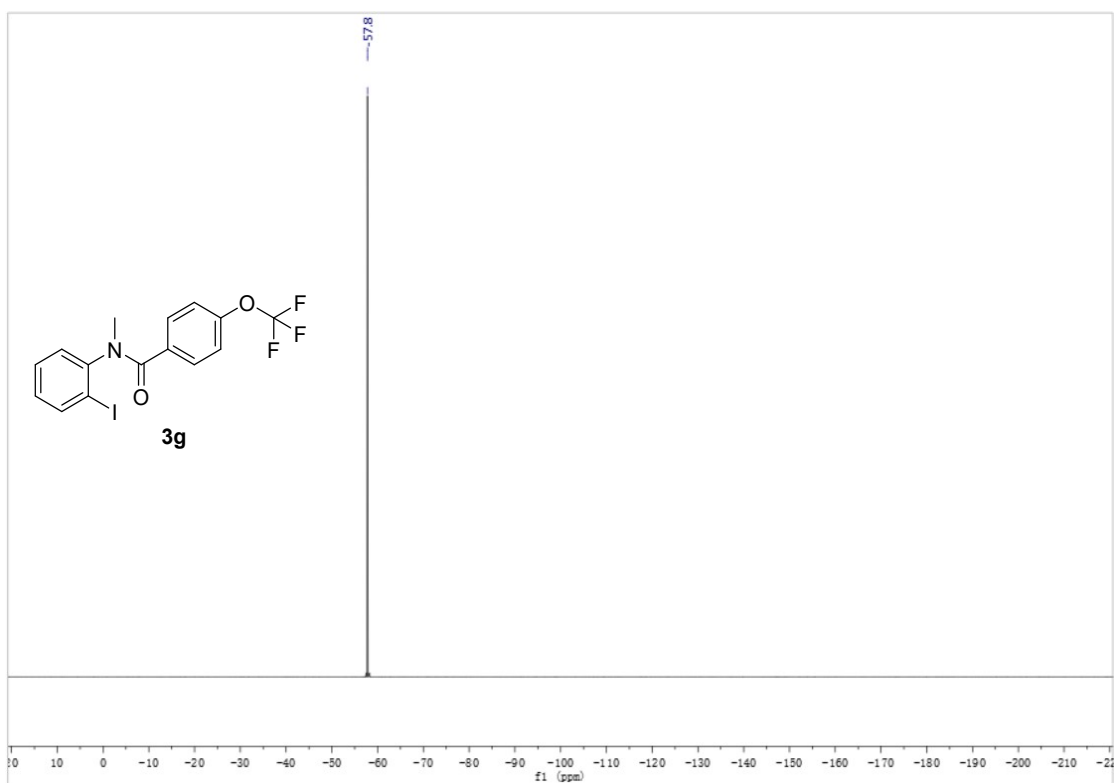
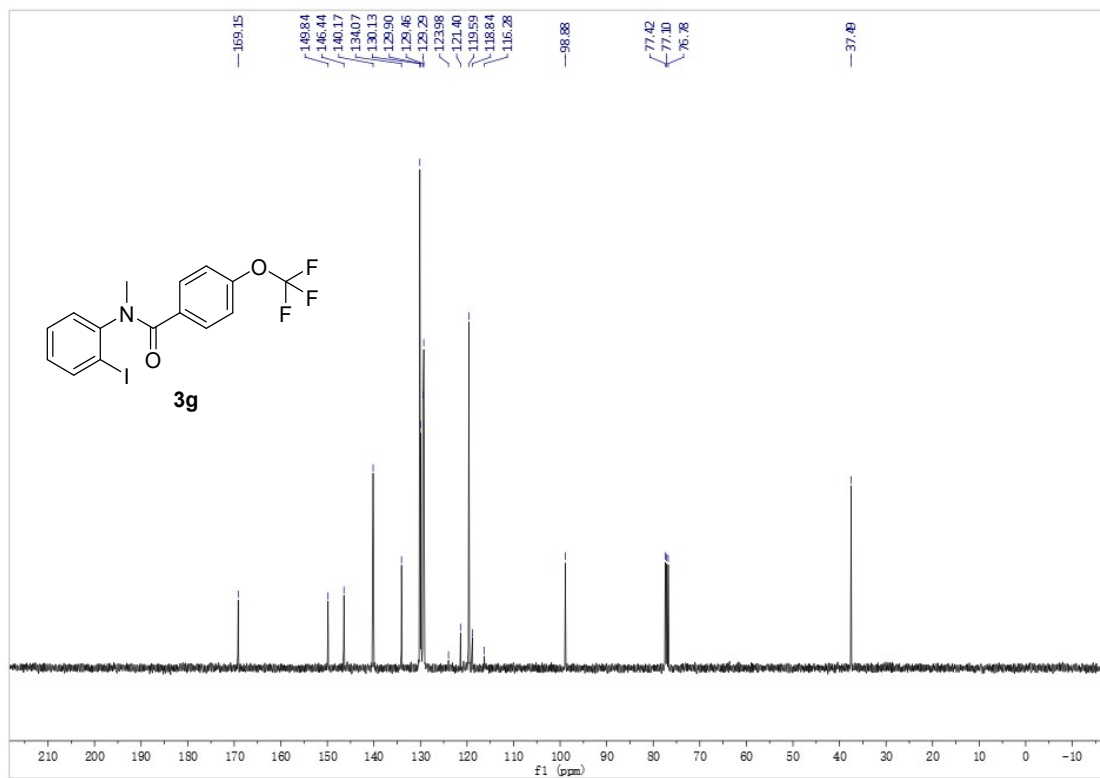


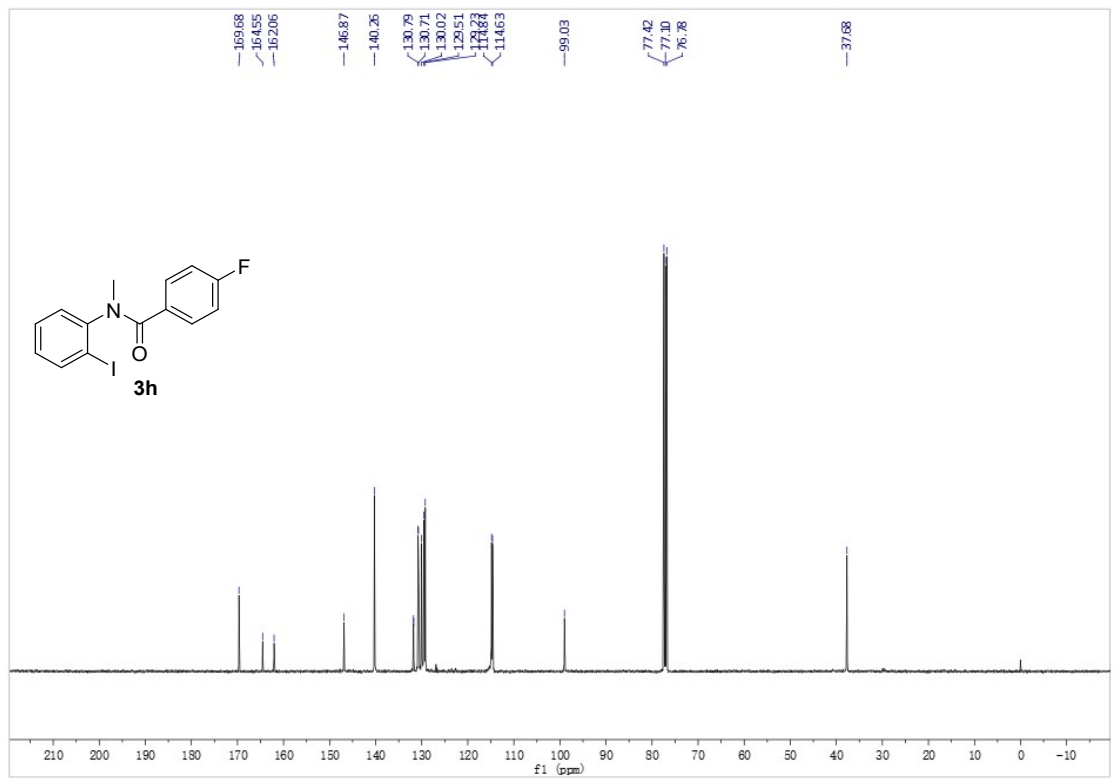
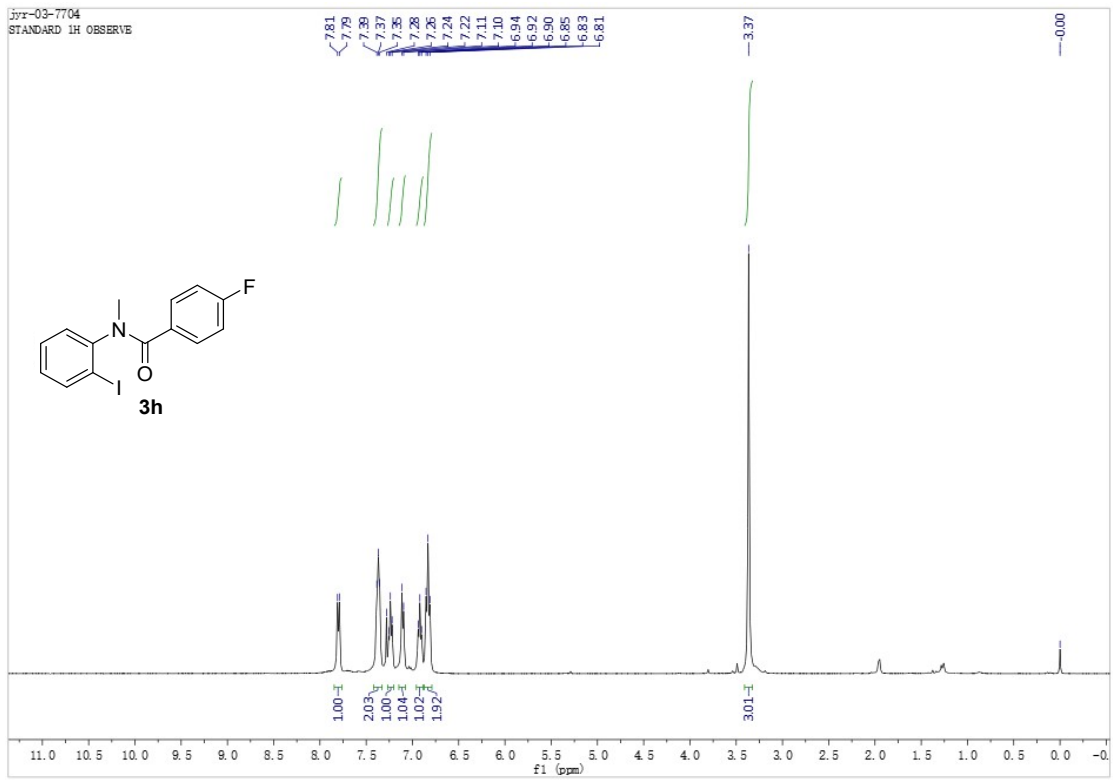


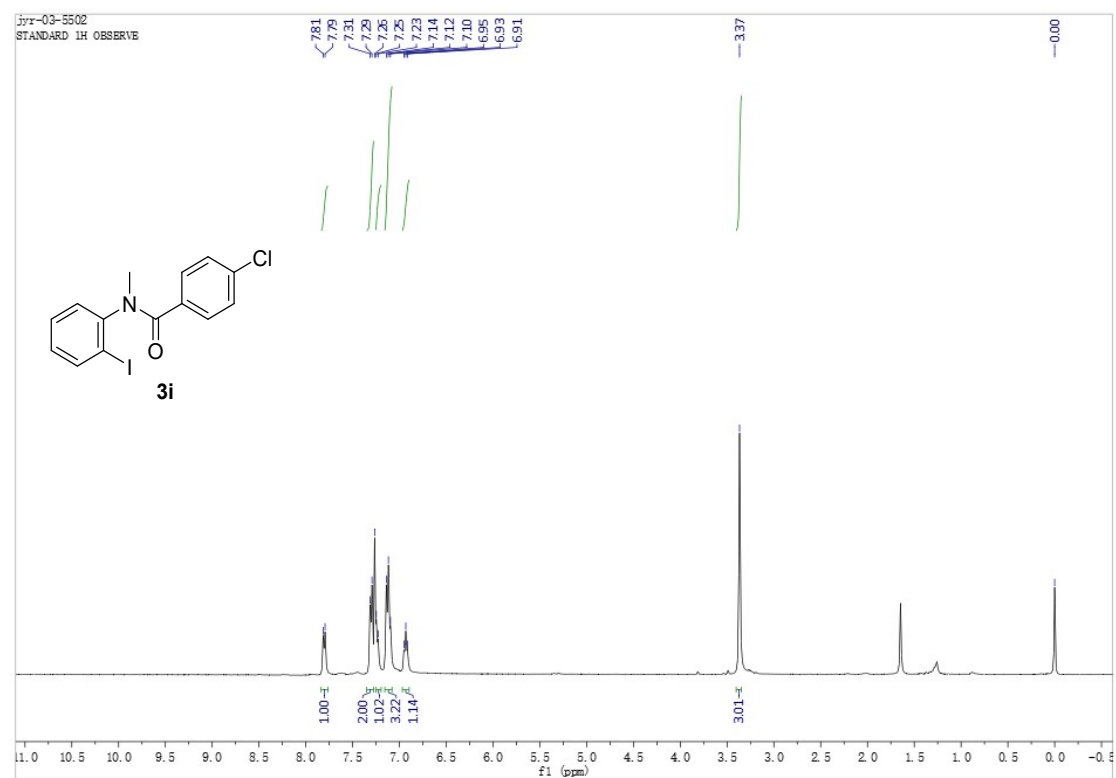
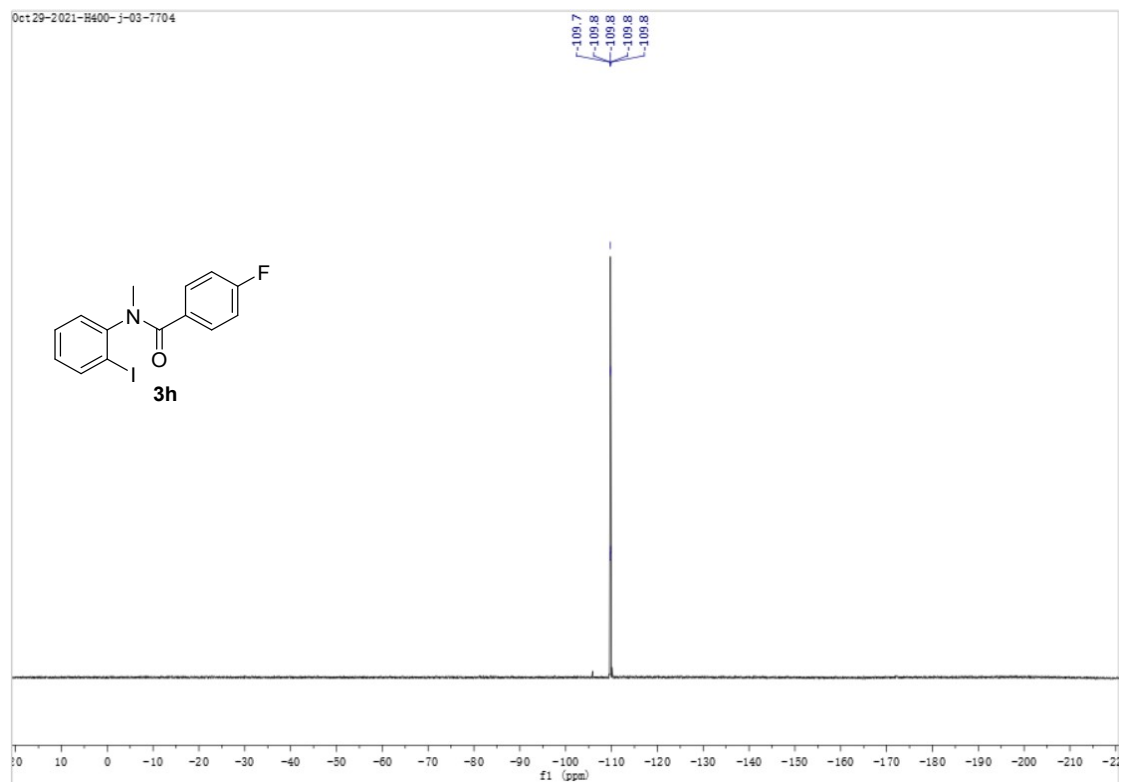


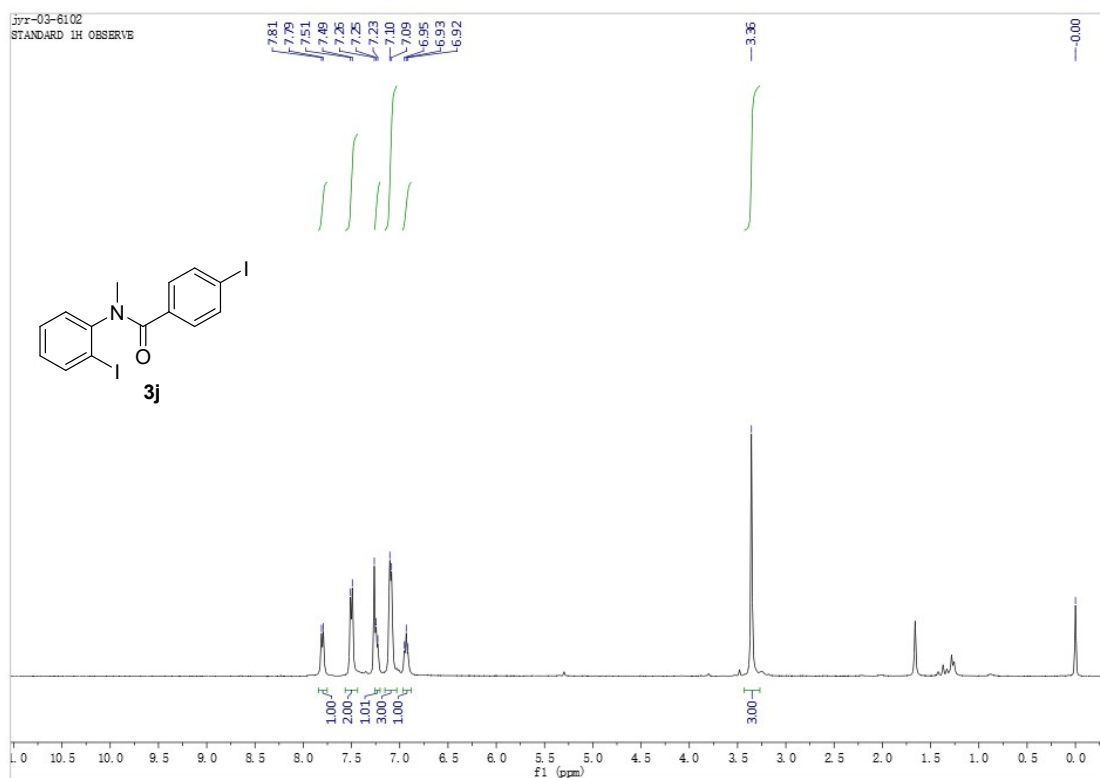
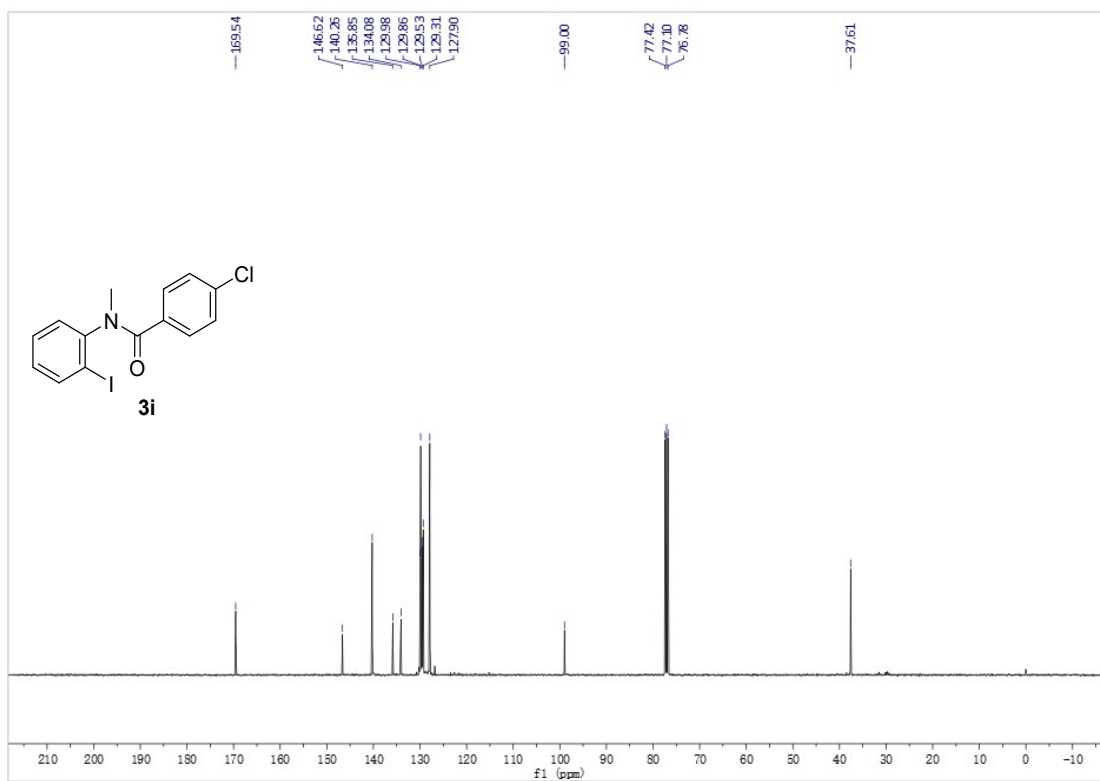


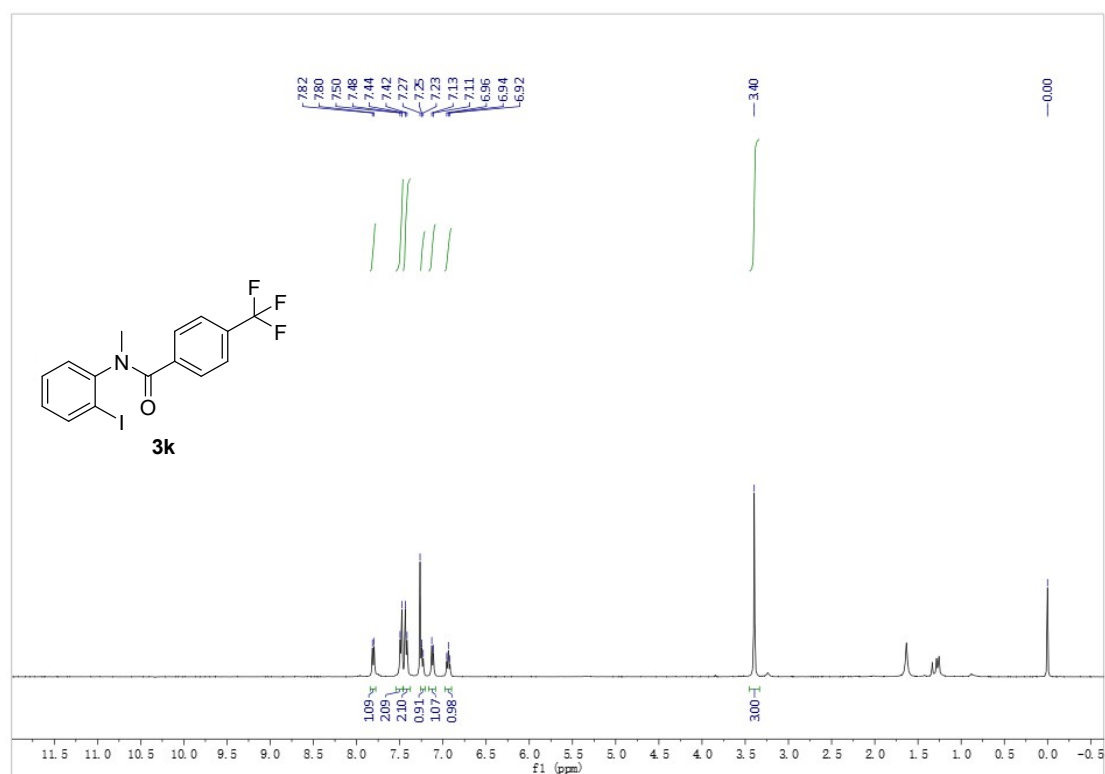
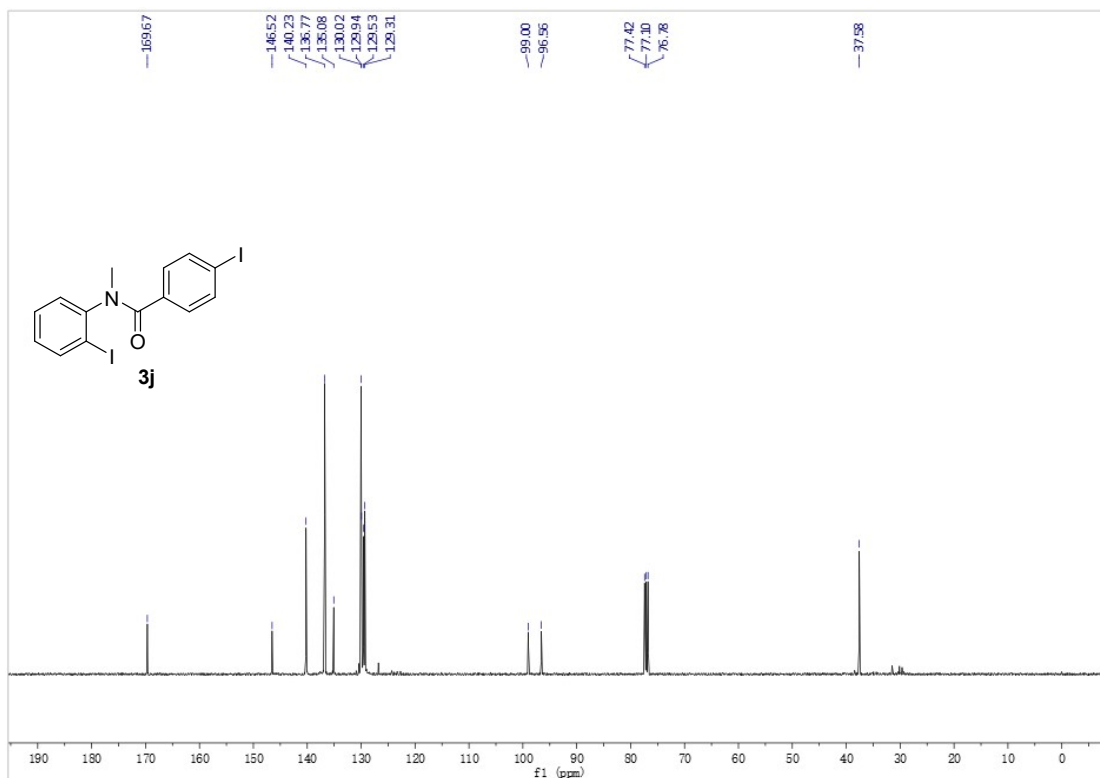


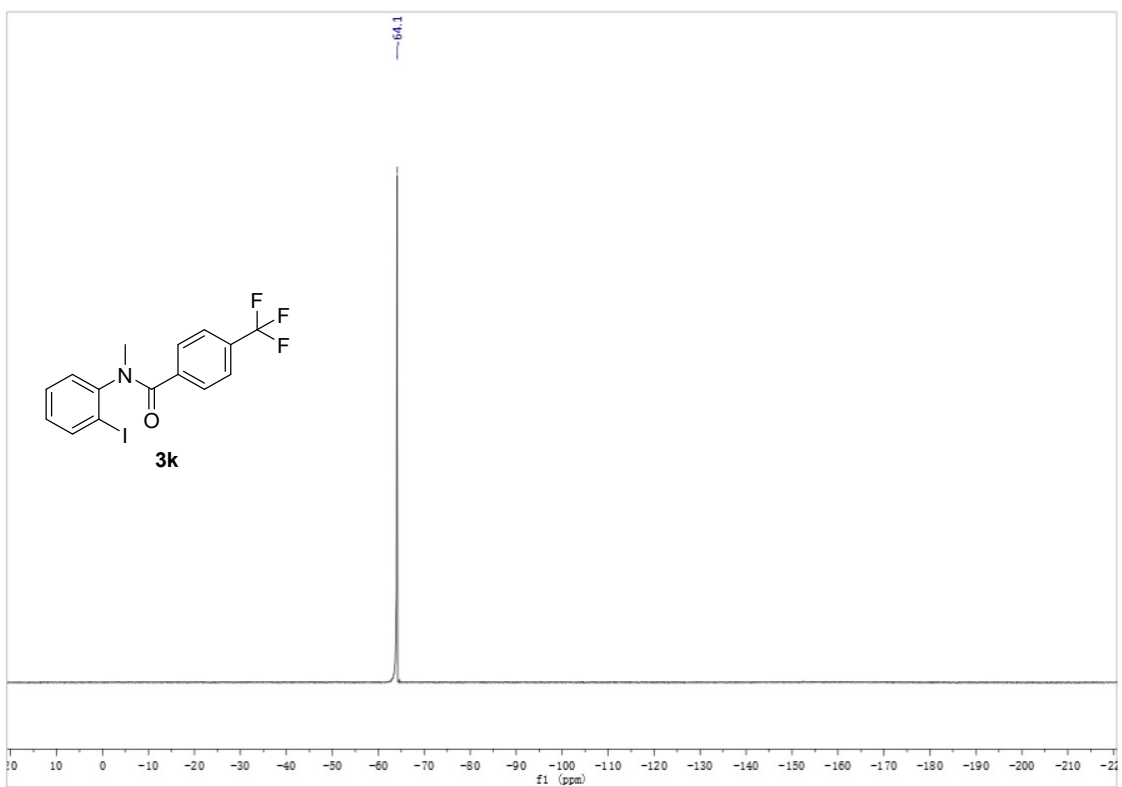
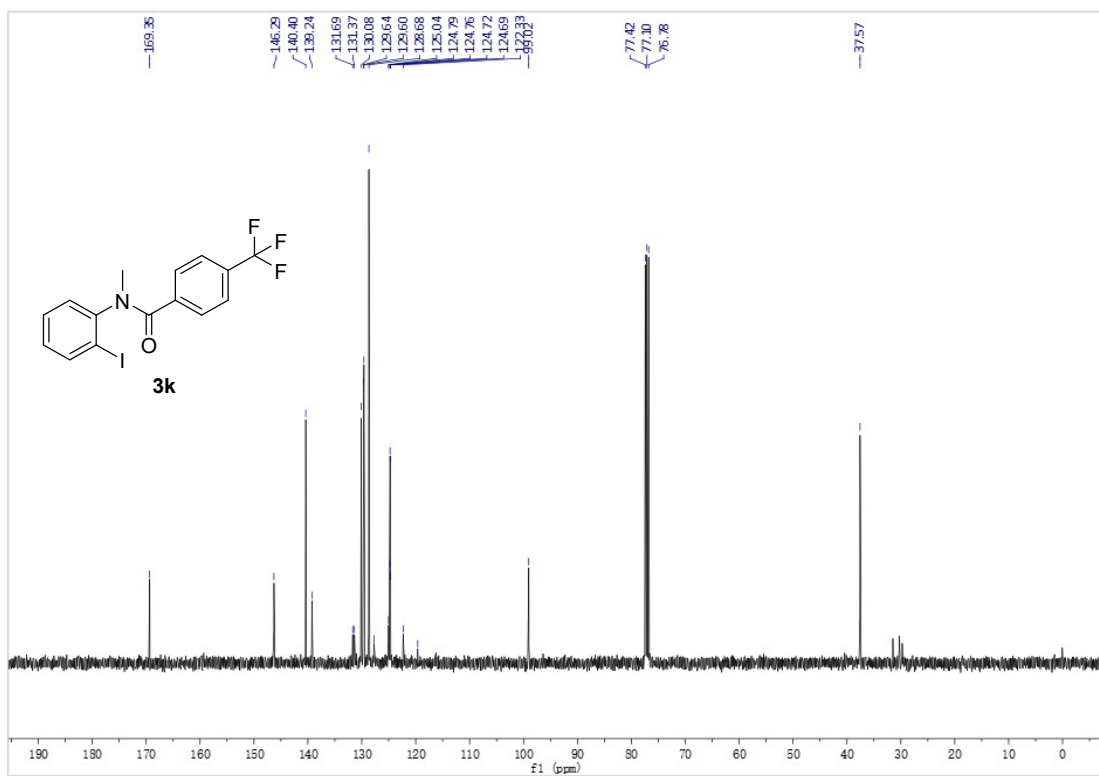


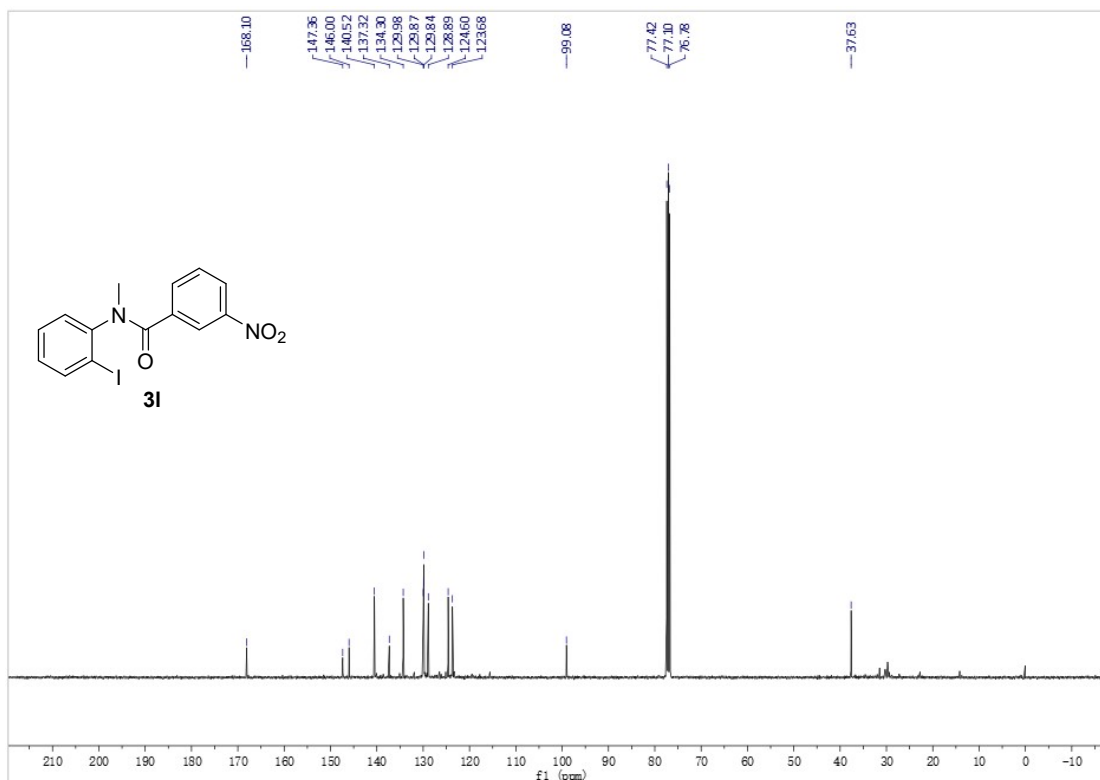
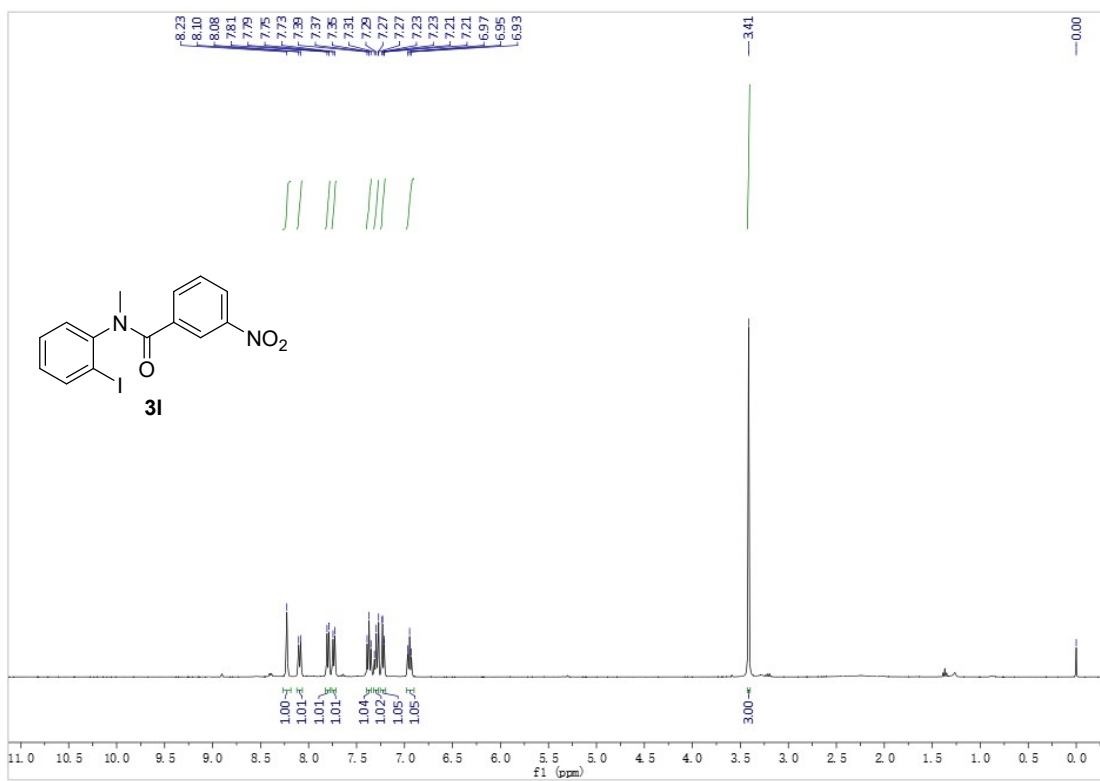


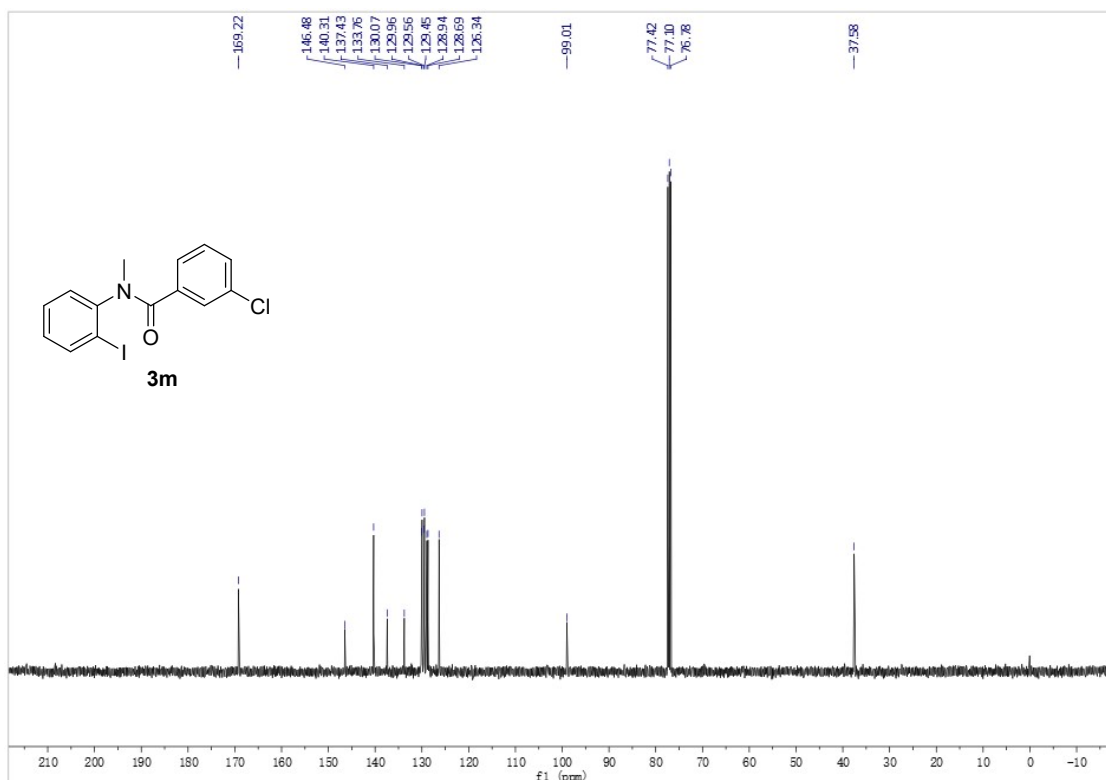
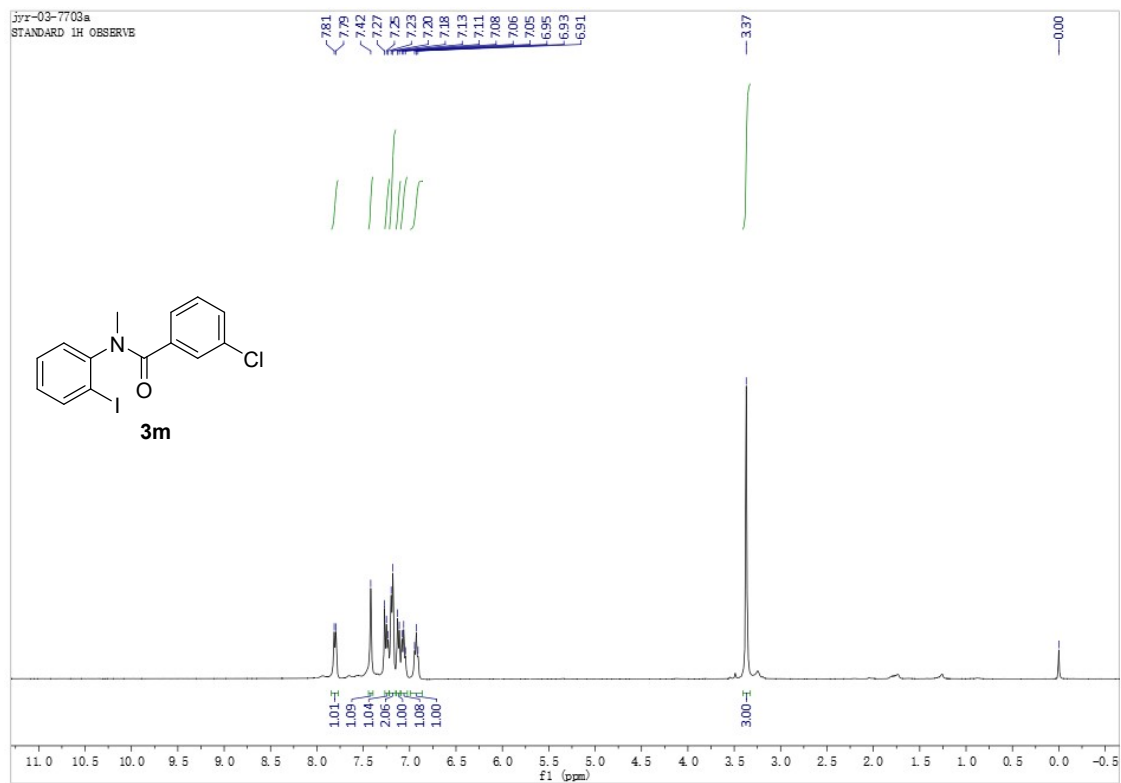


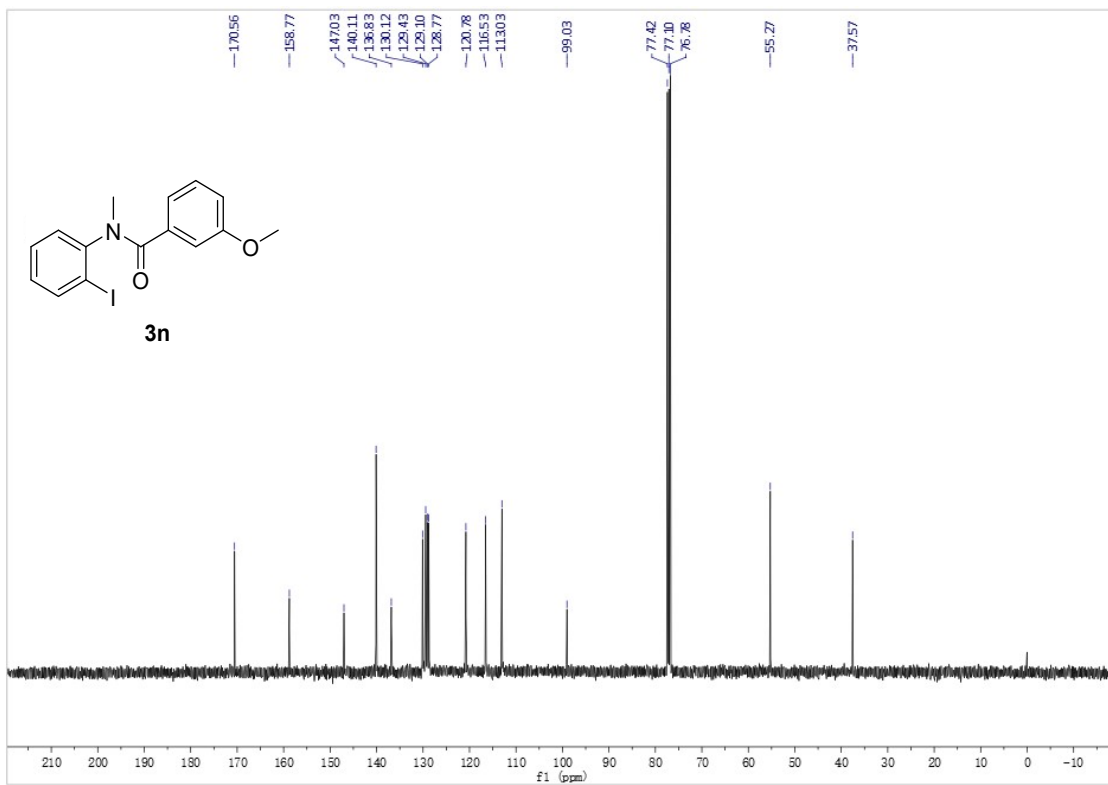
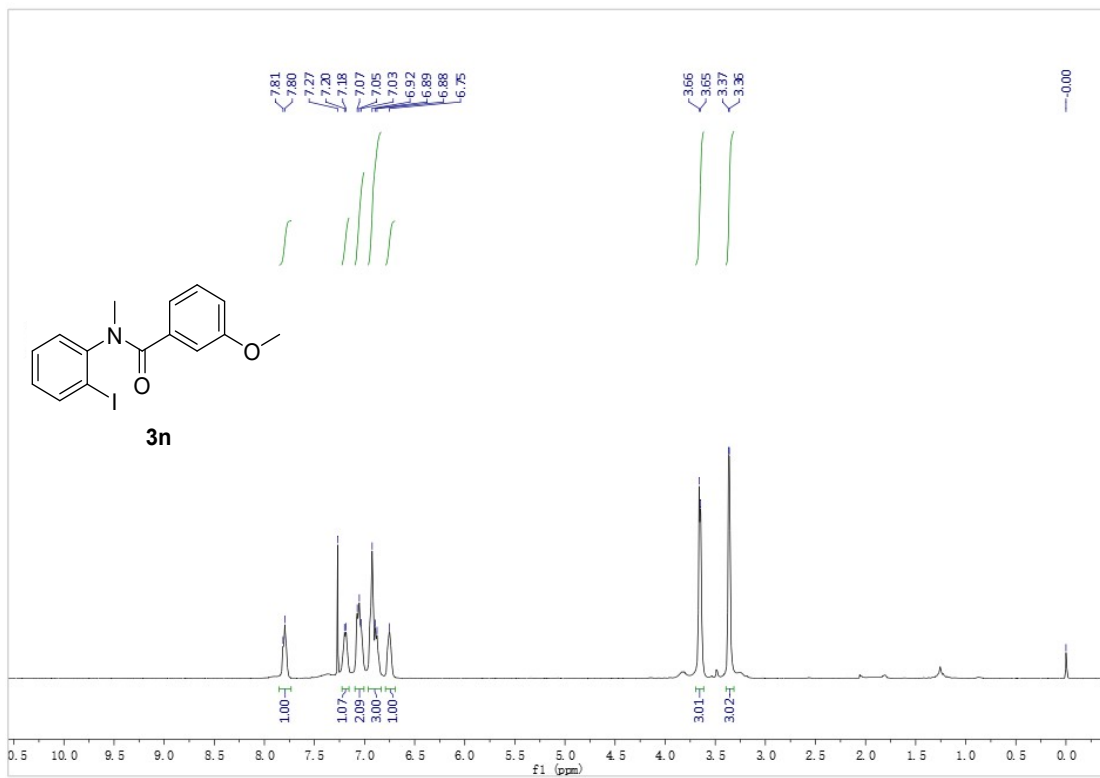


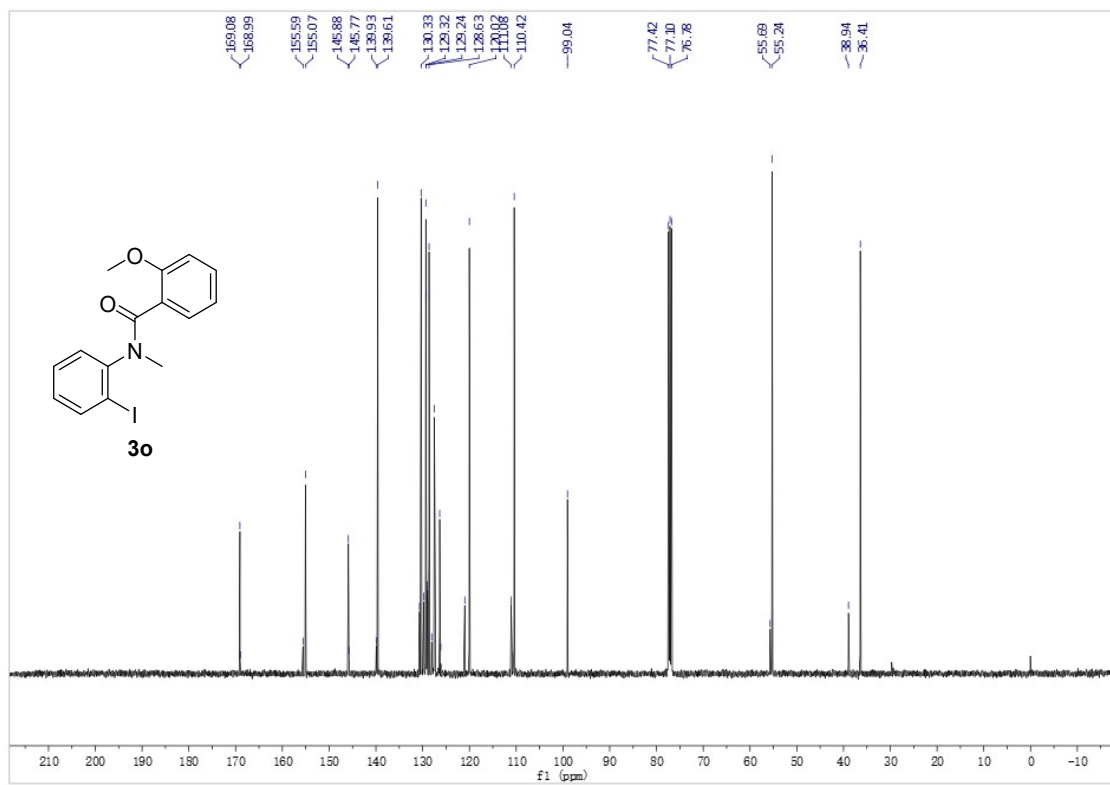
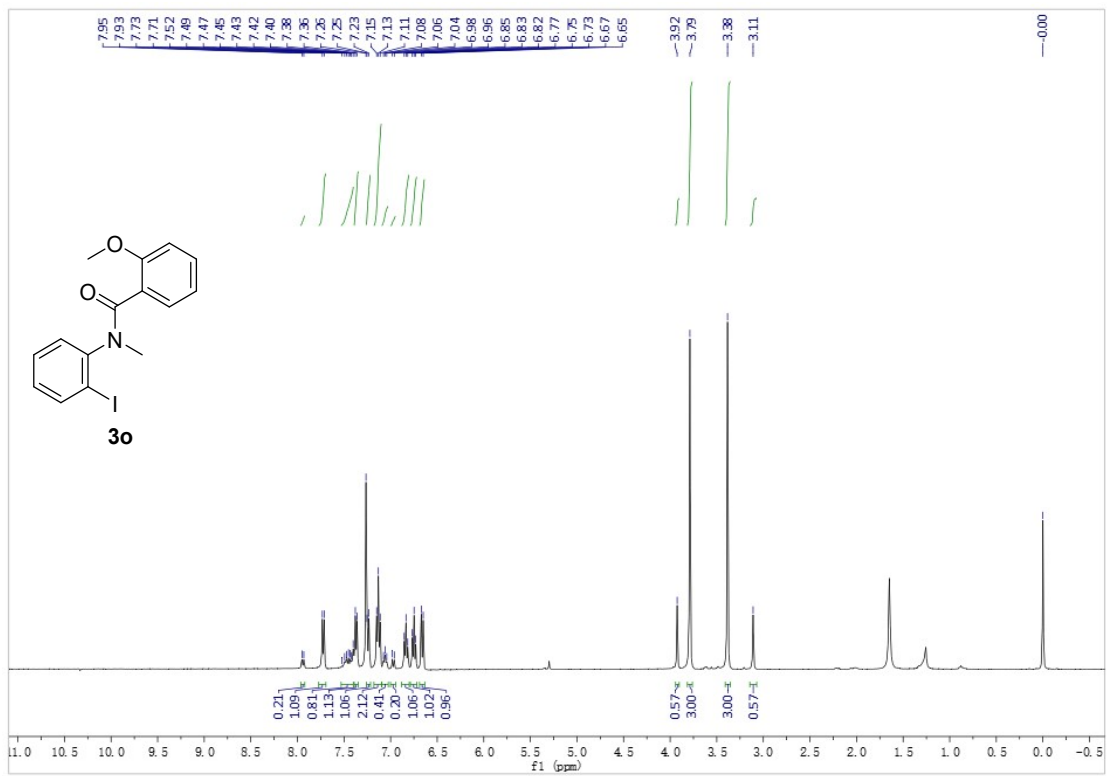


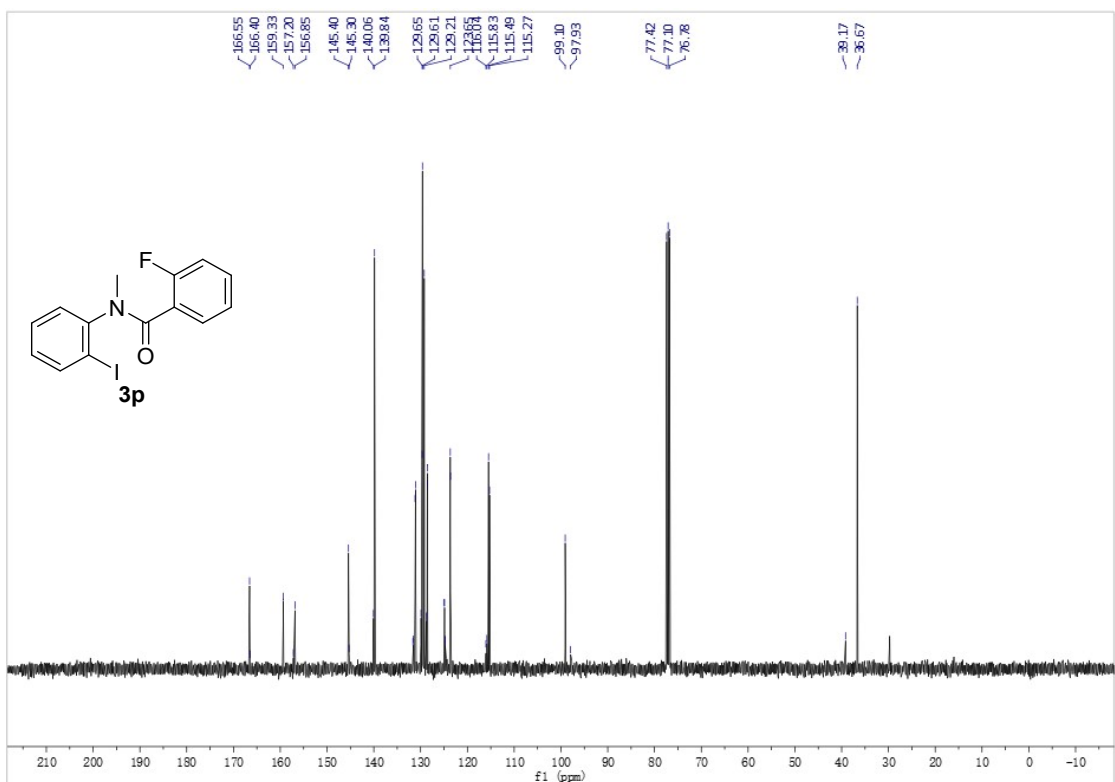
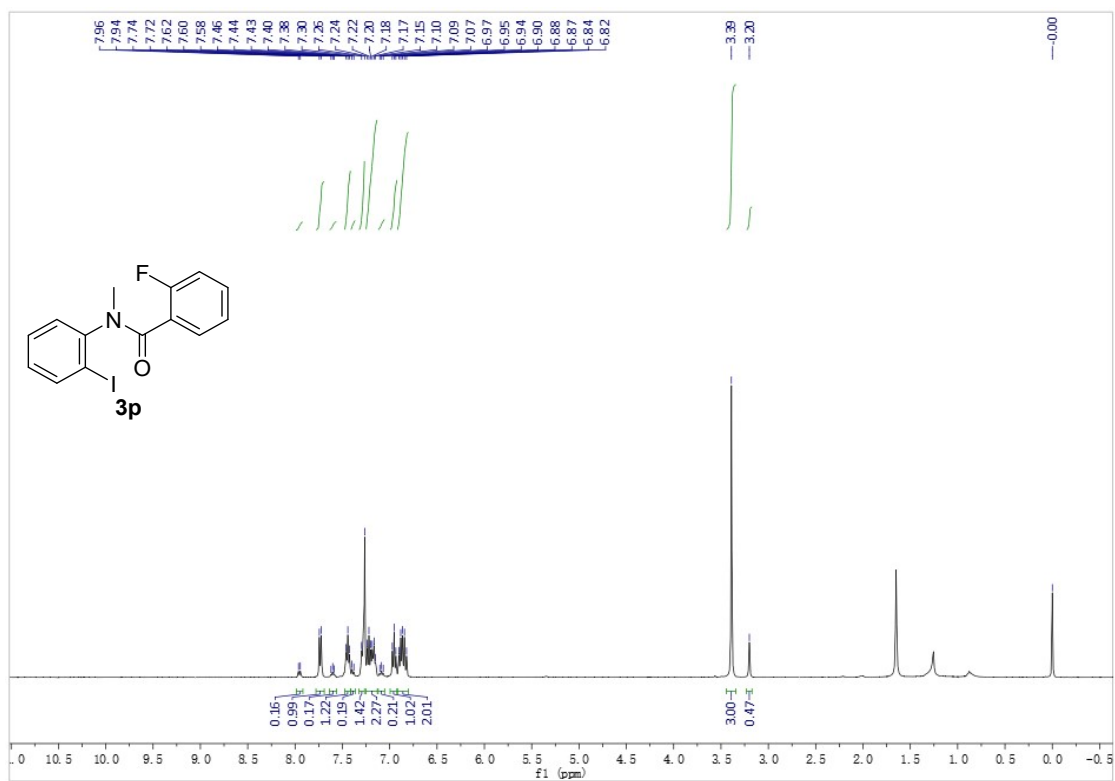




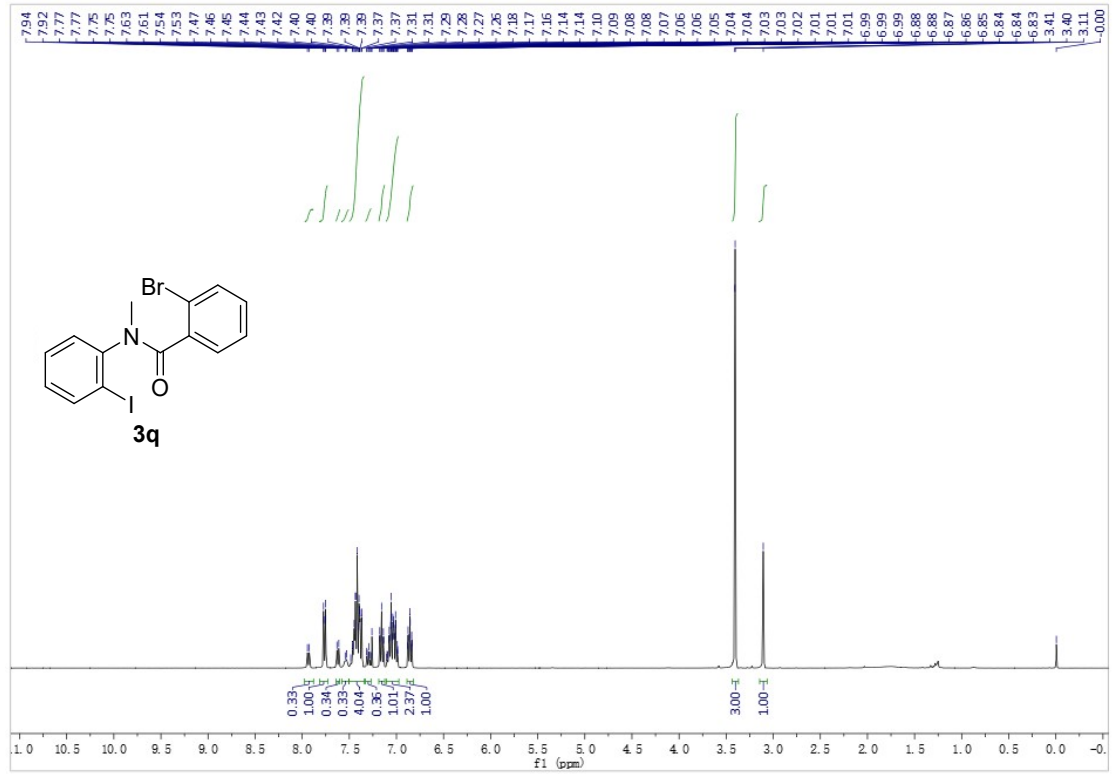


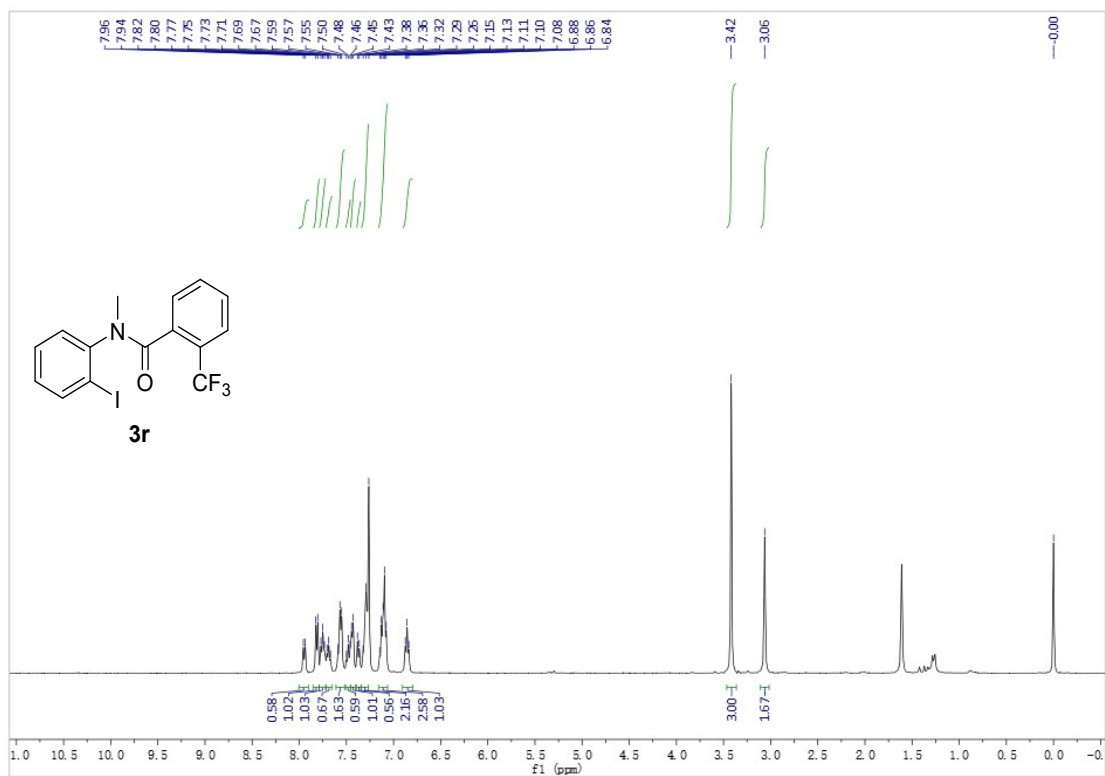
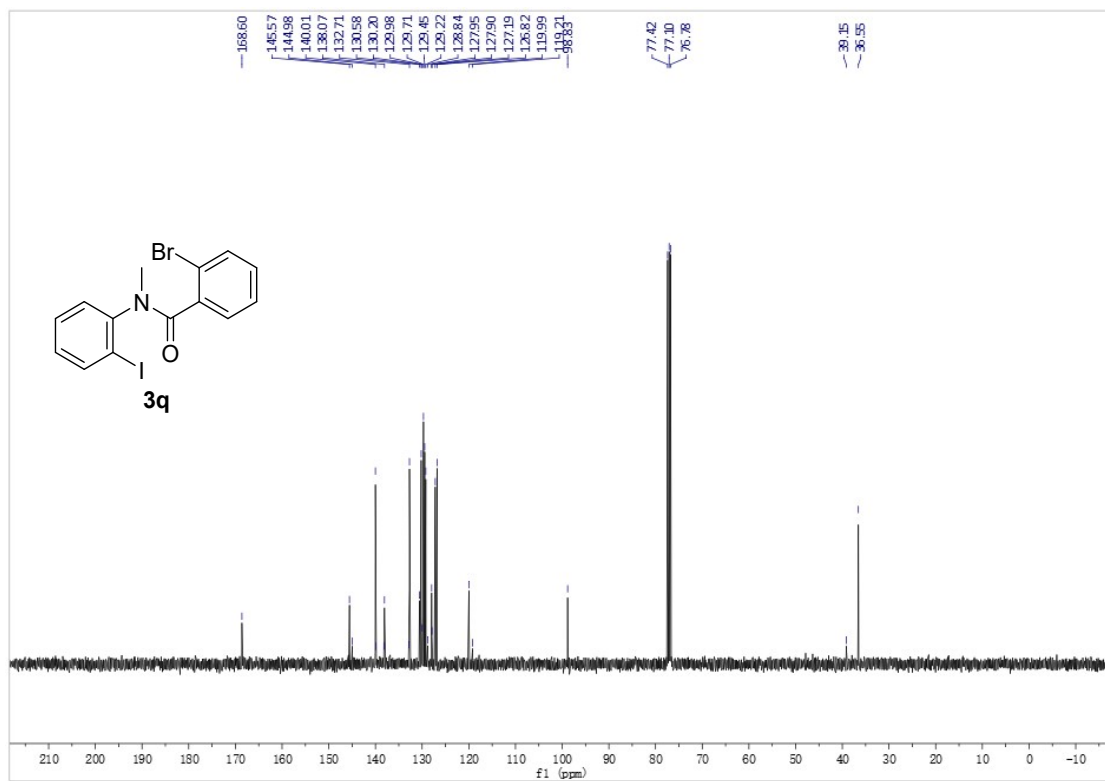


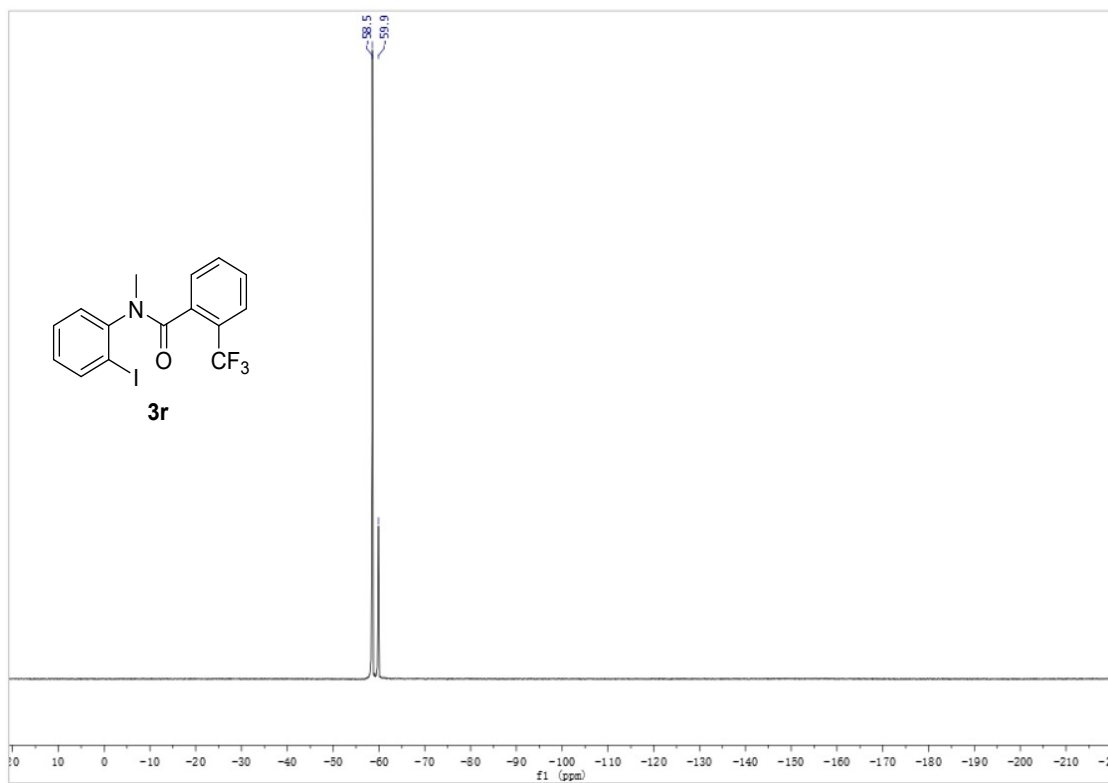
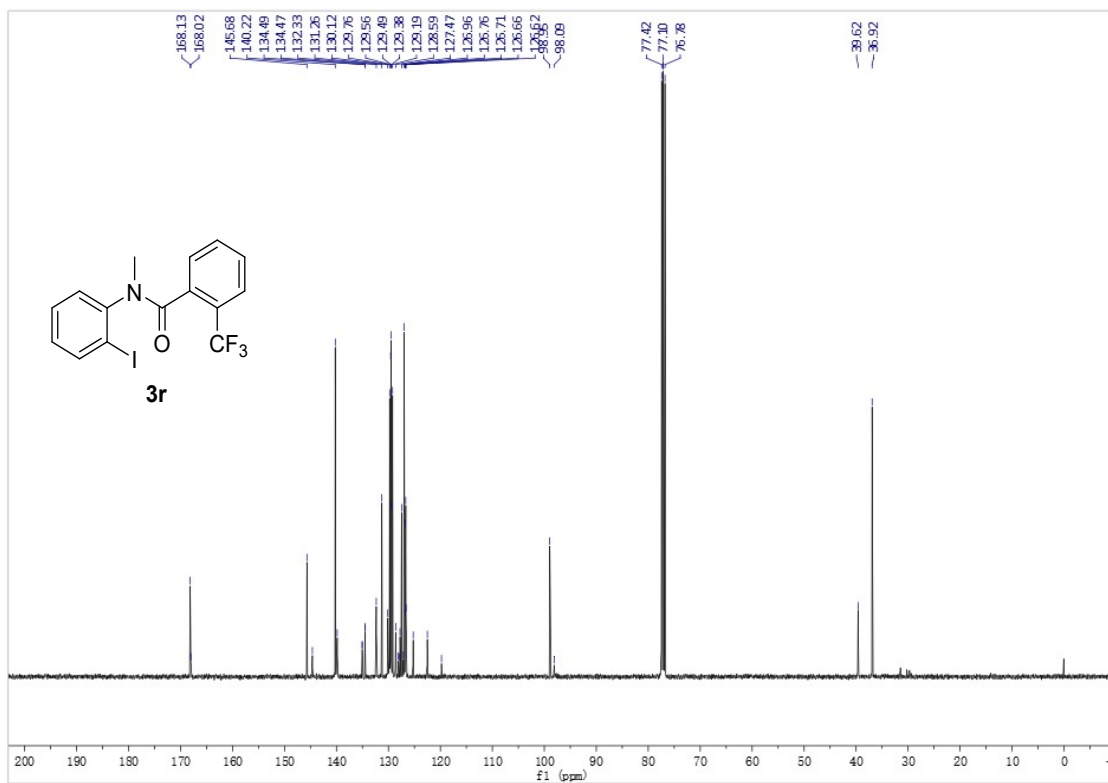


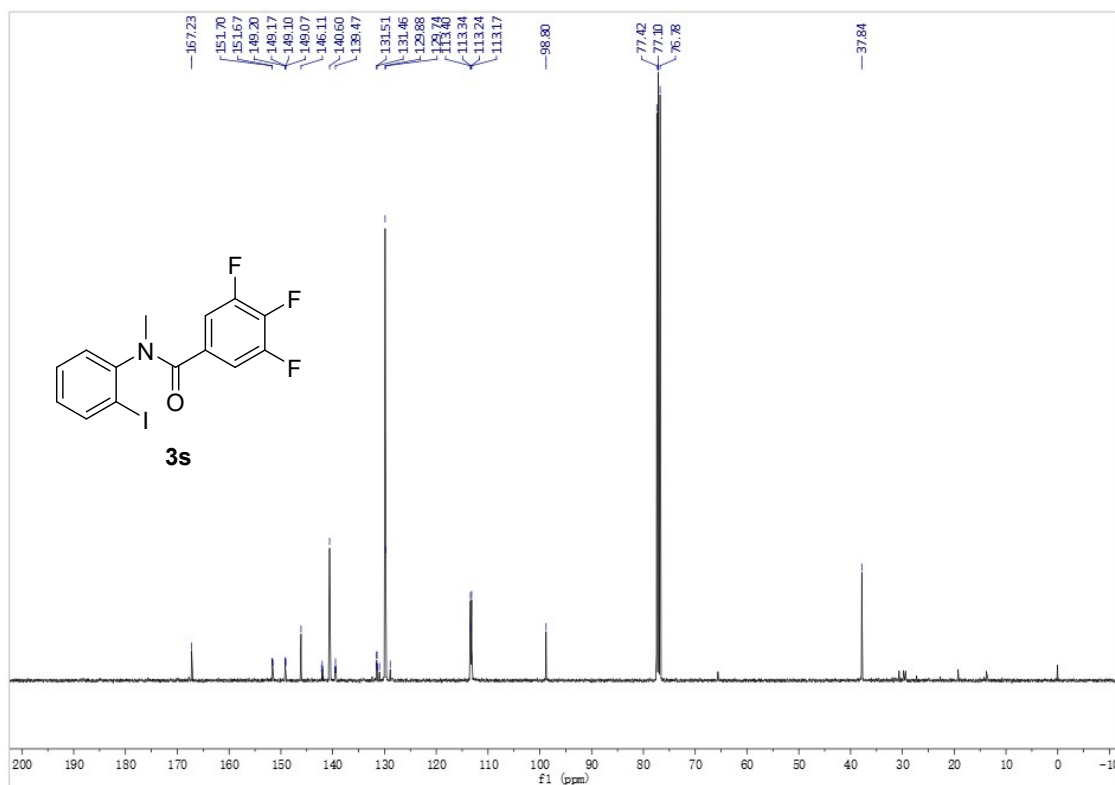
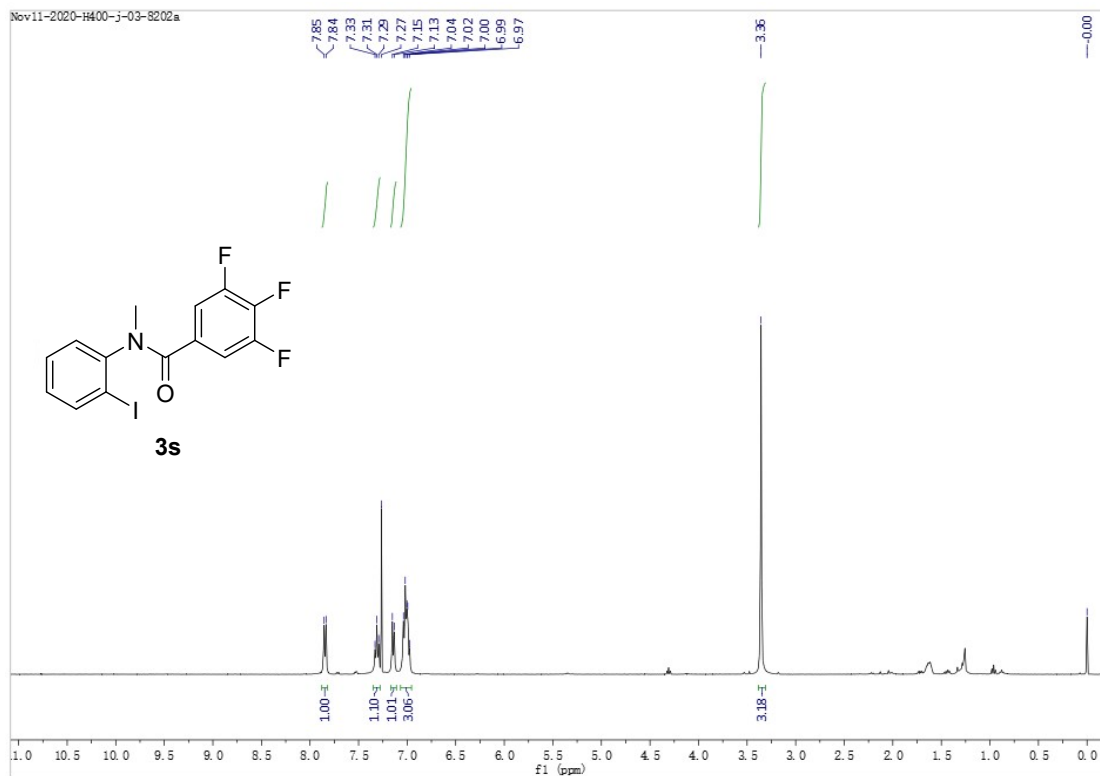


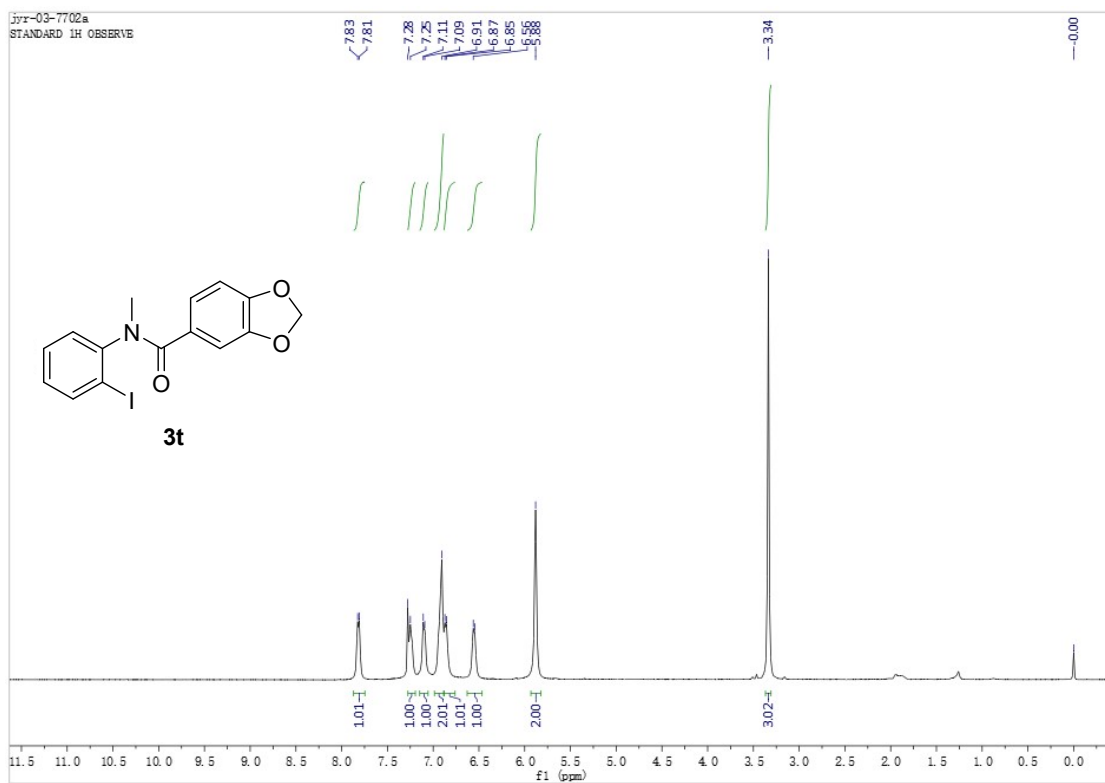
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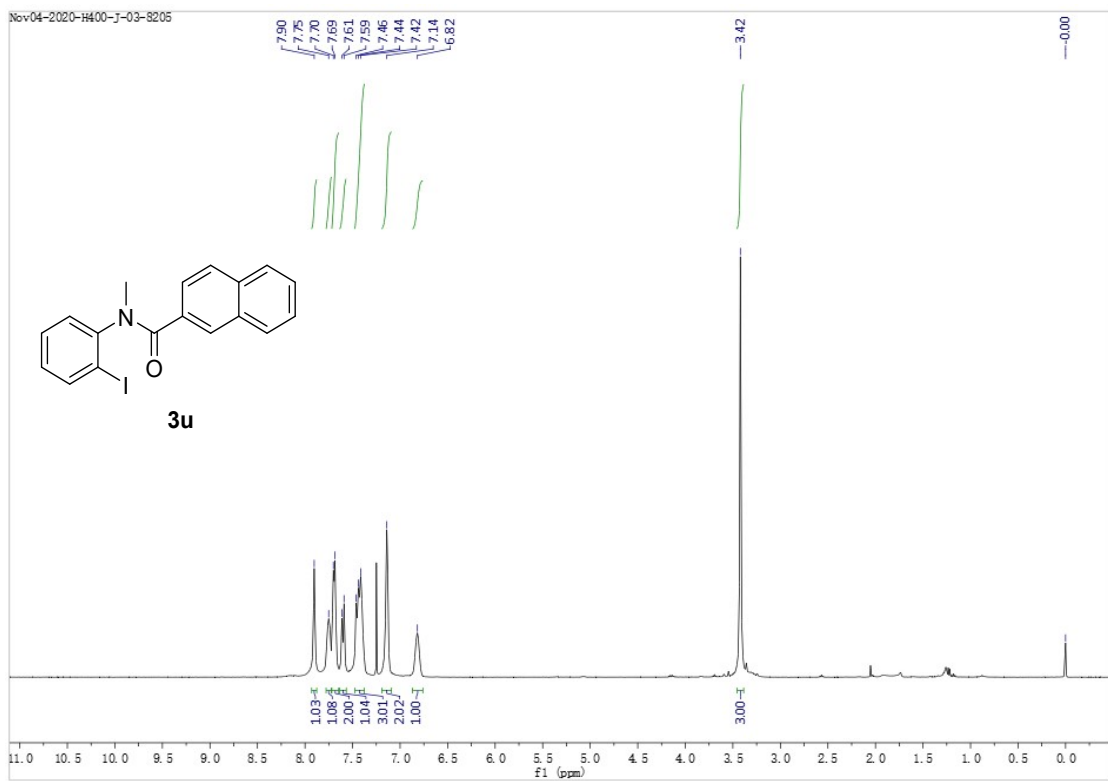
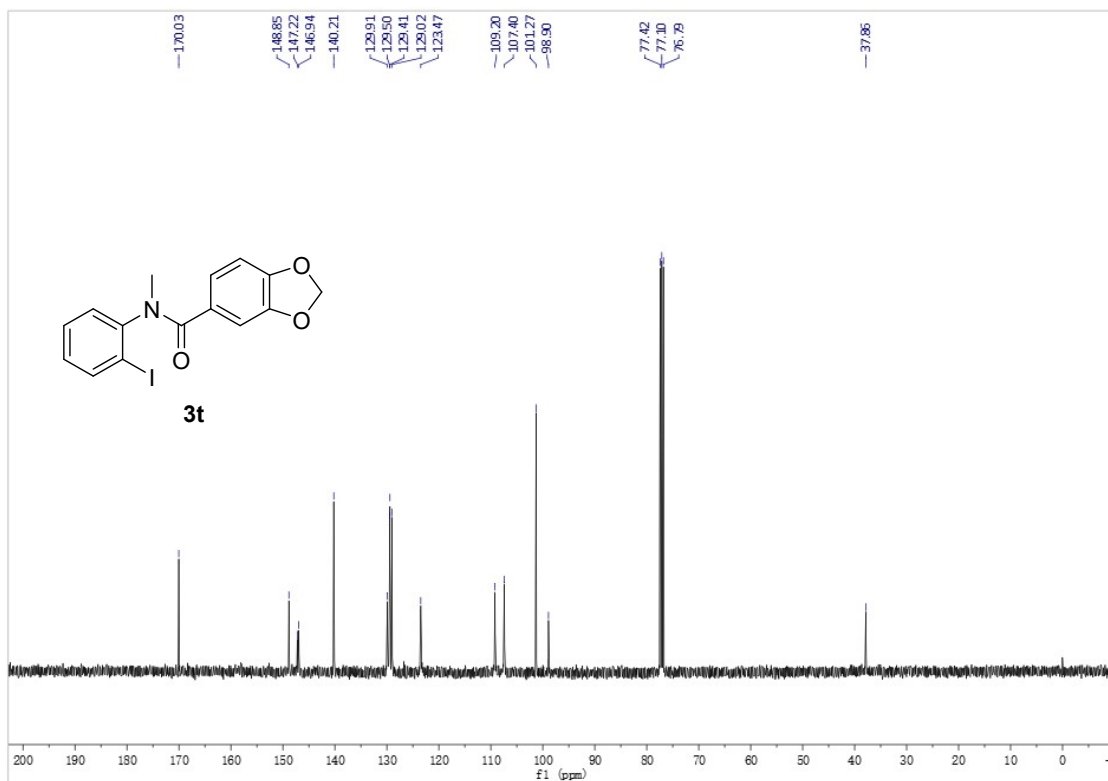


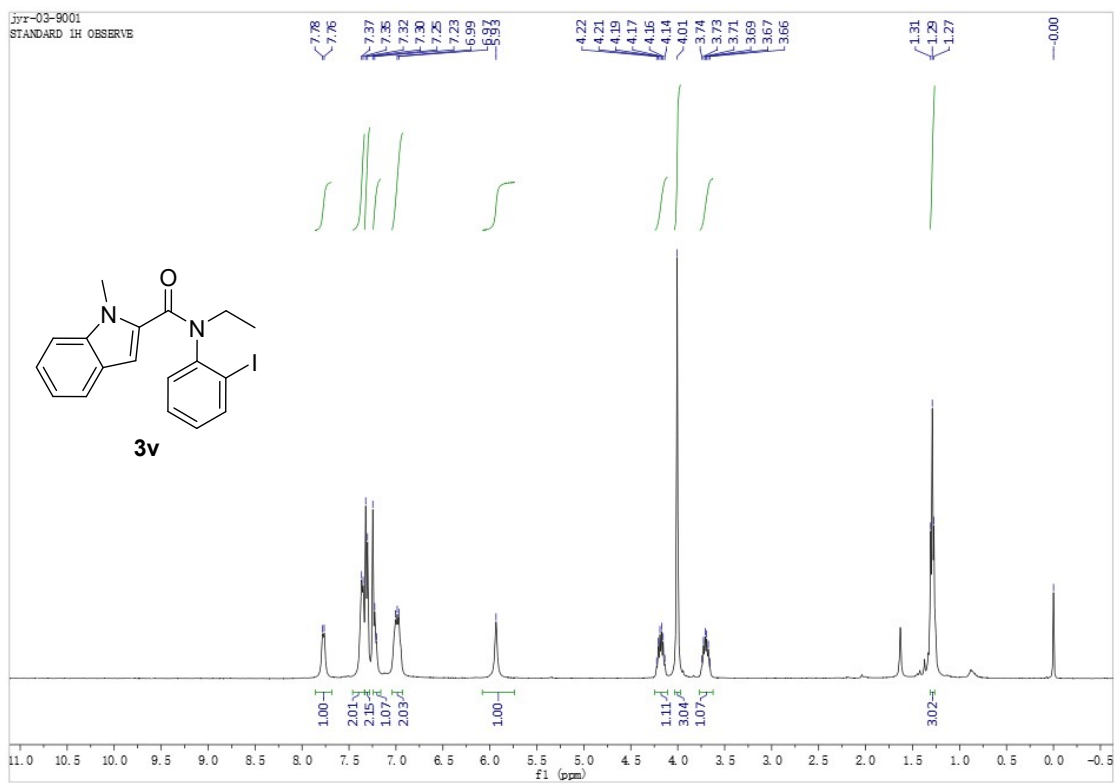
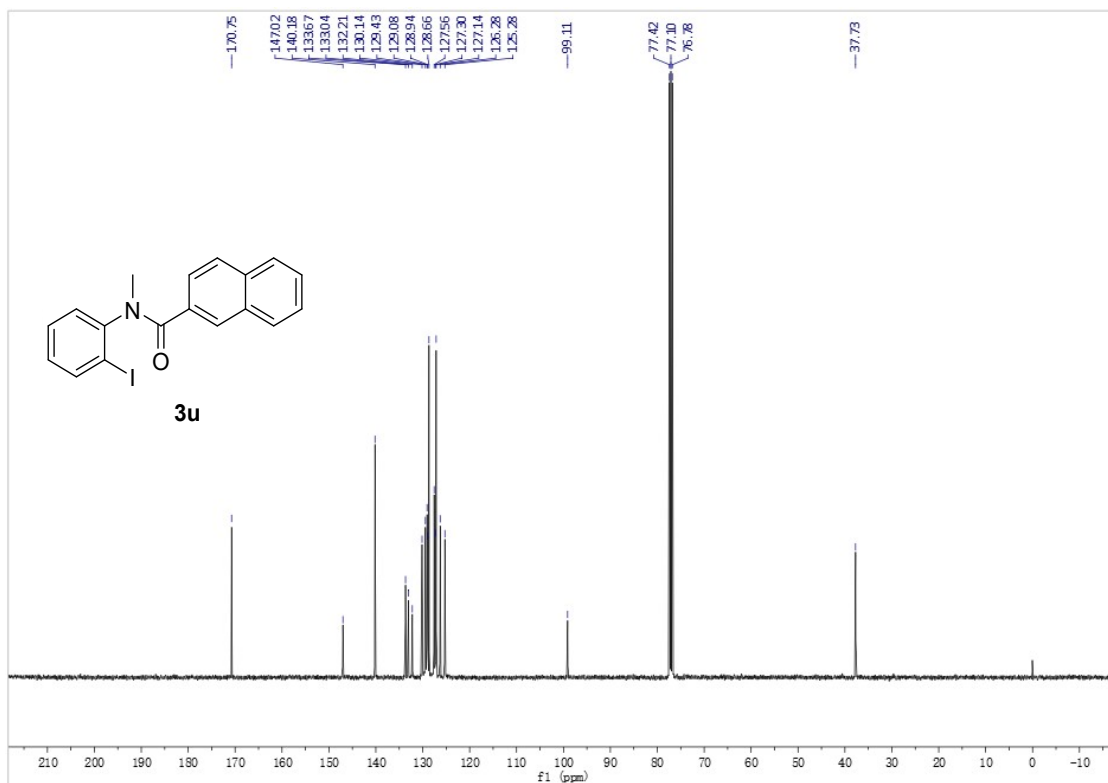


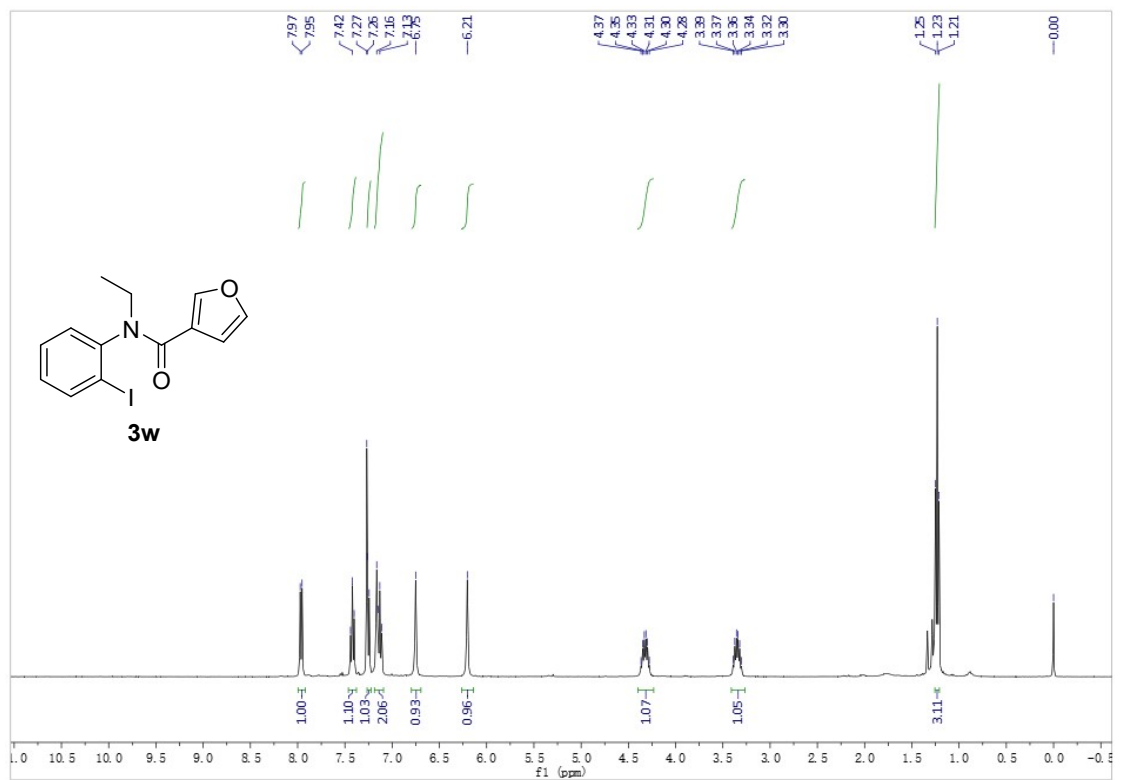
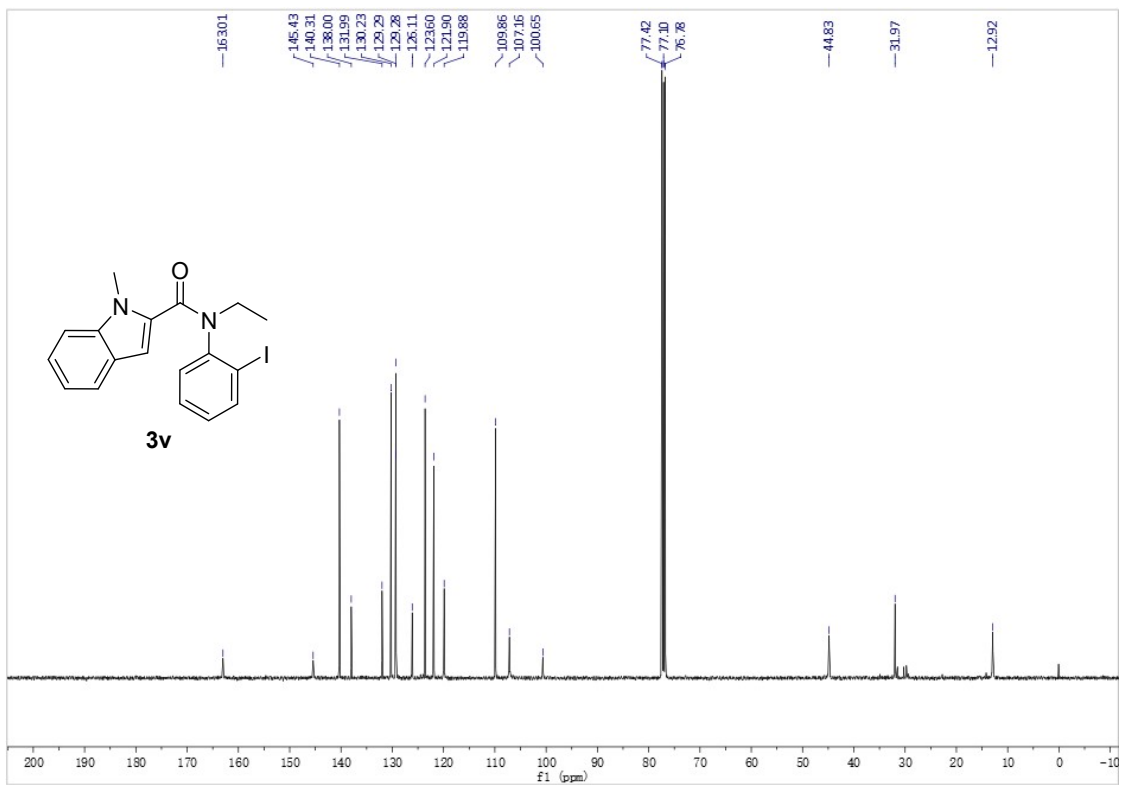


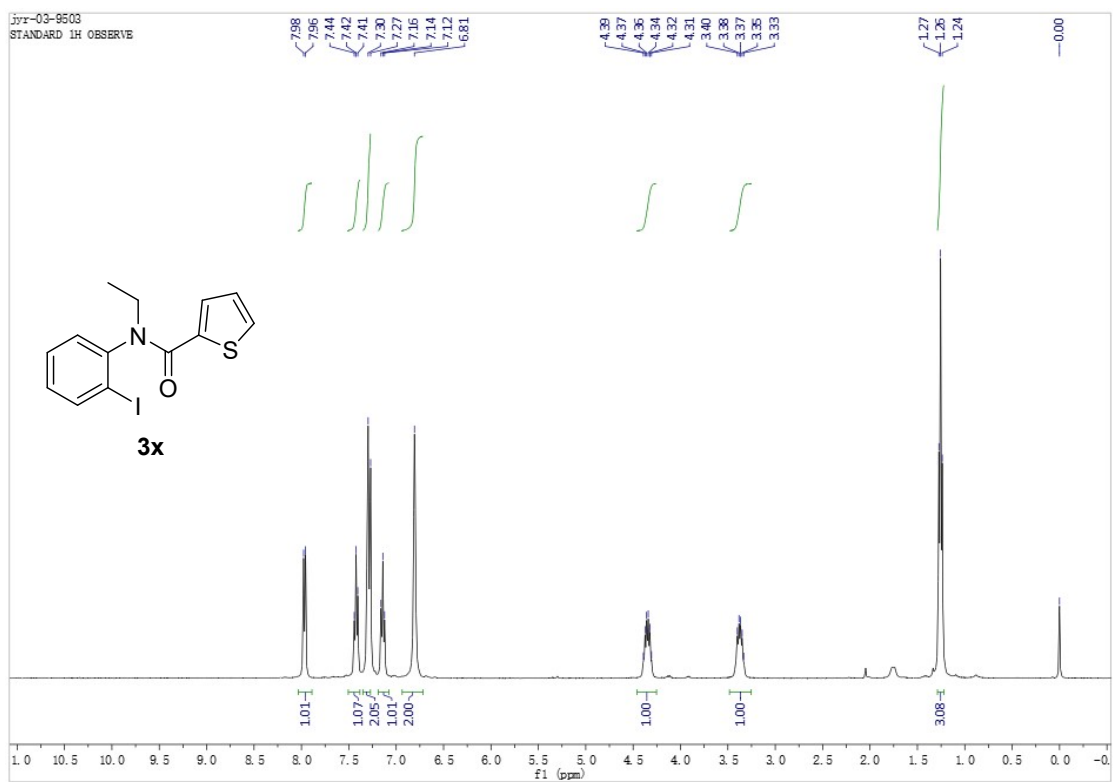
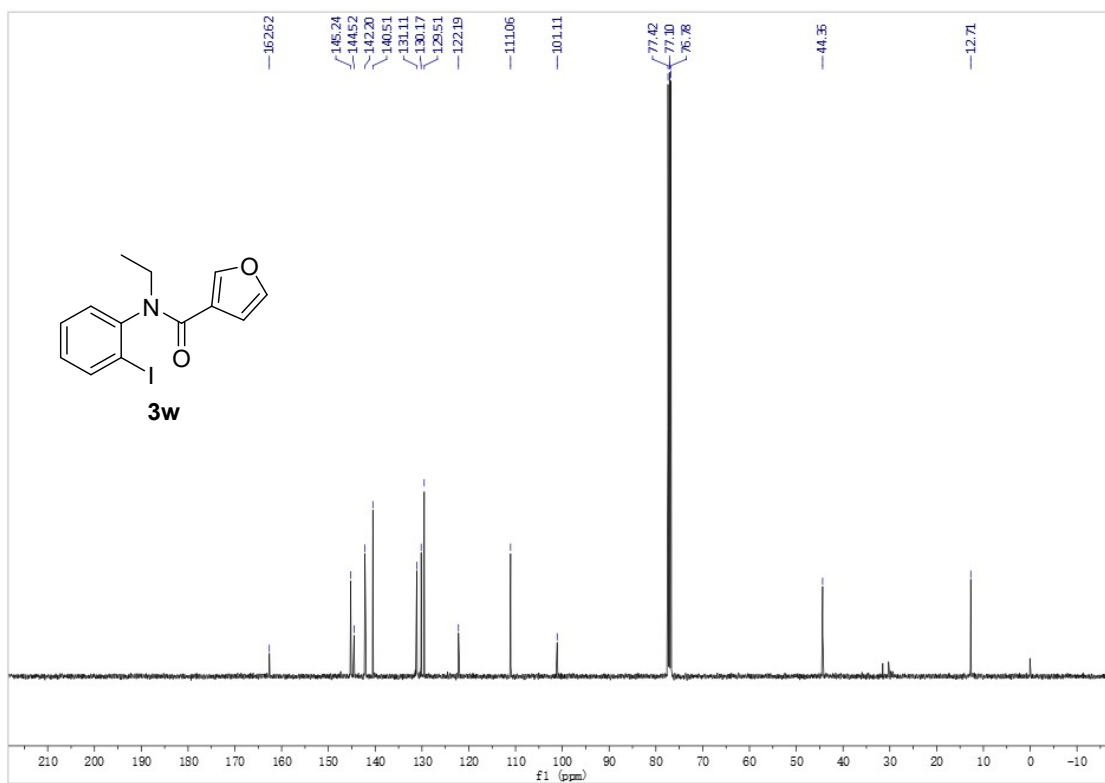


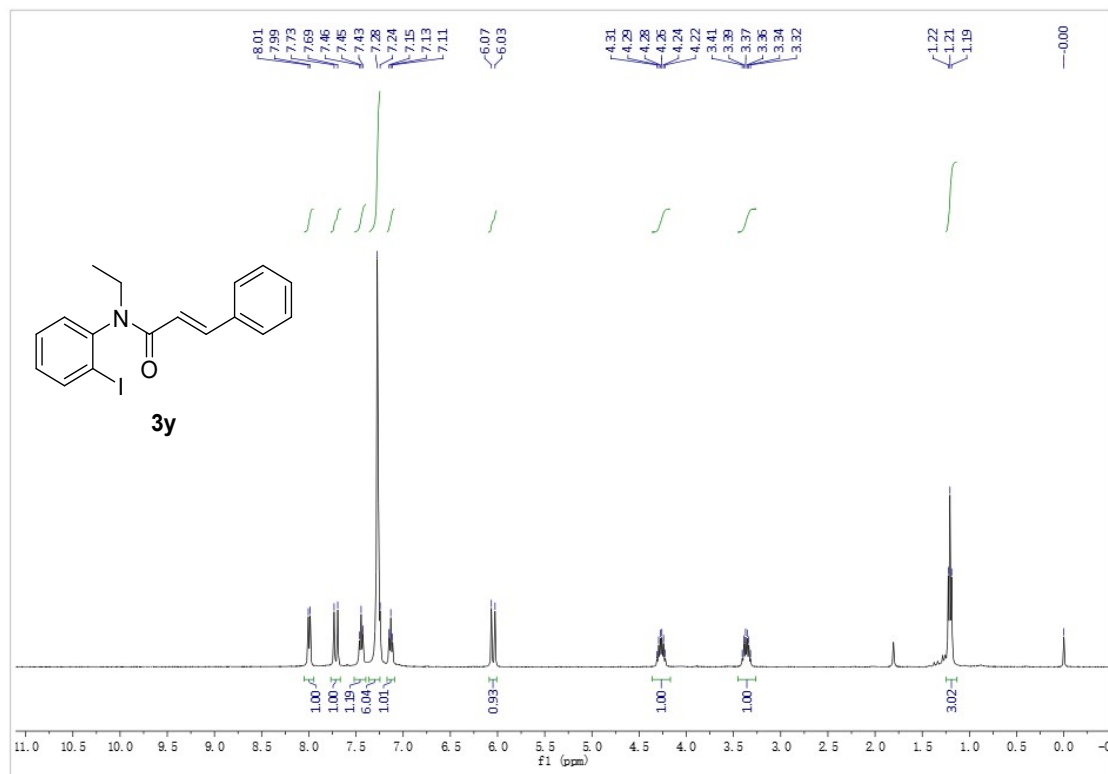
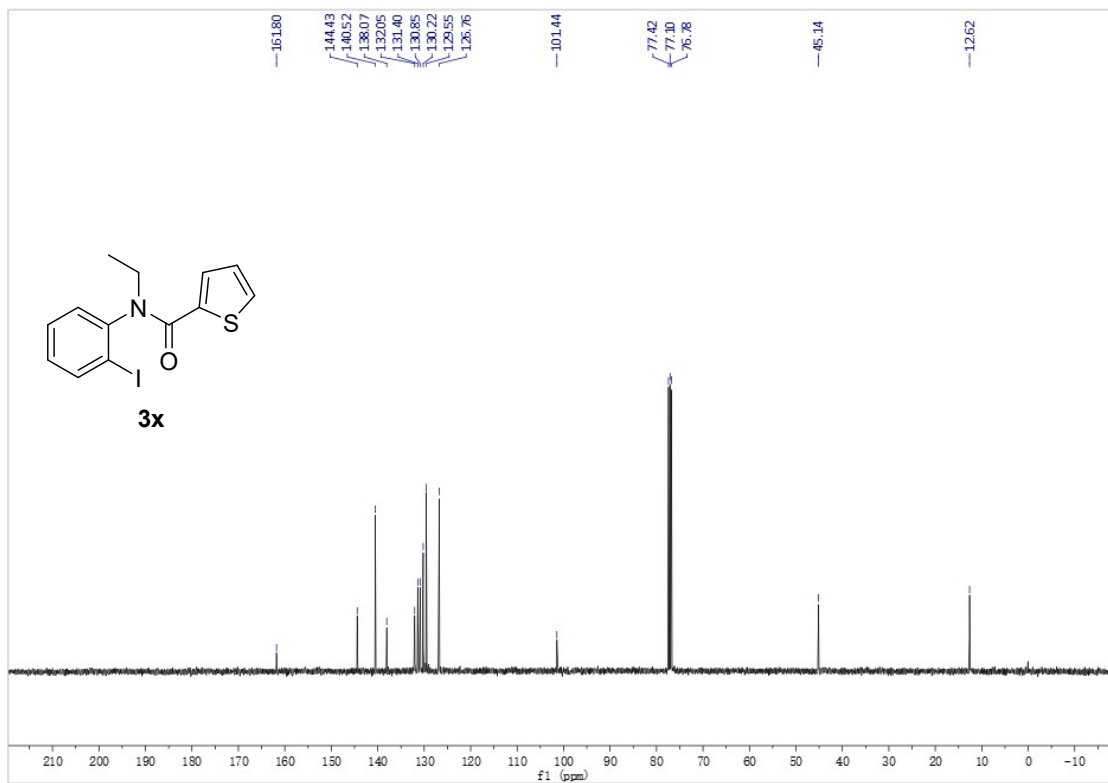


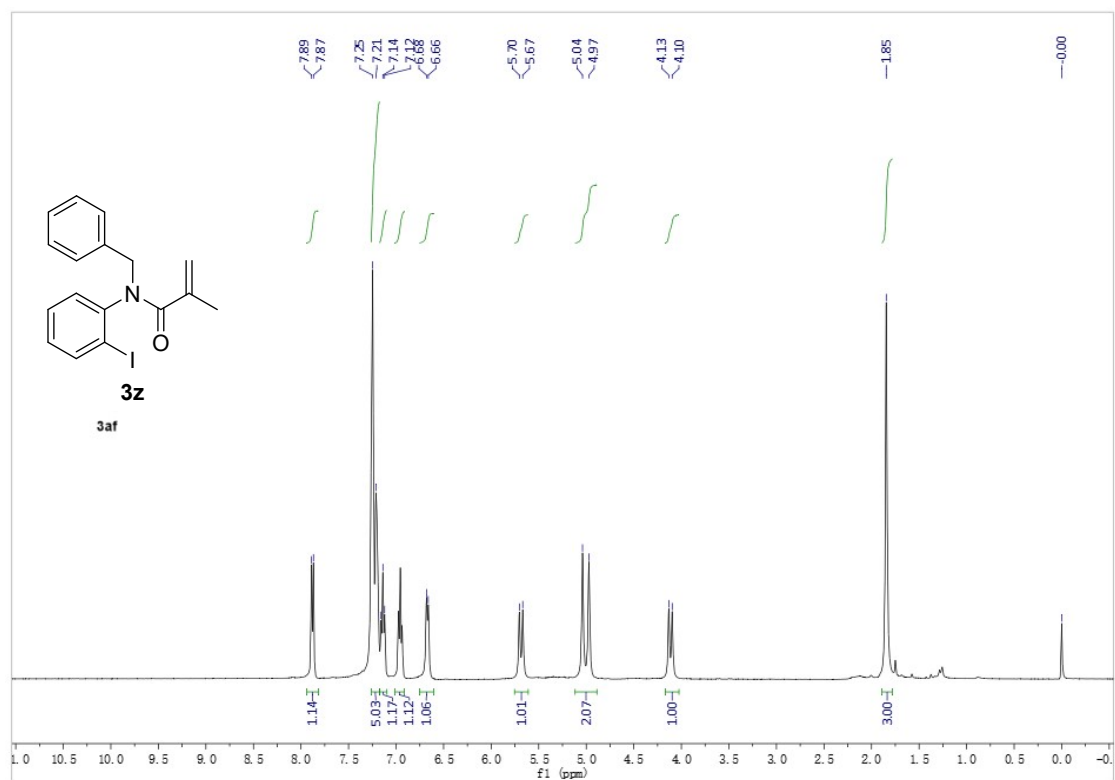
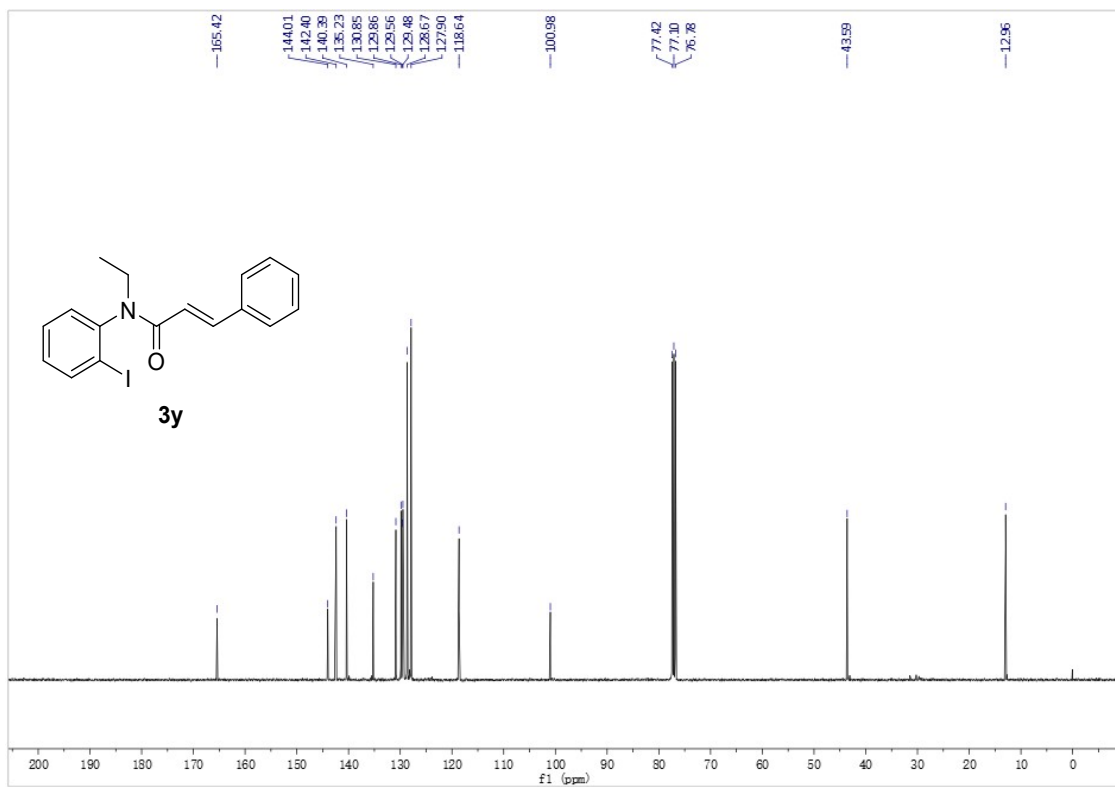


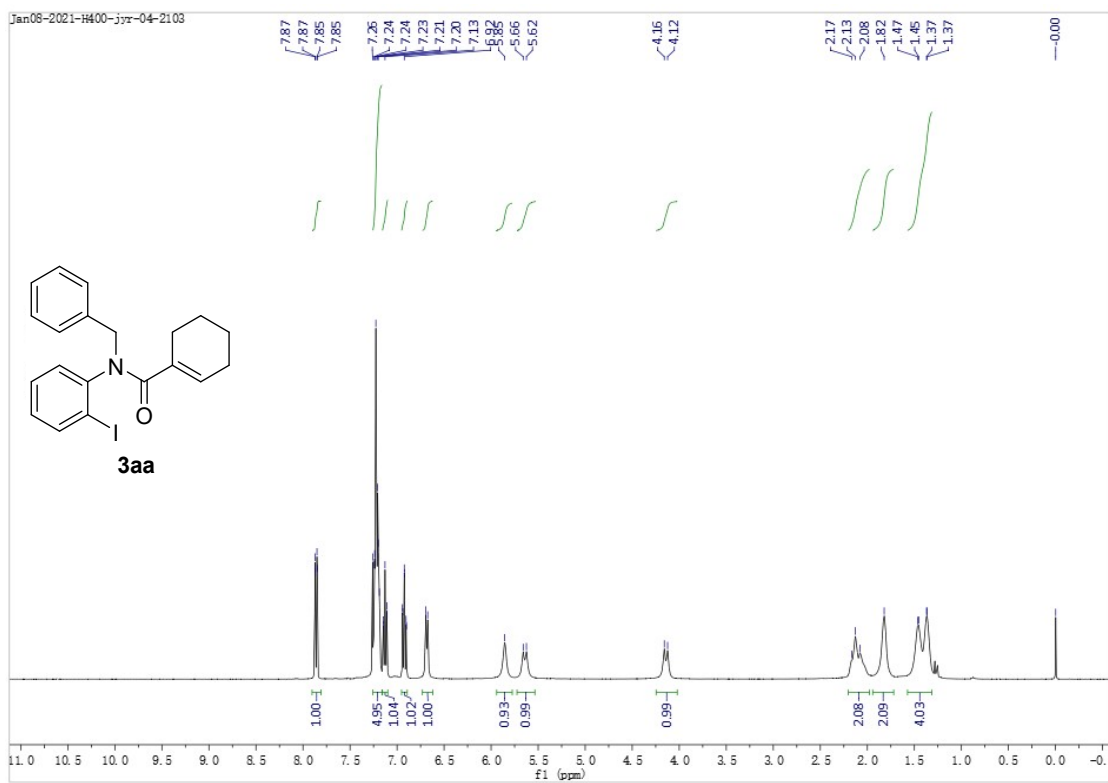
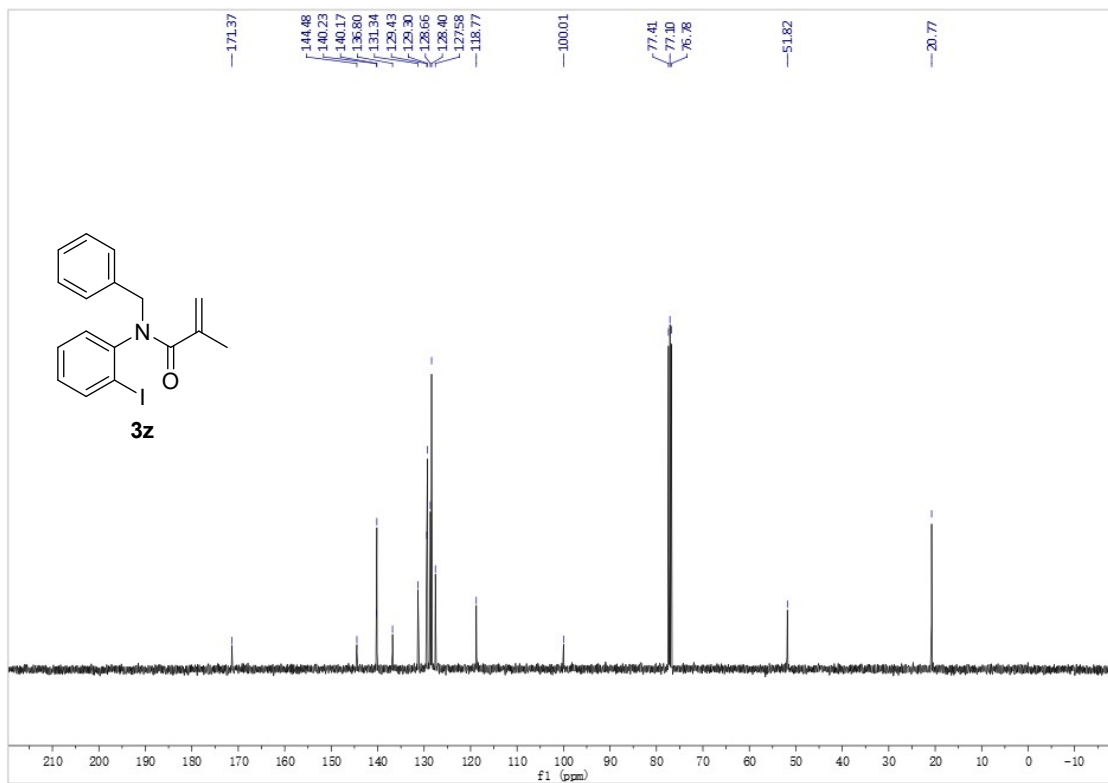


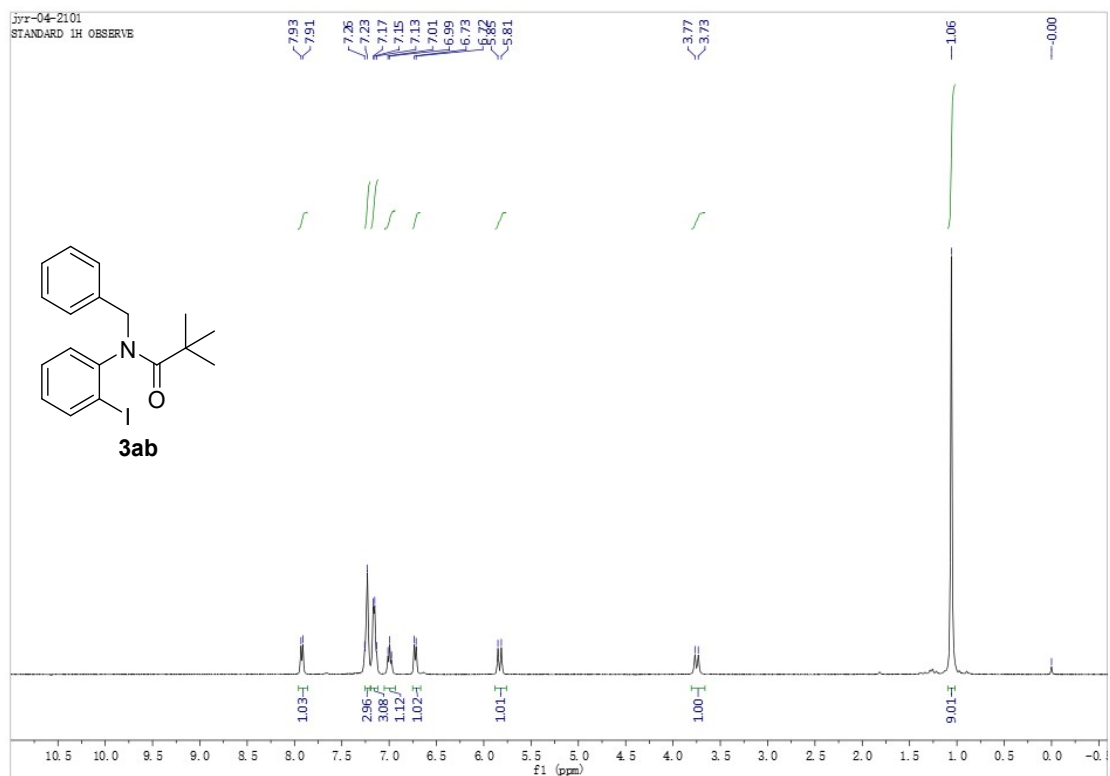
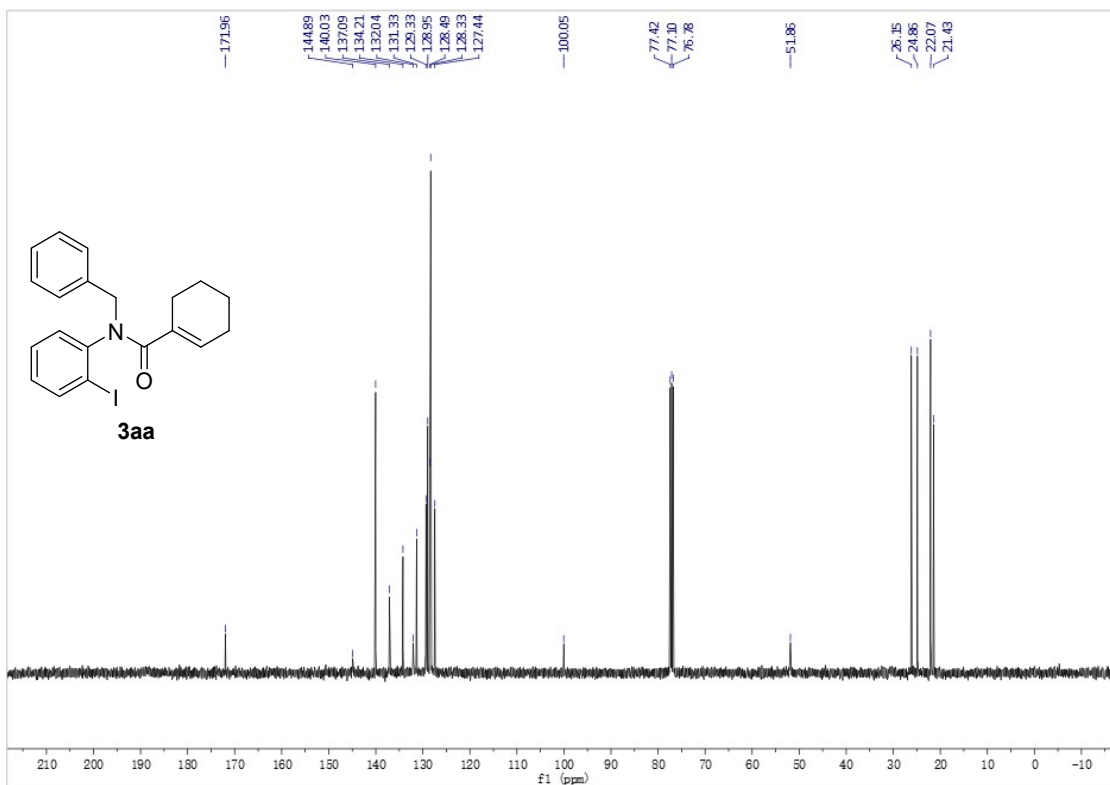


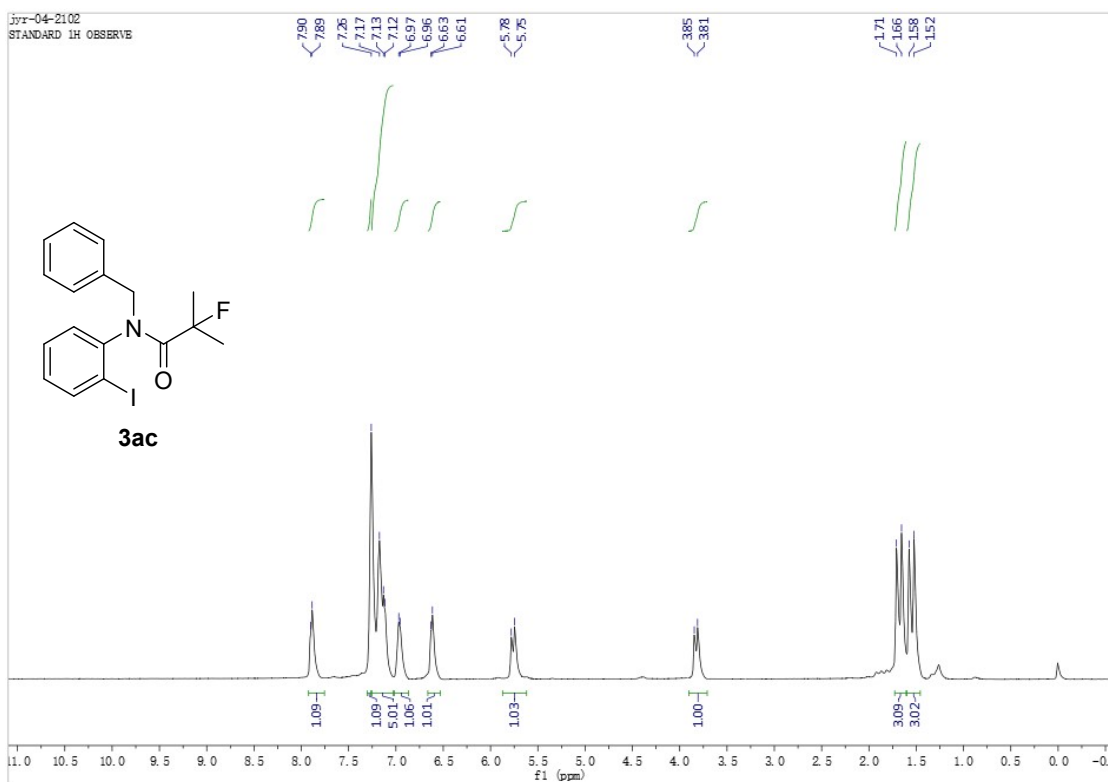
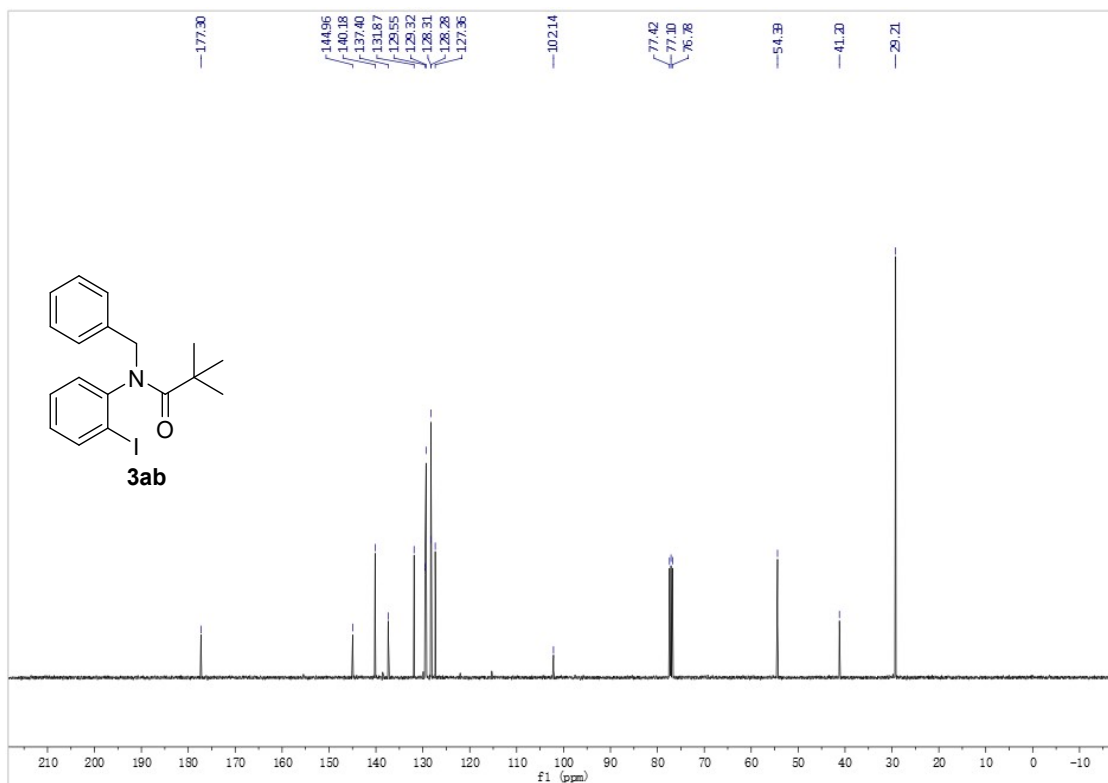


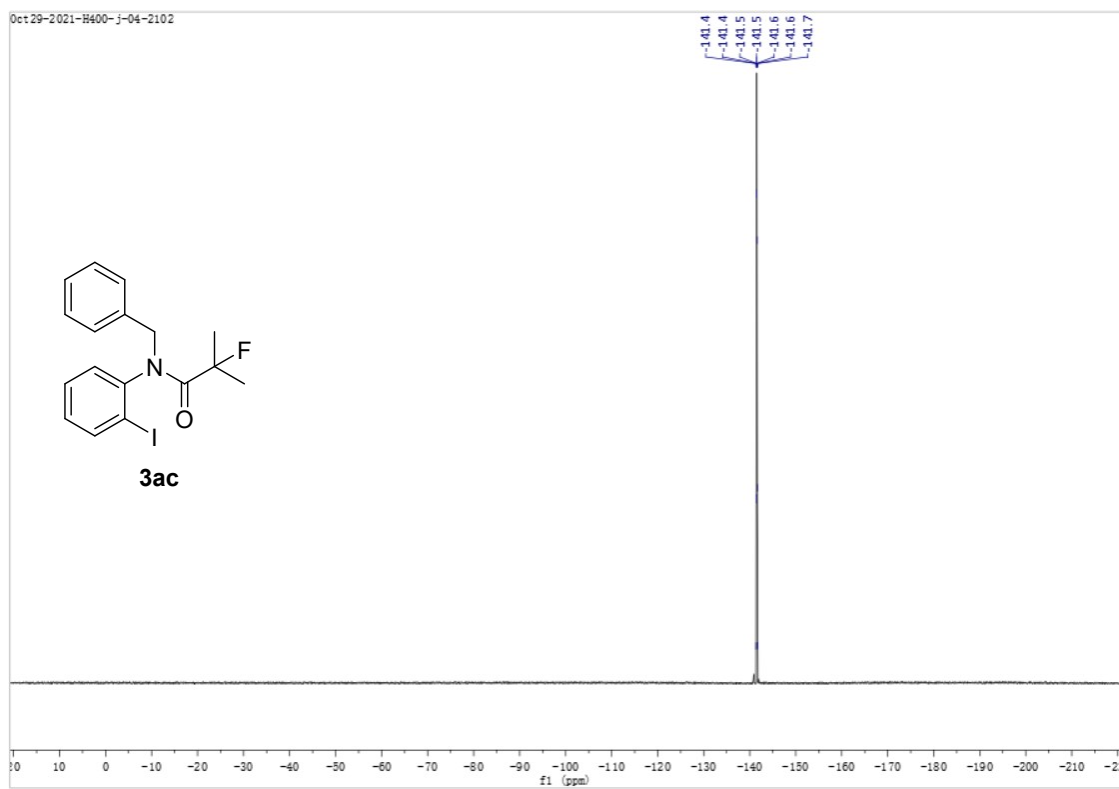
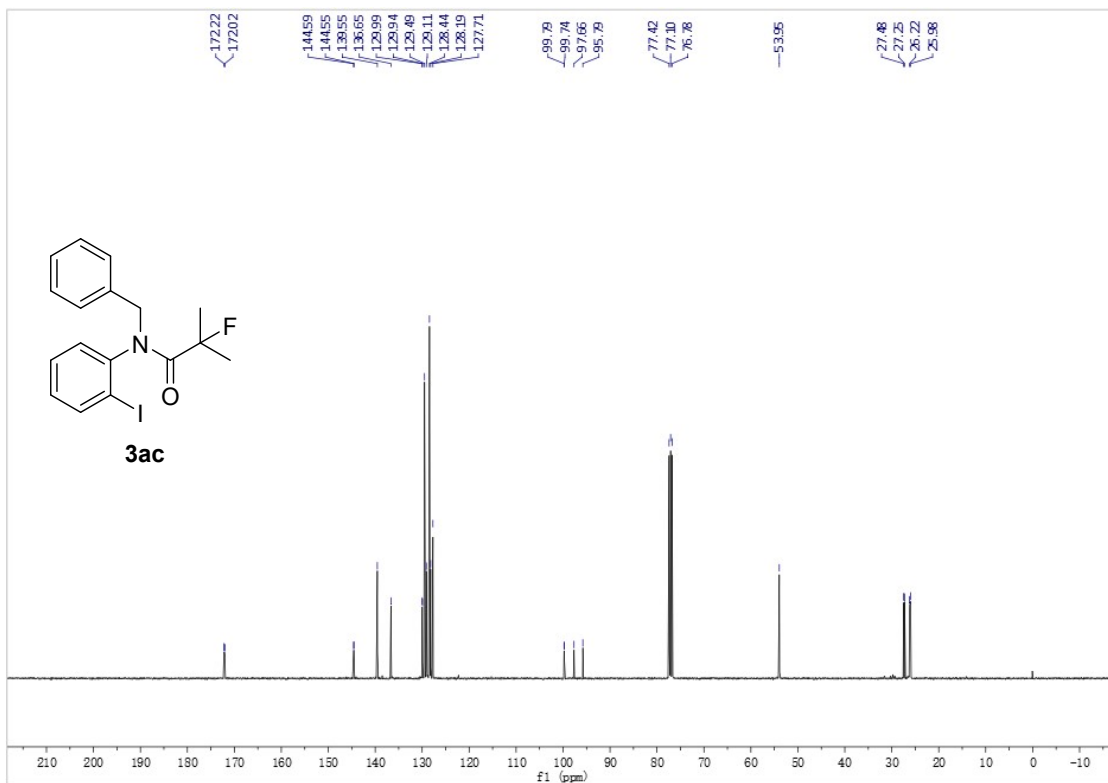


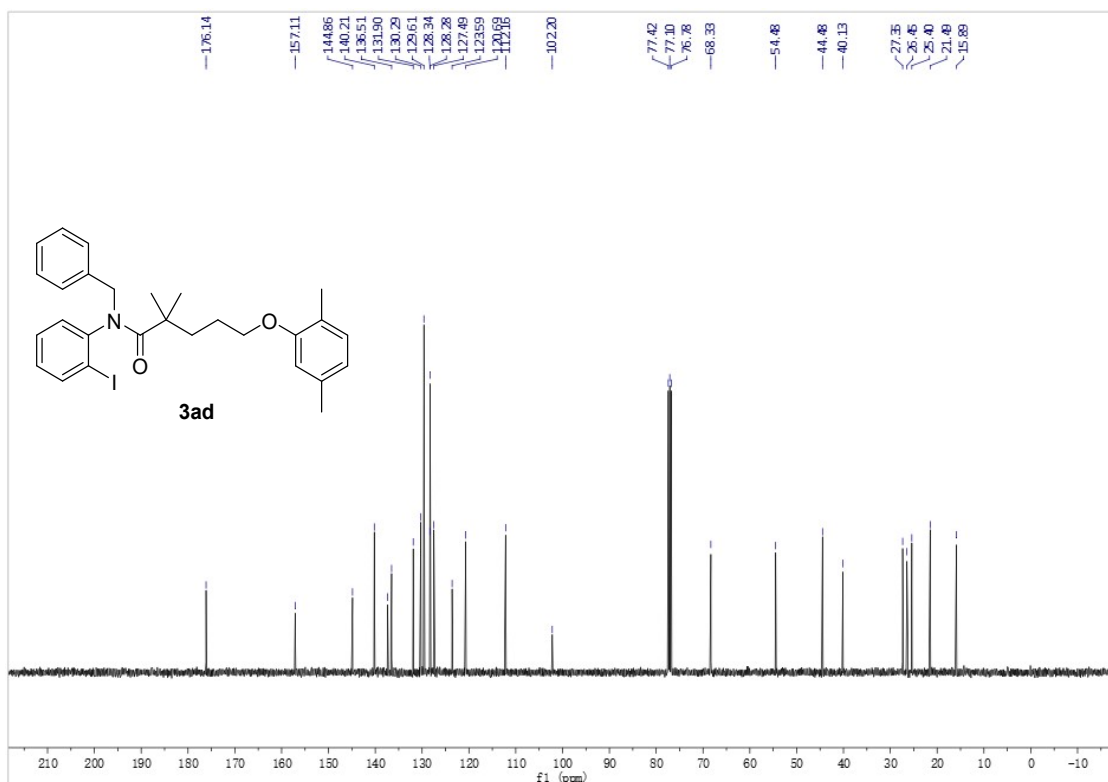
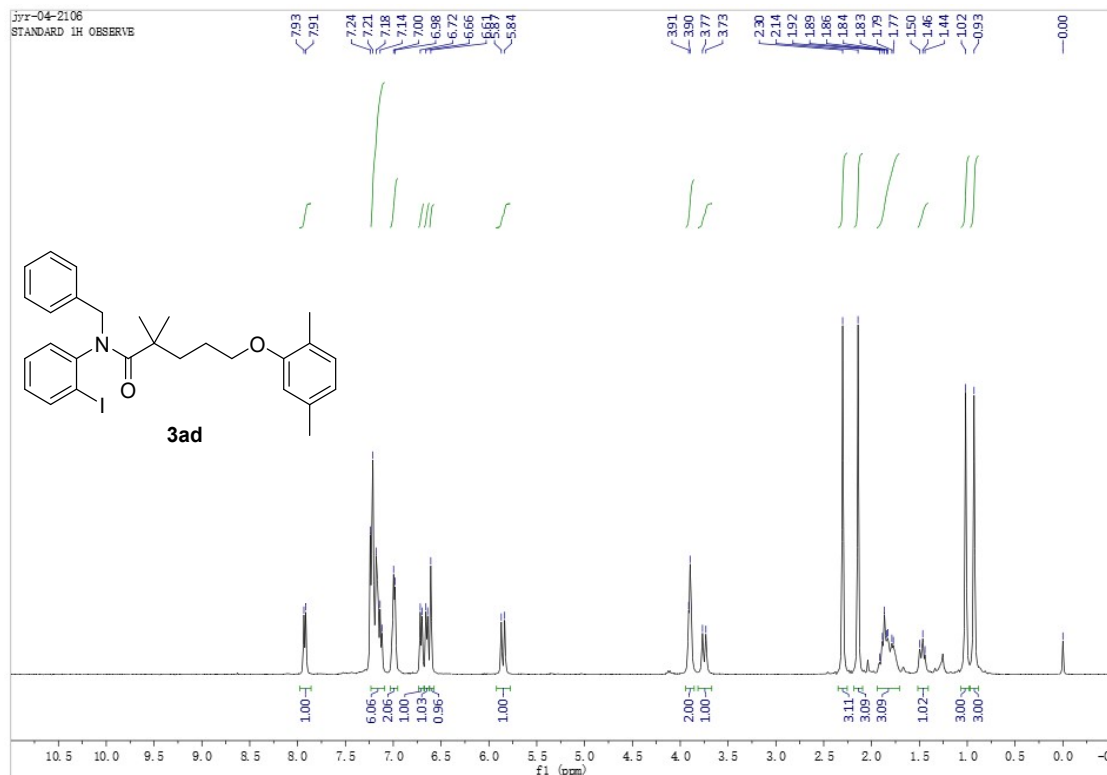


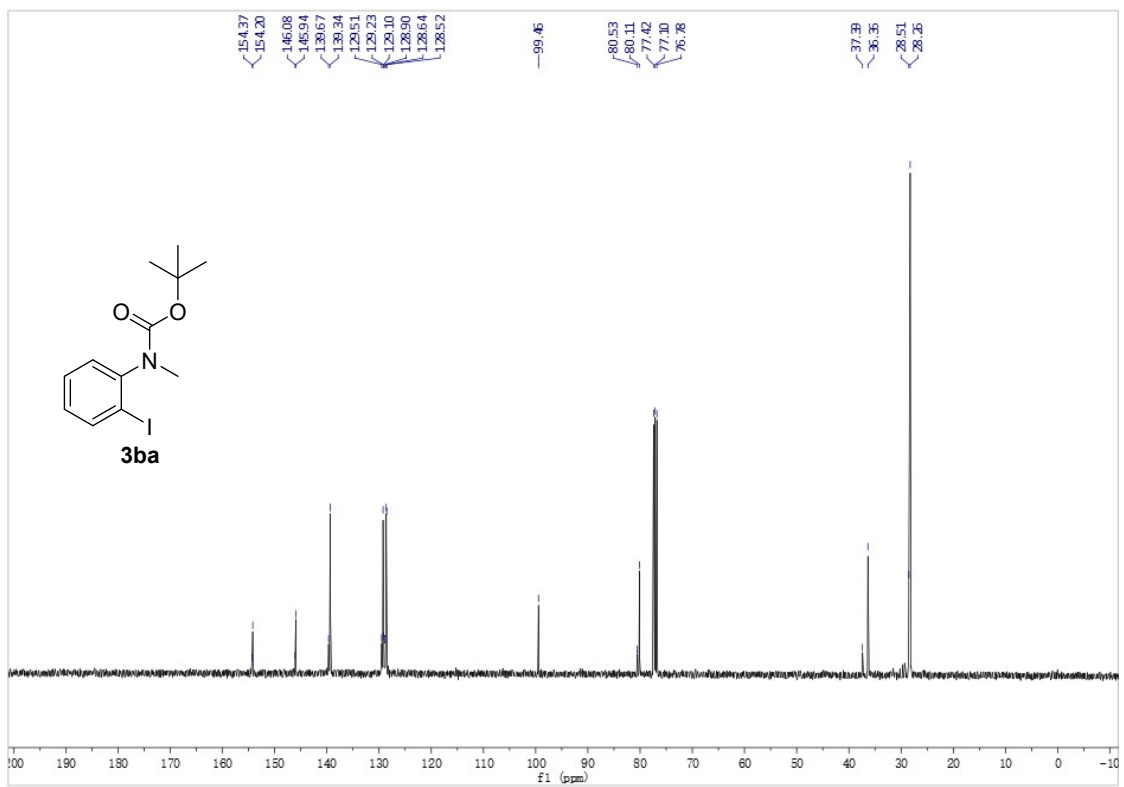
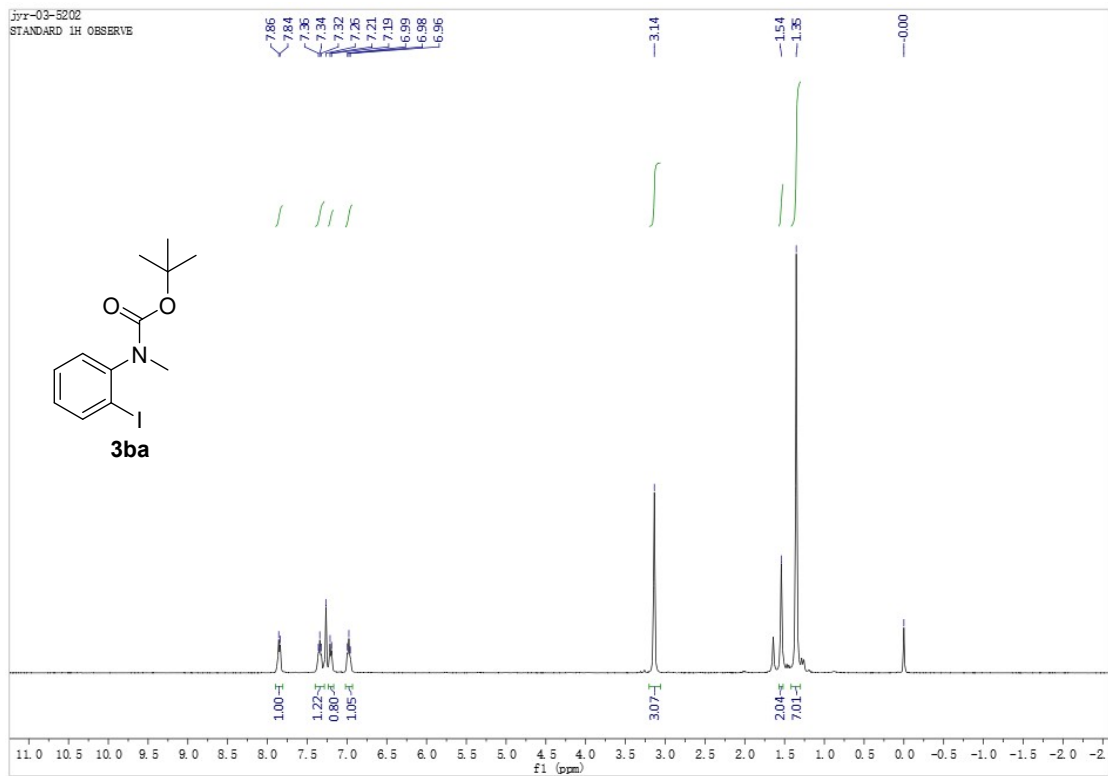


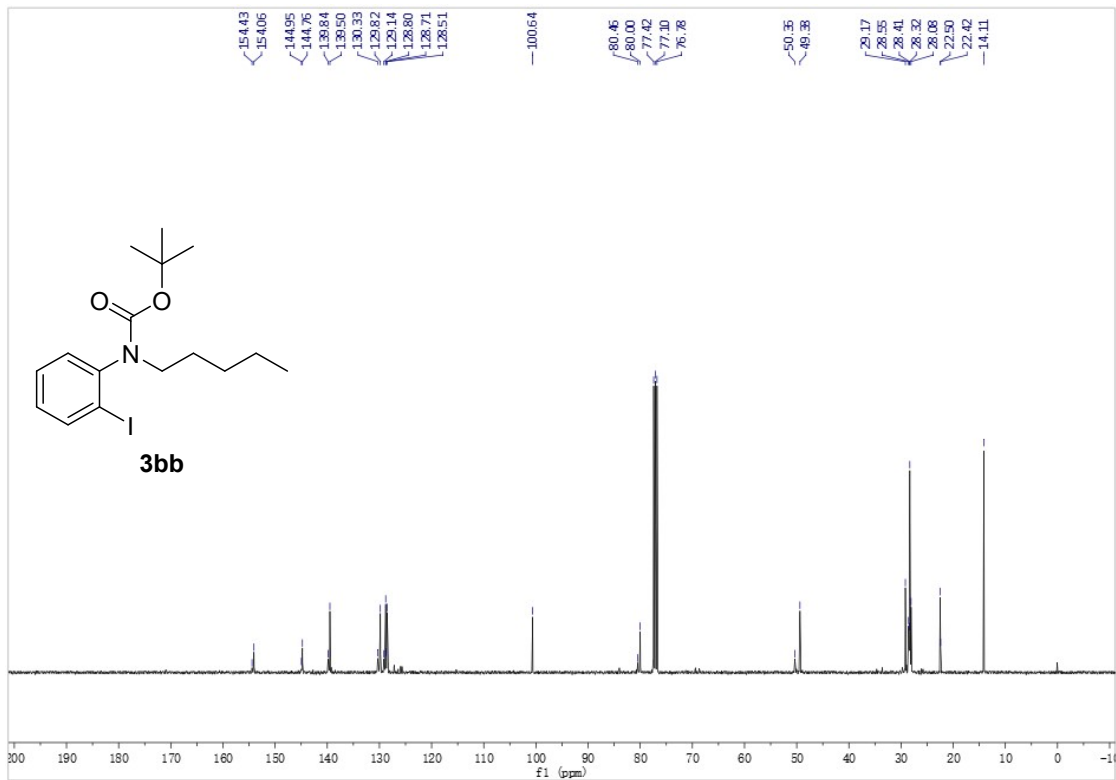
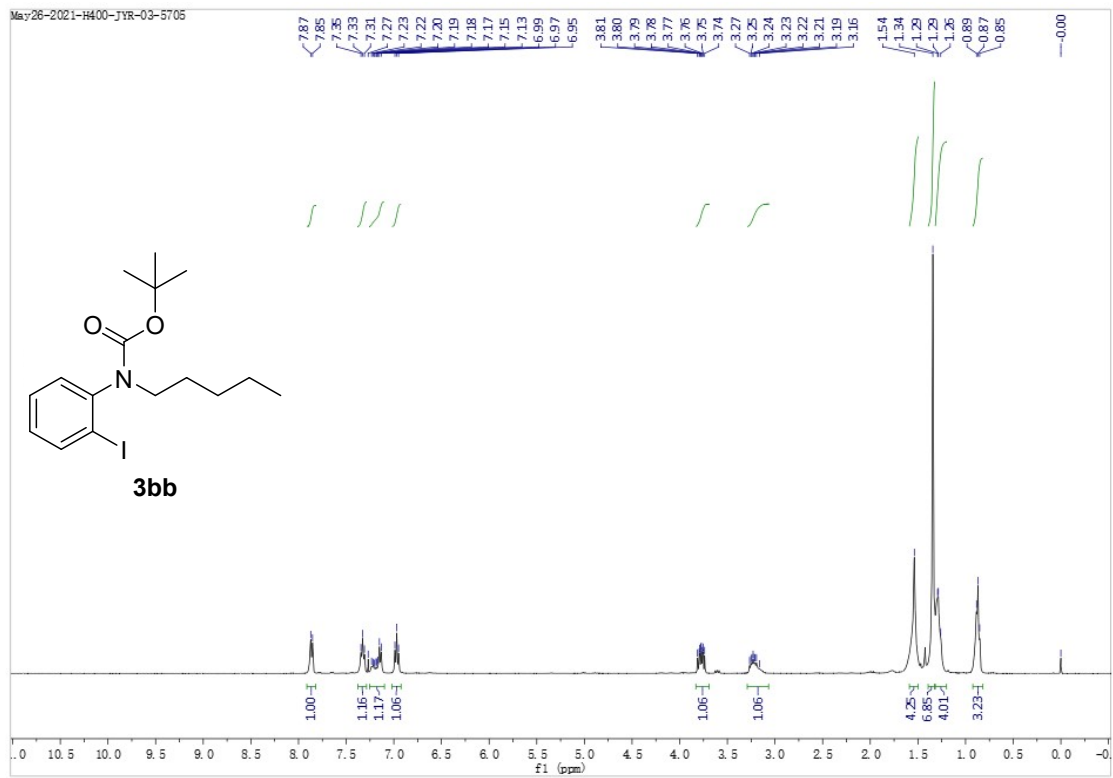


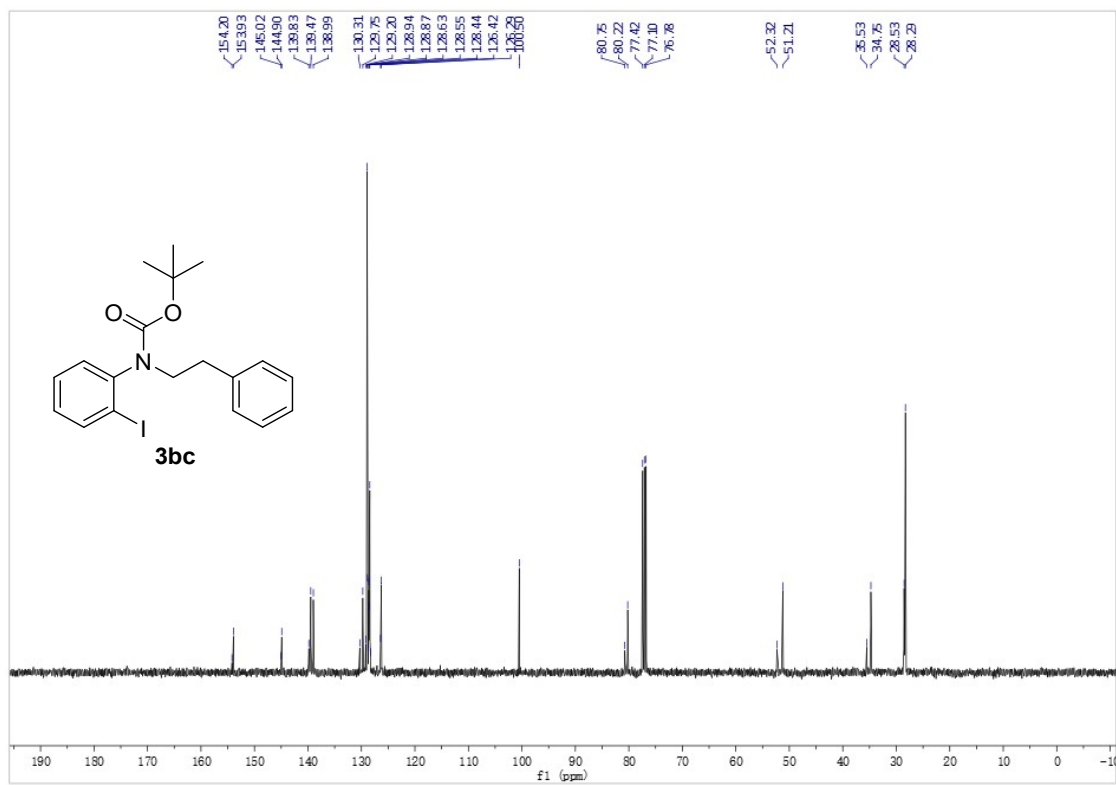
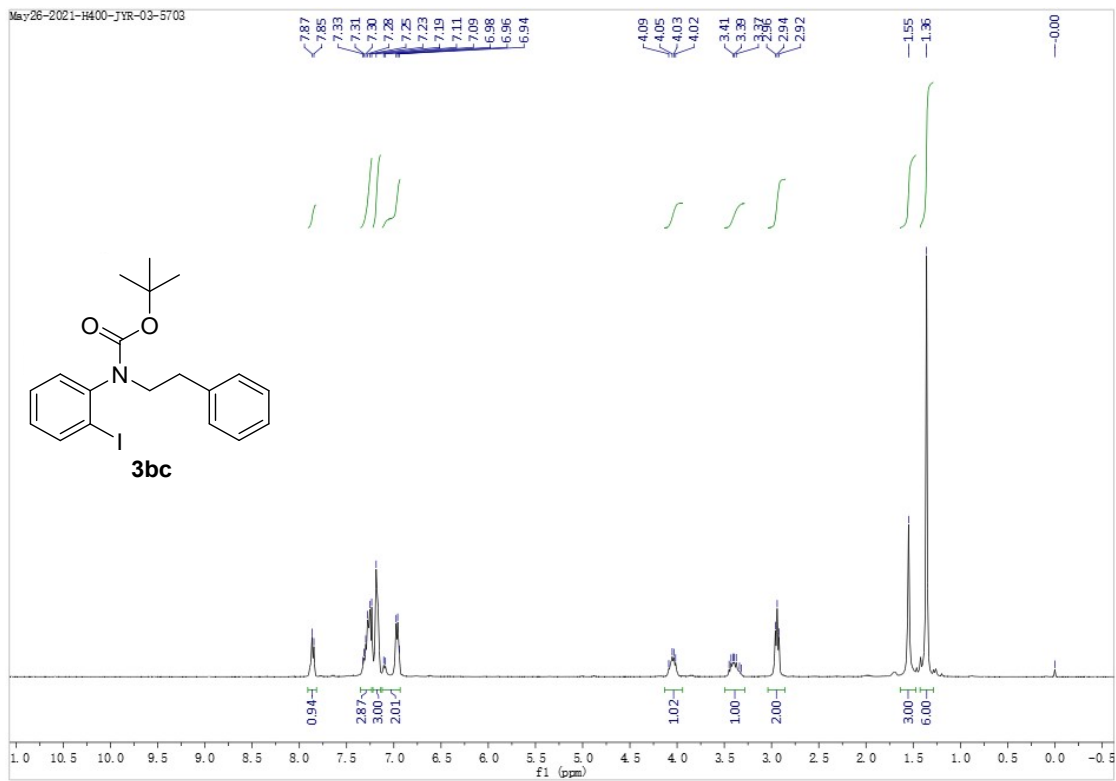


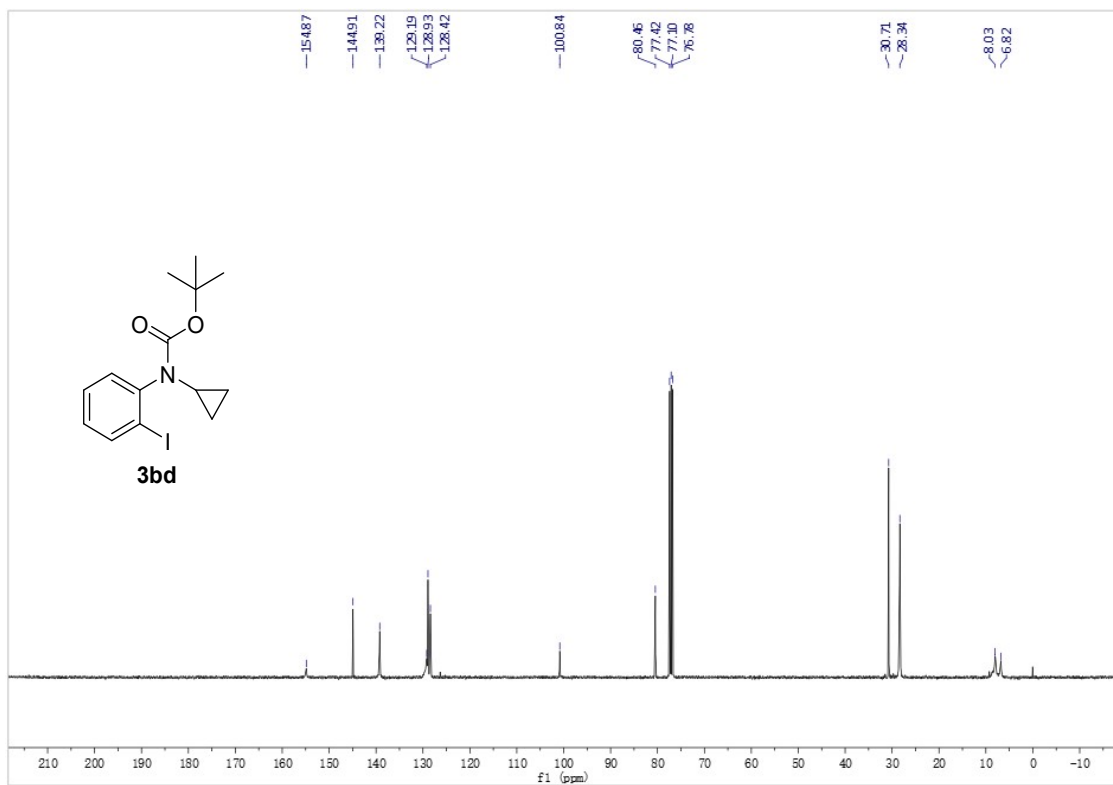
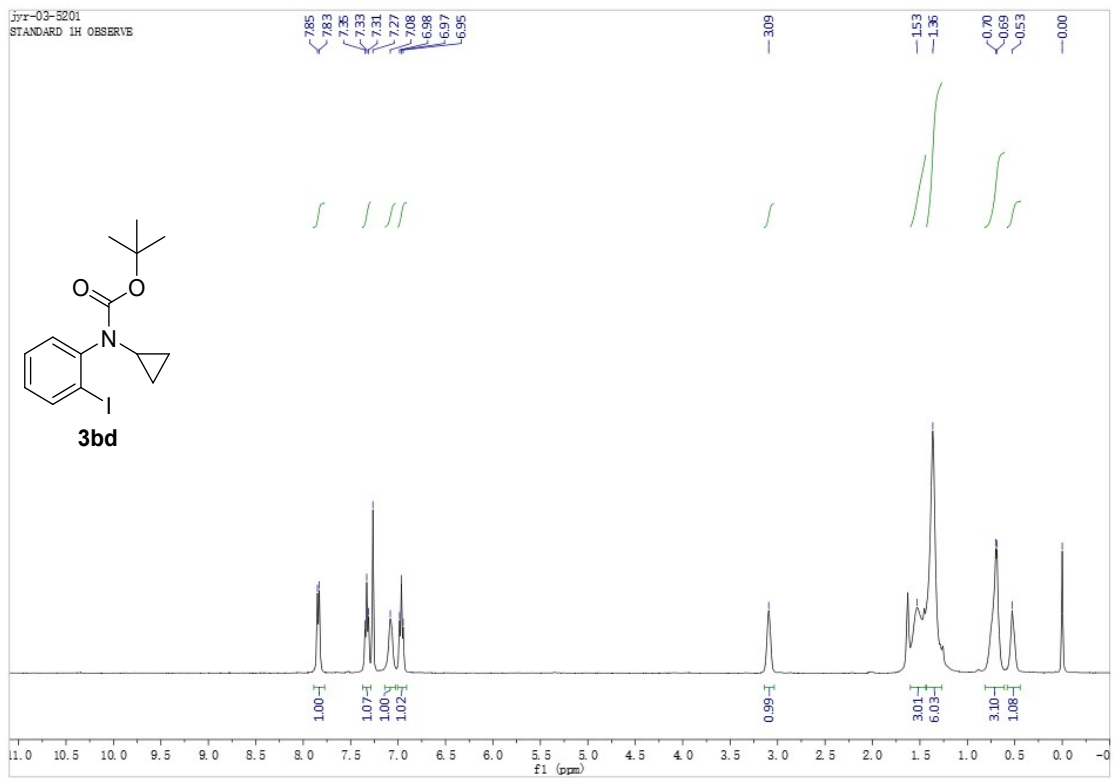


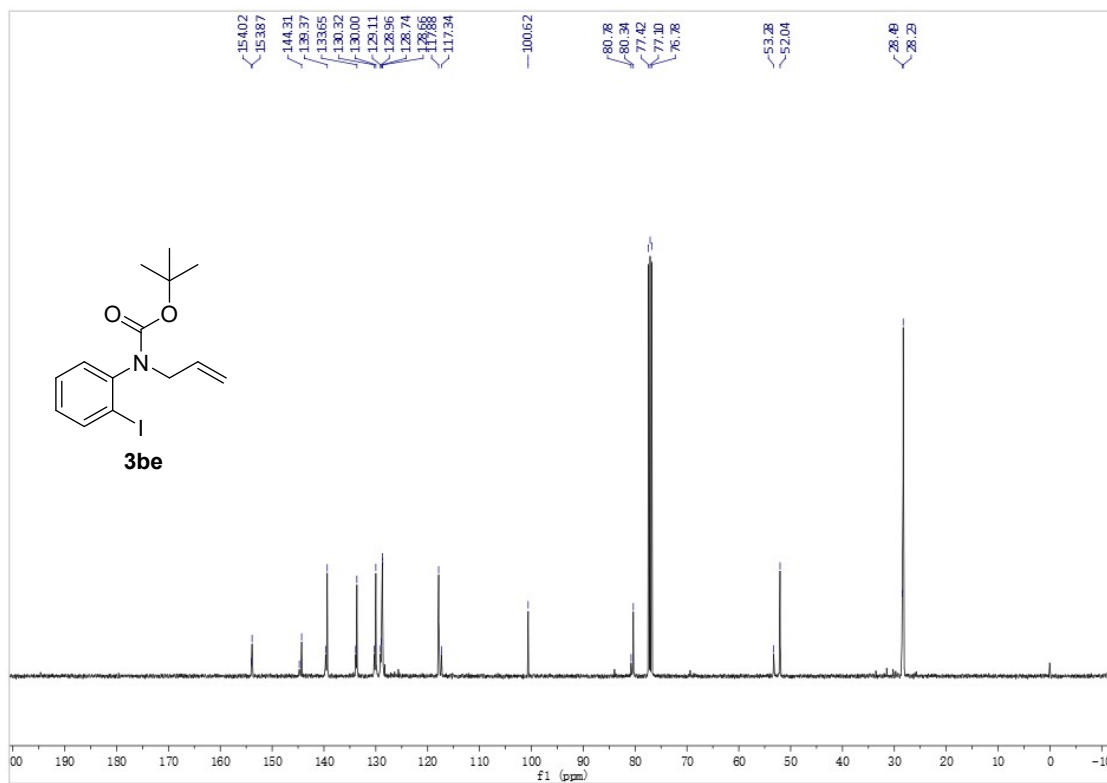
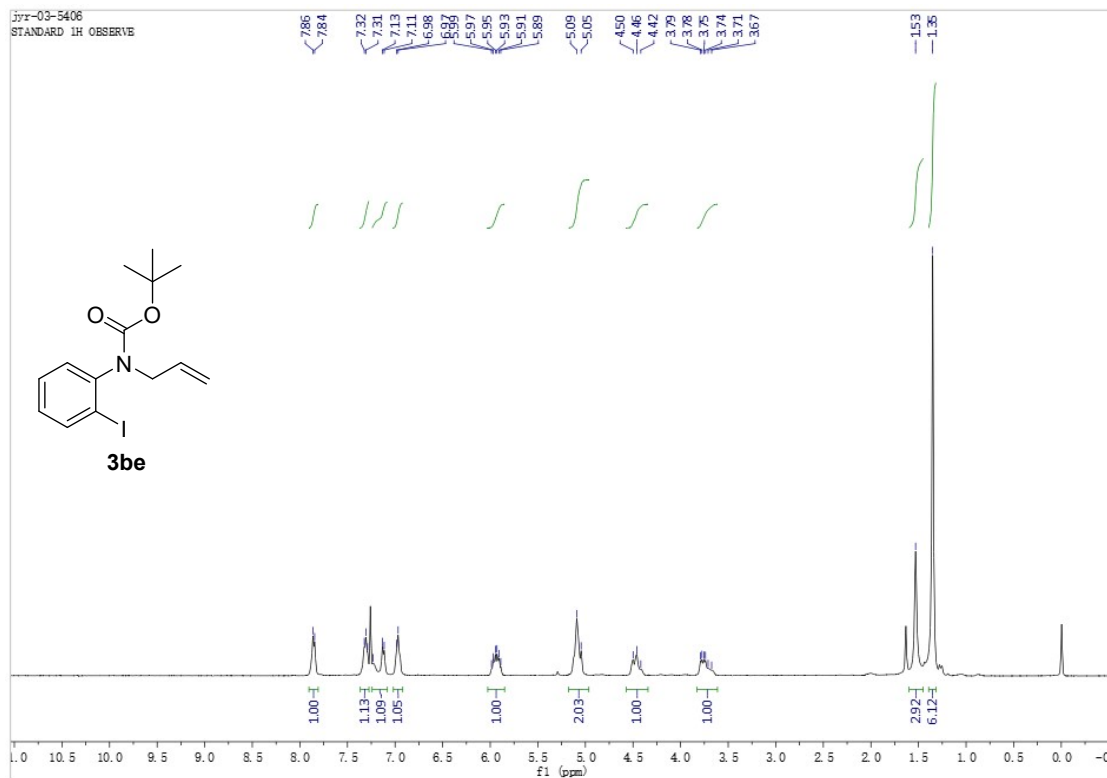




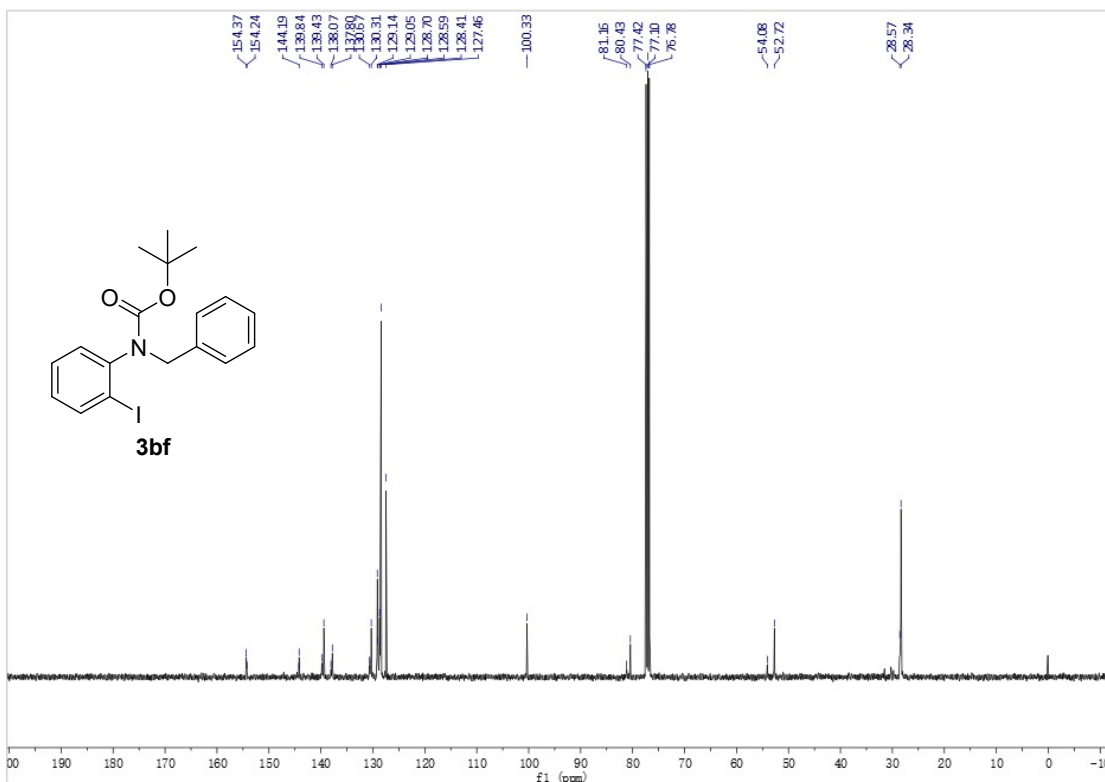
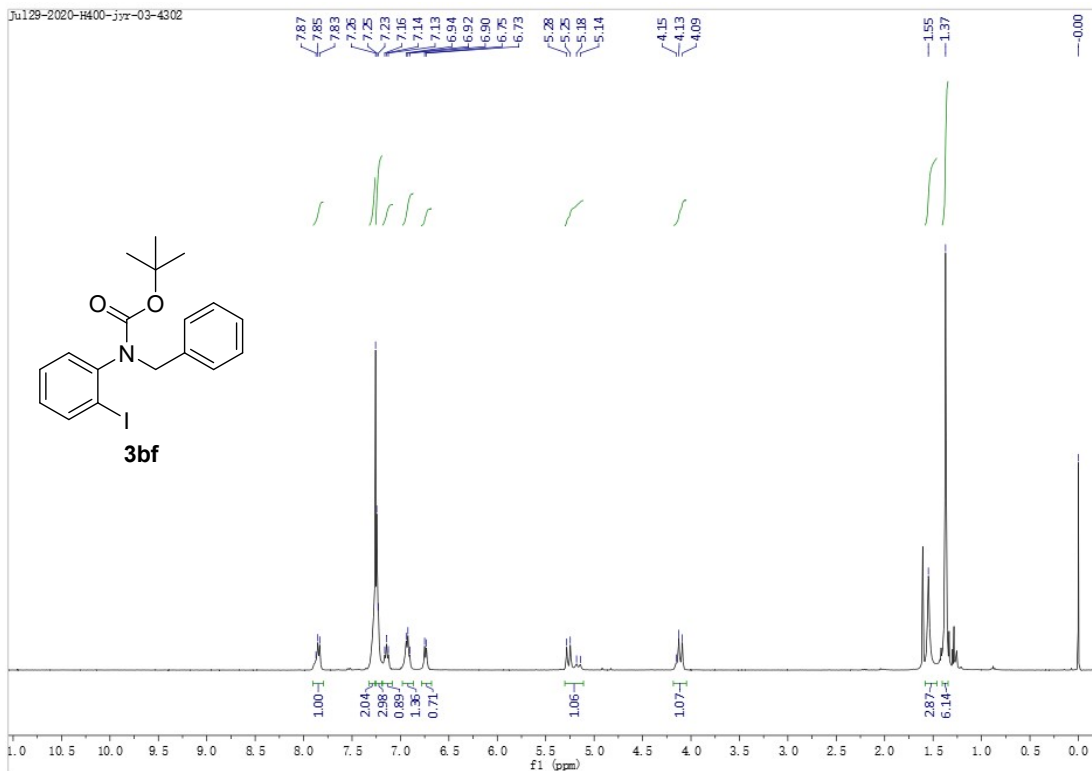


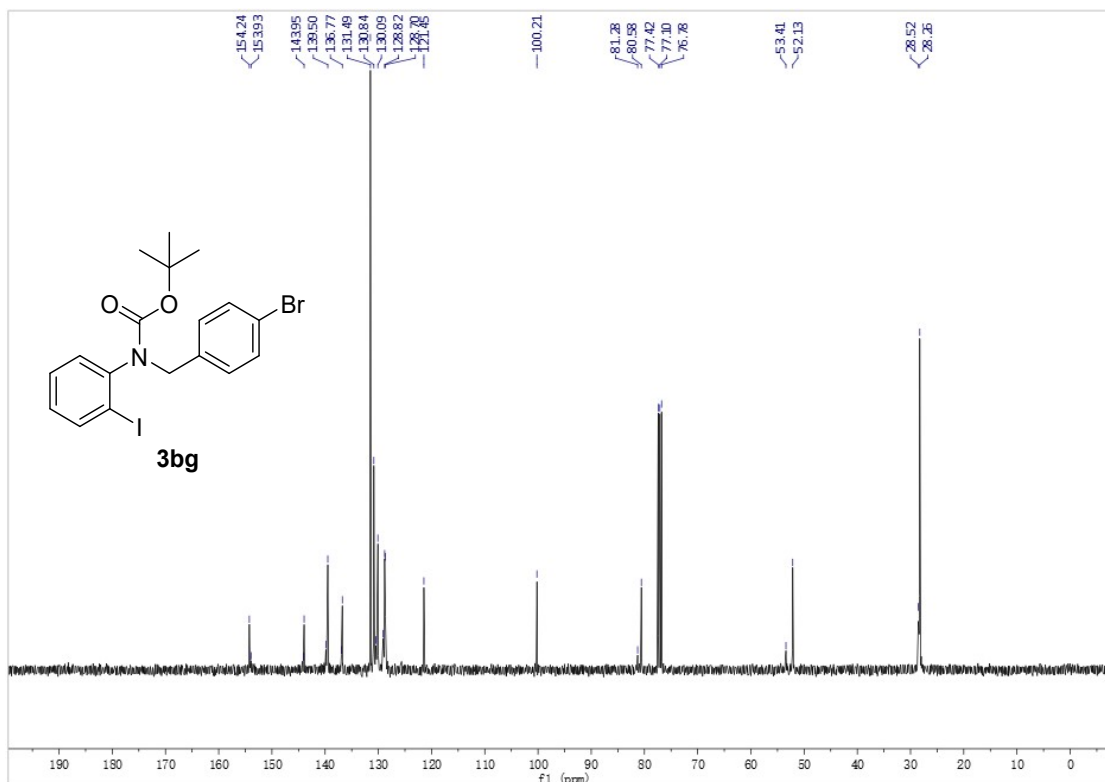
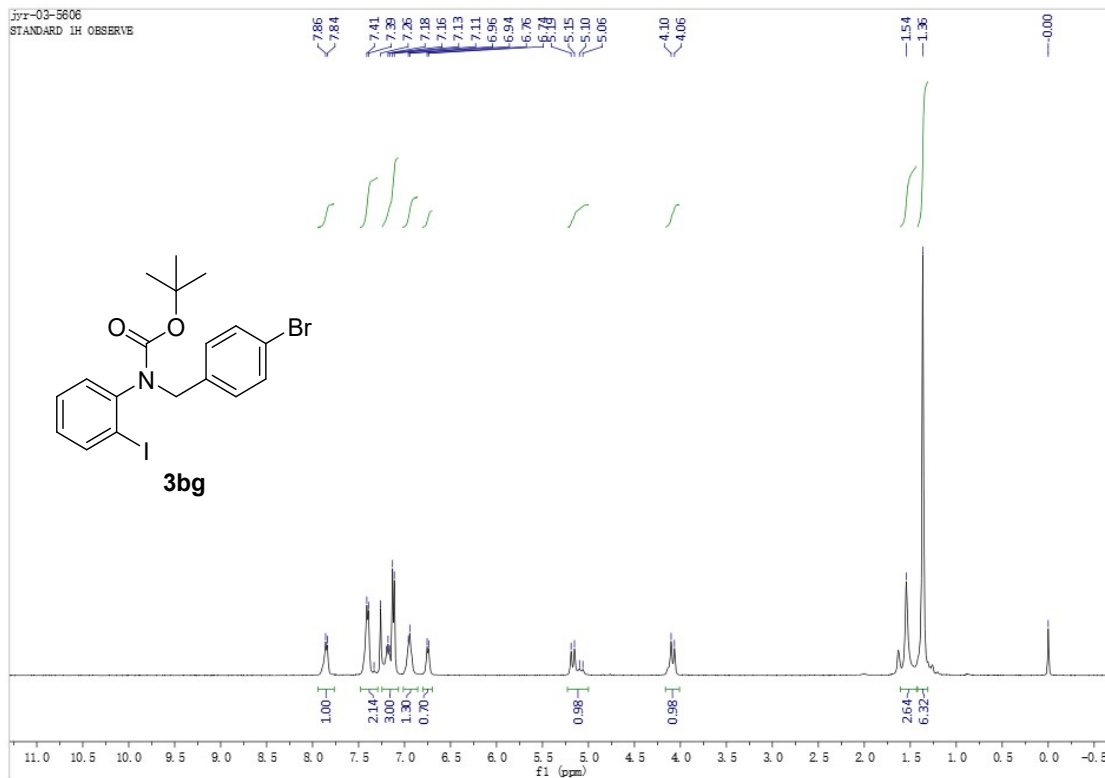




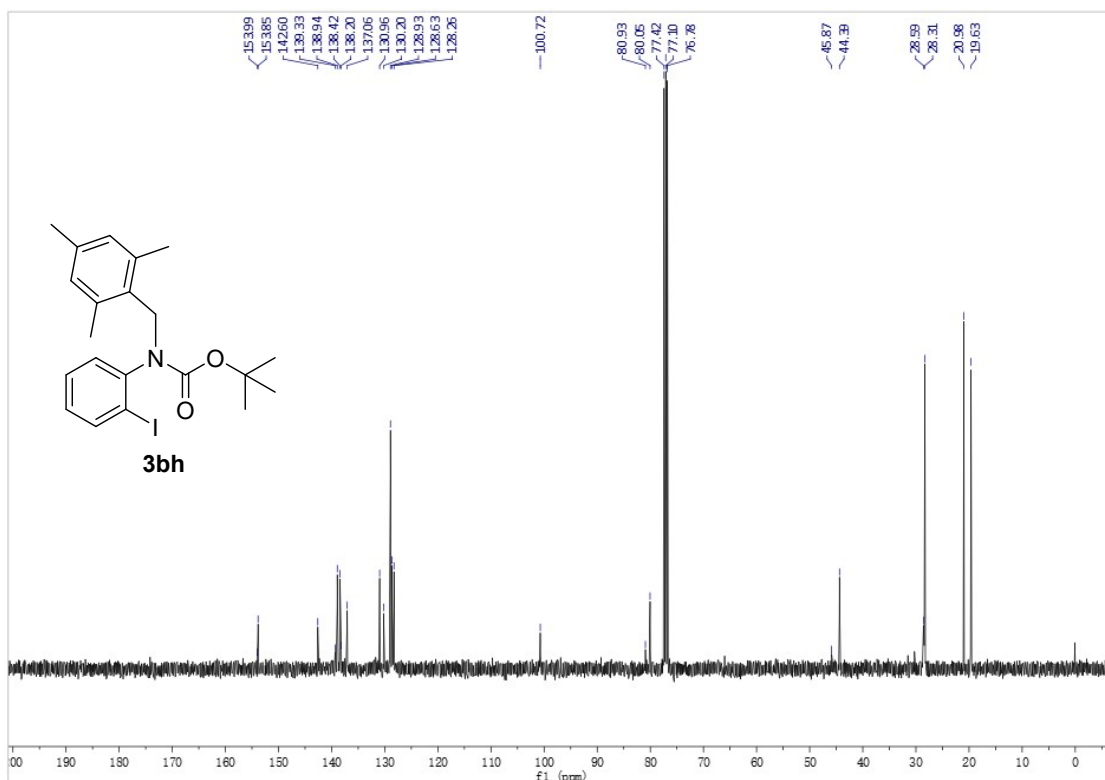
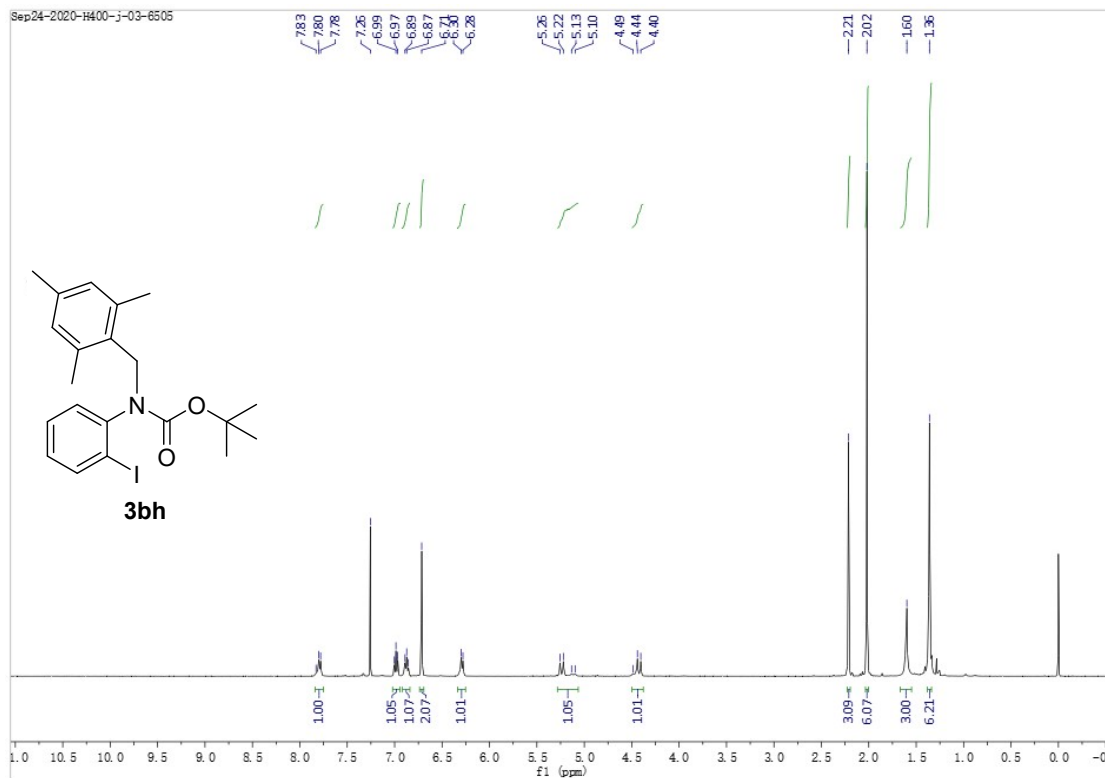


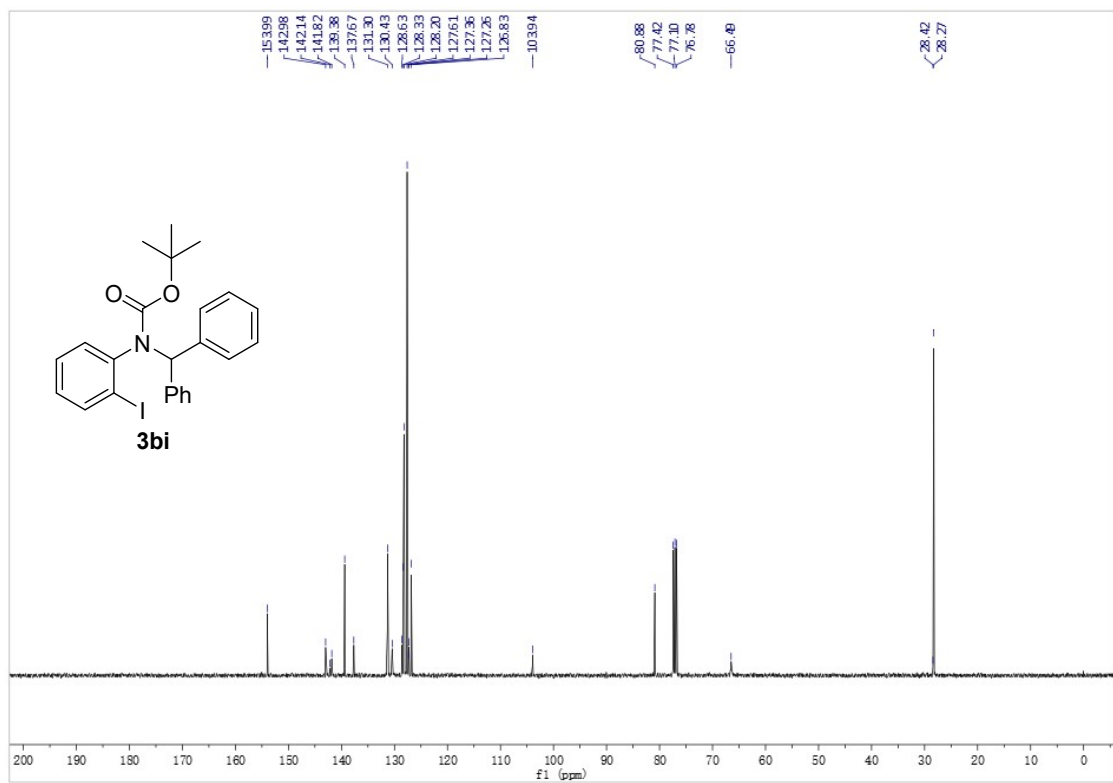
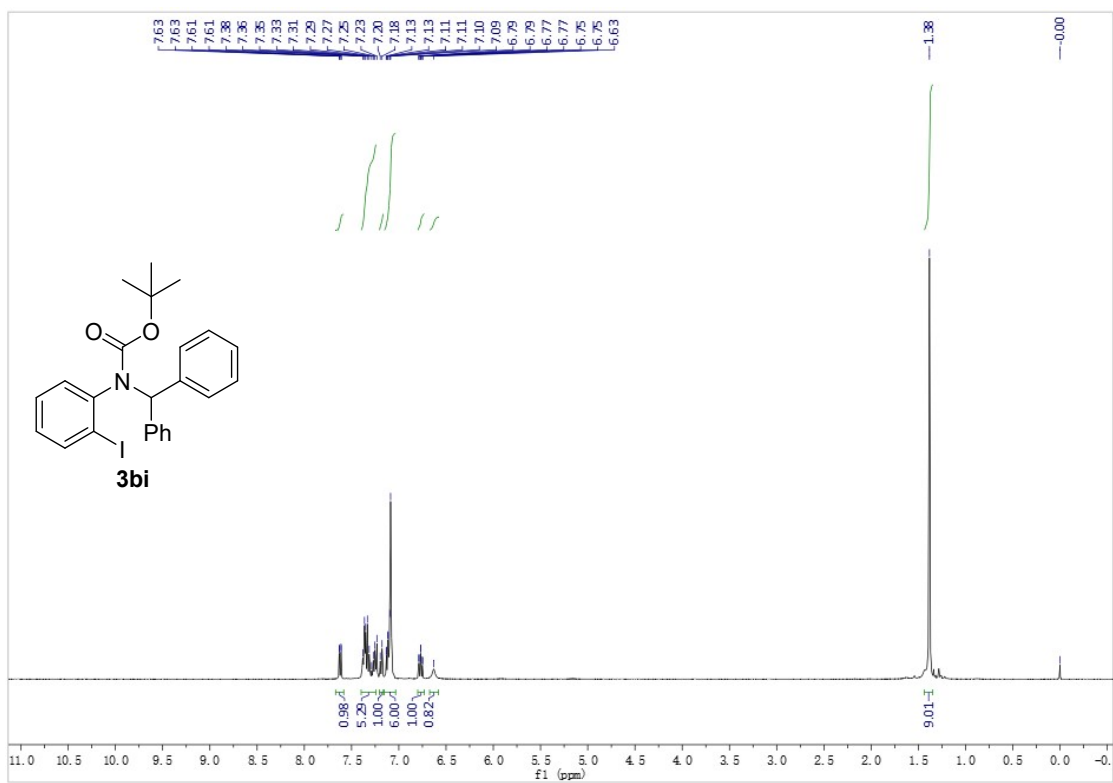
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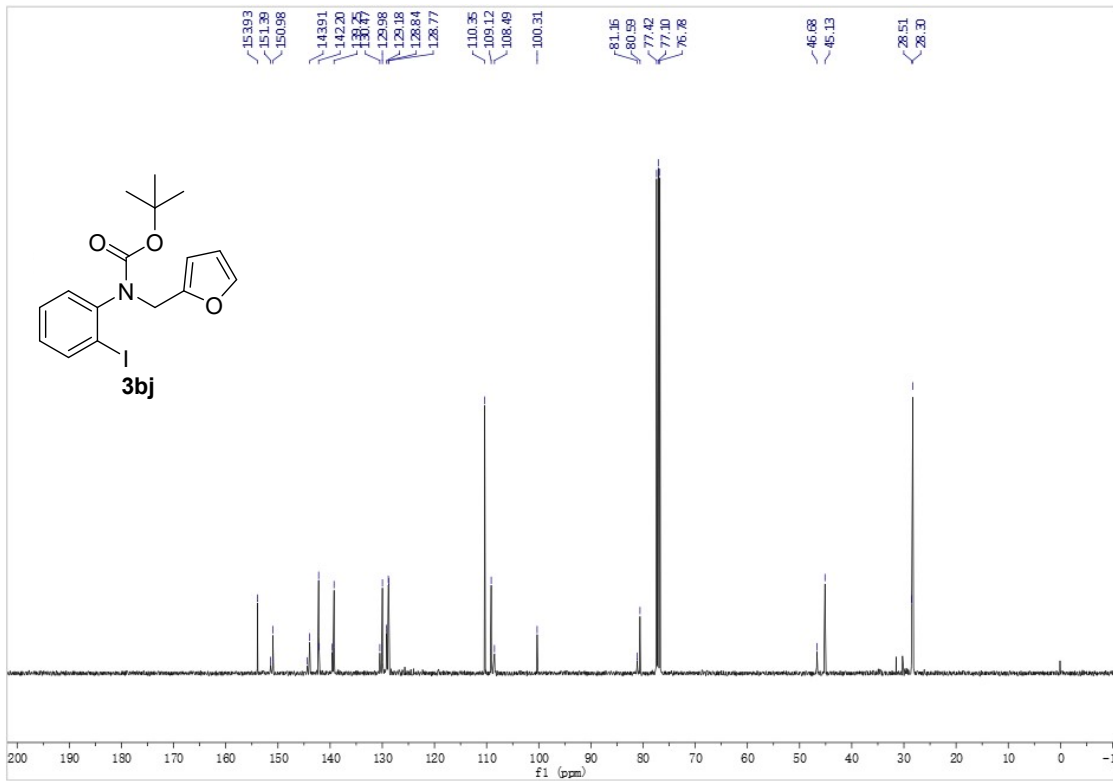
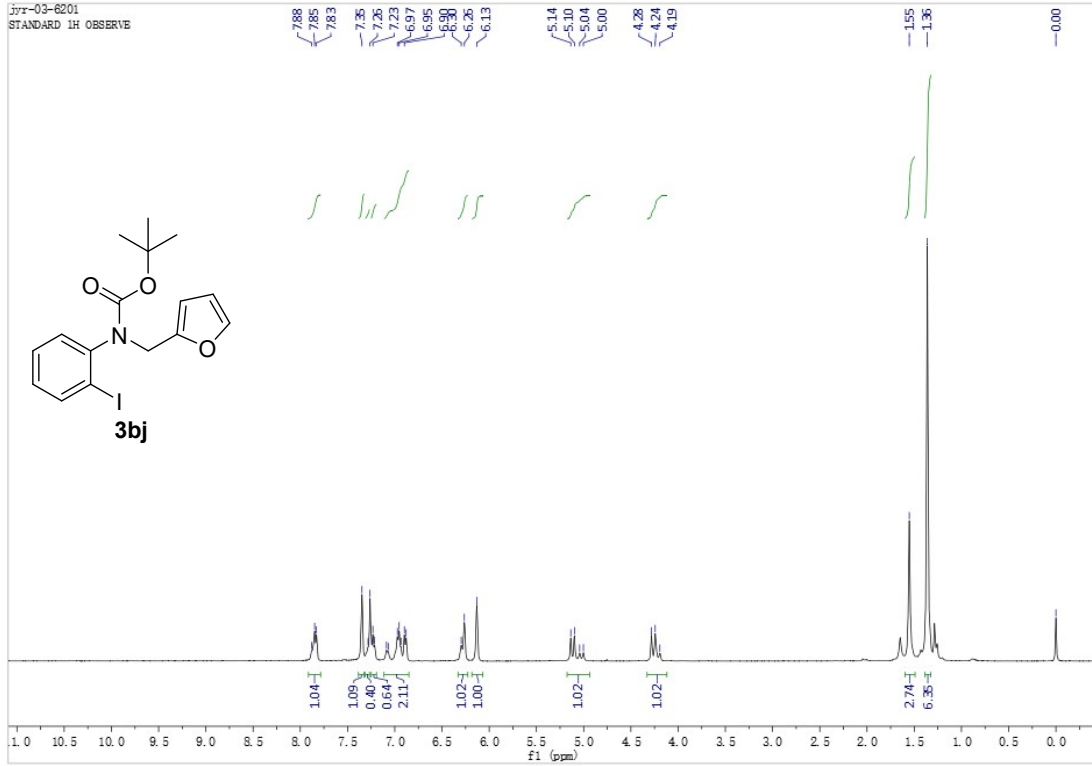


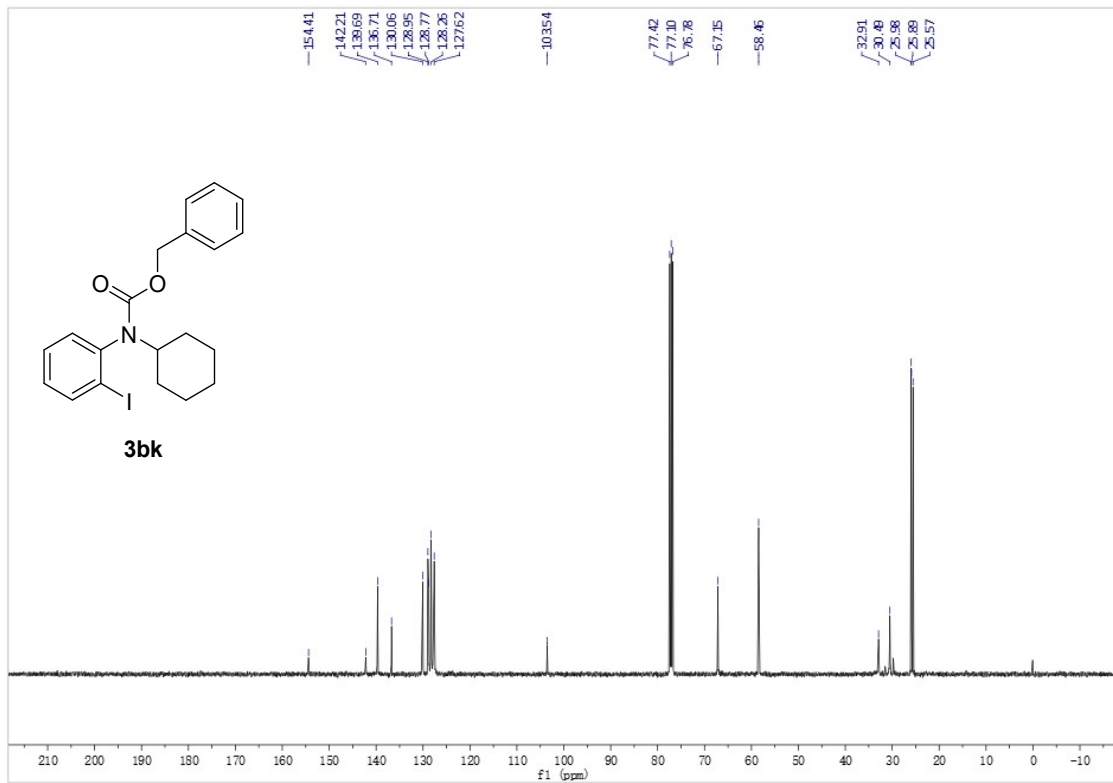
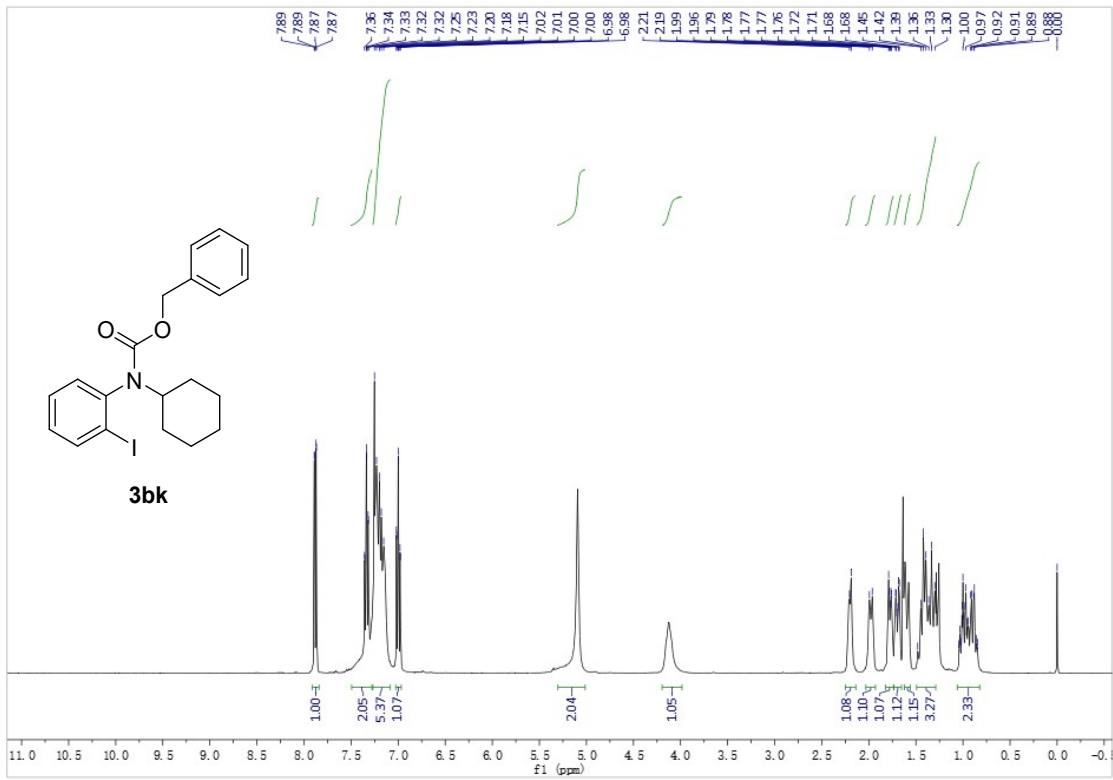


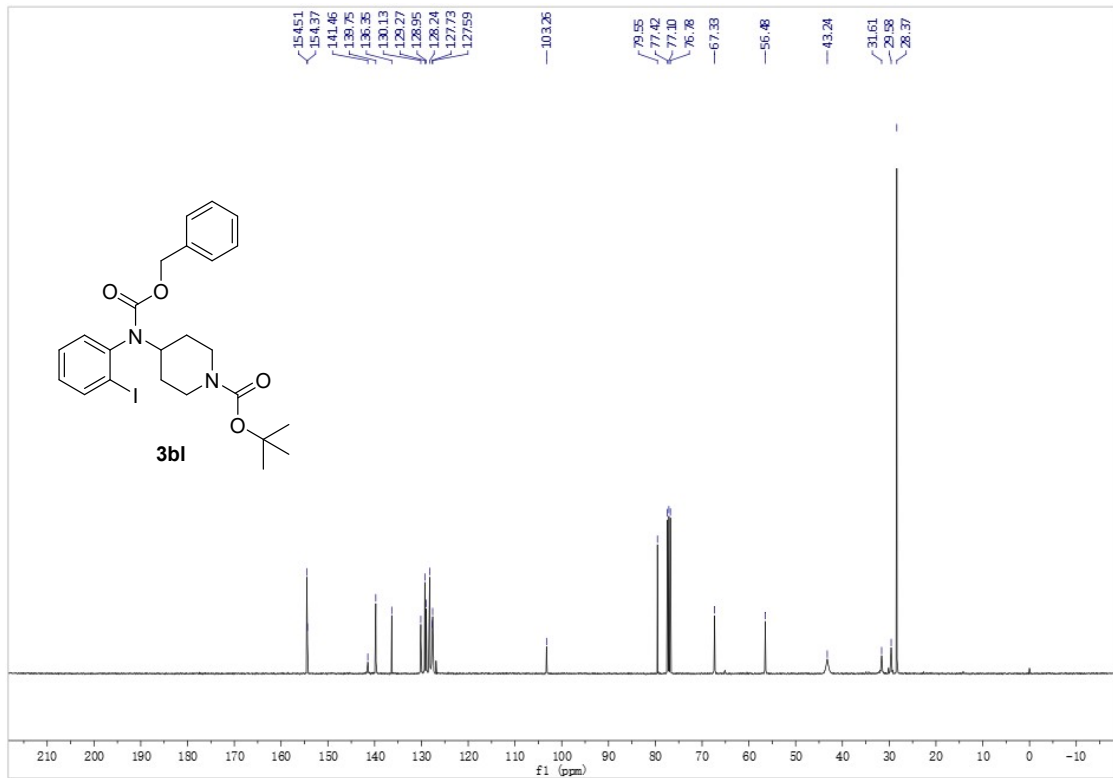
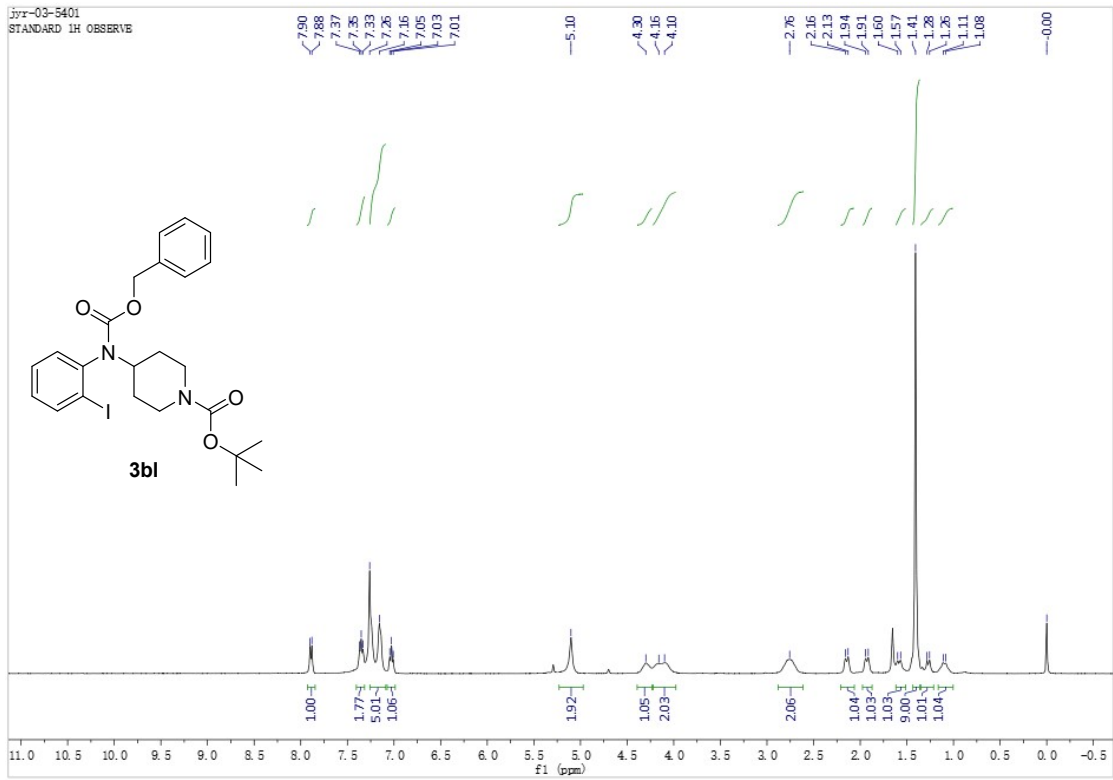
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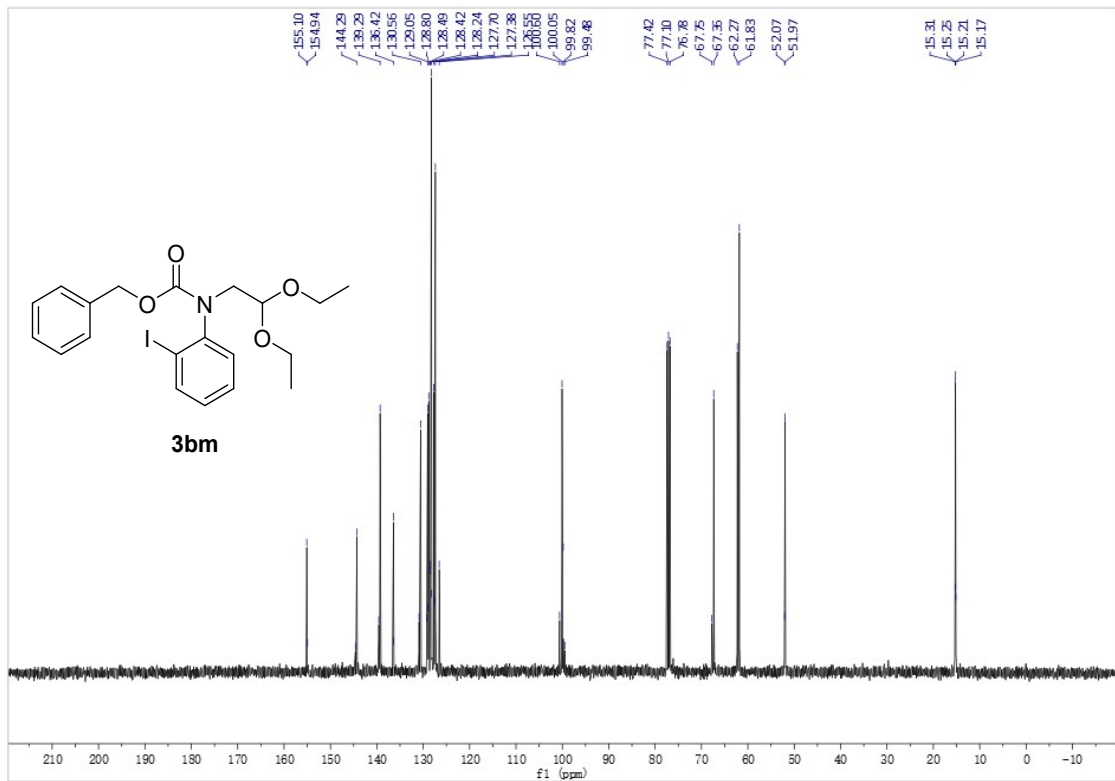
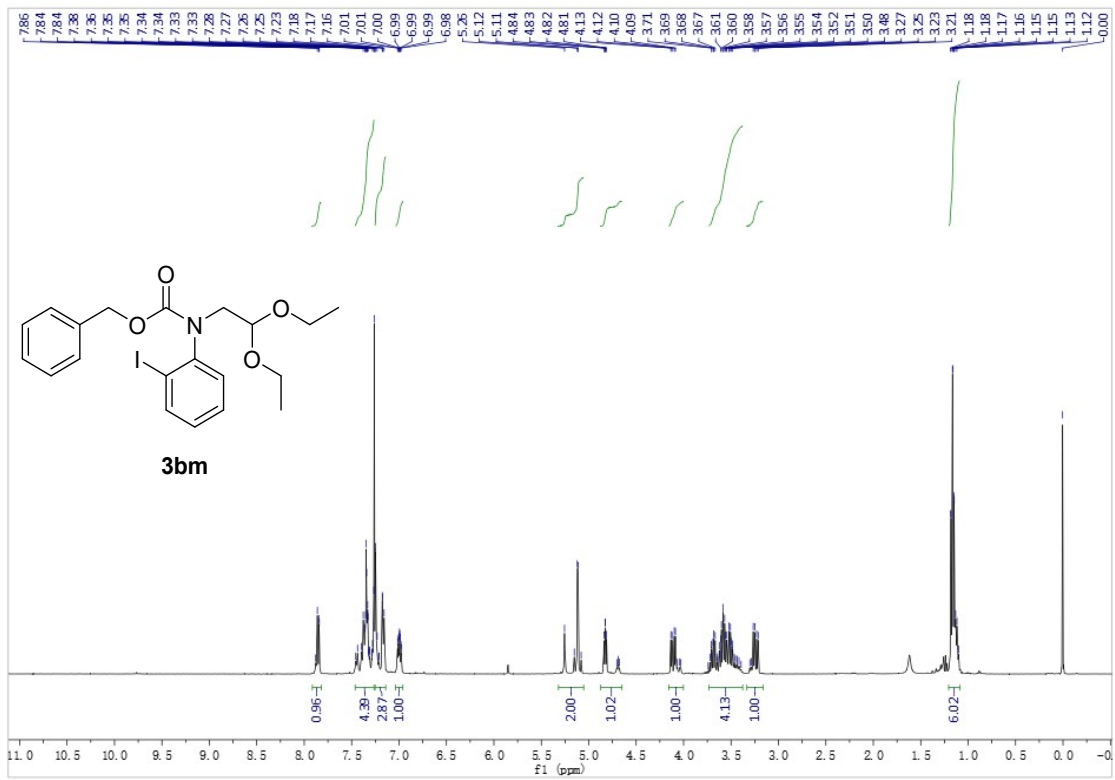


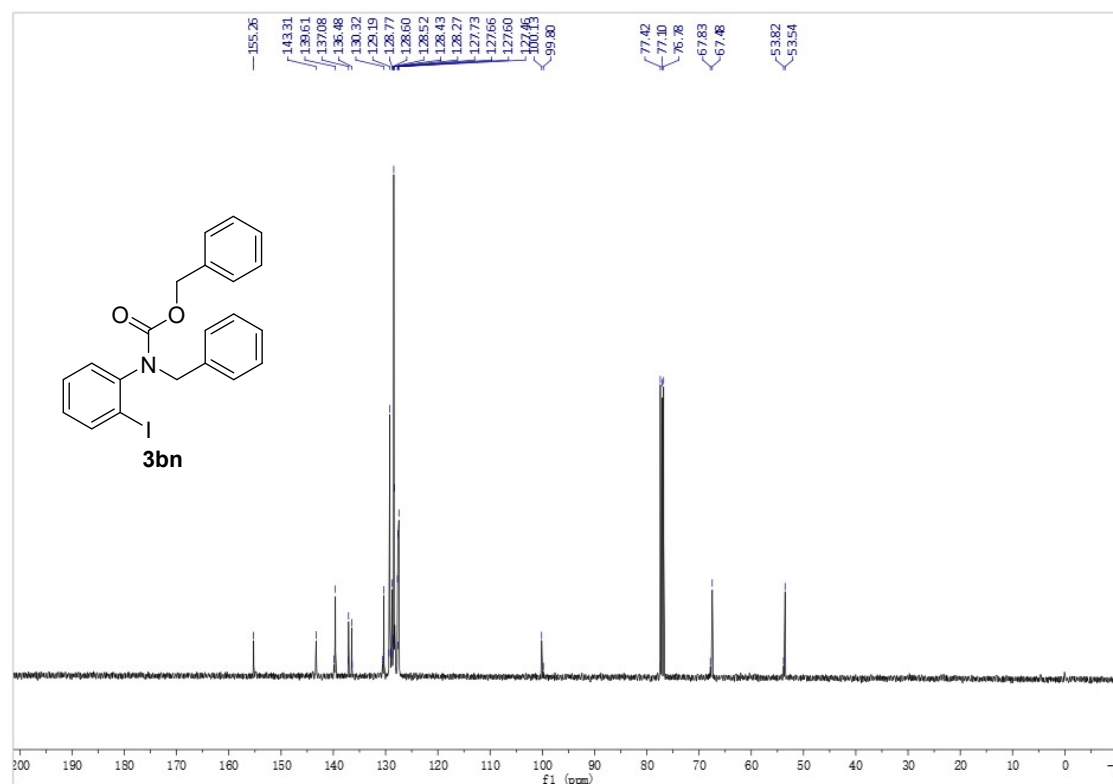
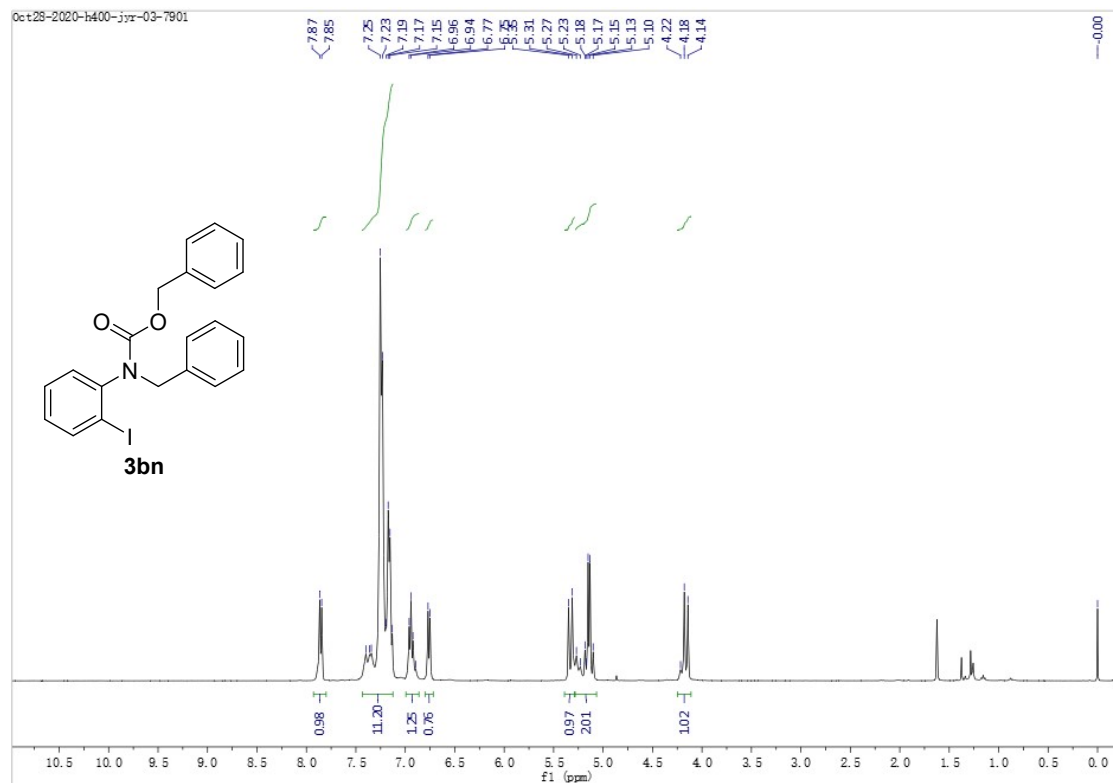












Oct-28-2020-H400-J-03-7902

