Synthesis of Unnatural α- Amino Acid Derivatives *via* Selective *o*-C-H Functionalization

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1. Procedure for the synthesis of compound 1a~1d

Procedure A for the synthesis of compound 1a and 1d. A suspension of α-phenylglycine or phenylalanine (10 mmol) in MeOH (30 ml) was cooled to 0 ºC in an ice-water bath and $SOCl_2$ (1.5 mL) added dropwise over 10 min. The clear solution was elevated to ambient temperature and stirred for 4 h, continuously evaporated with DCM. The solwent was removed under vacuum to give the crude amino acid ester hydrochloride as a white solid, which was used directly for the next step. (Reference：B. R. Aluri, B. Niaz, M. K. Kindermann, P. G. Jones and J. Heinicke, *Dalton Trans.*, 2011, **40**, 211-224.)

Pyridine-2-carboxylic acid (11 mmol) was treated with $SOCl_2$ (10.0 mL) at reflux temperature for 3 h. Excess $SOCl_2$ was then removed under vacuum to give the crude acid chloride. The crude acid chloride was diluted with dry CH_2Cl_2 (20 mL). A solution of the crude amino acid ester hydrochloride (10 mmol) and NEt_3 (40 mmol) in dichloromethane (20 mL) was added dropwise to the acid chloride solution at 0 °C. The resulting mixture was allowed warm to r.t., and then stirred overnight. The mixture was quenched with saturated $NaHCO_3$ solution and extracted with CH_2Cl_2 three times. These extracts were combined and dried over $MgSO_4$. After evaporation in vacuo, the crude amide product was purified by silica gel chromatography using CH_2Cl_2 /ethyl acetate as the eluent to give the desired product. (Reference：R. Shang, L. Ilies, S. Asako and E. Nakamura, *J. Am. Chem. Soc.*, 2014, **136**, 14349-14352.)

Procedure B for the synthesis of compound 1b. methyl 2-(4-hydroxyphenyl)-2-(picolinamido) acetate (**1e**; 1.43 g, 5 mmol) was dissolved in 20 mL of dichloromethane and treated sequentially with imidazole (374 mg, 5.5 mmol) and tertbutyldimethylsilyl chloride (829 mg, 5.5 mmol). The reaction was stirred for 16 h at room temperature and concentrated to remove dichloromethane. The

crude oil was then taken up in EtOAc and washed with saturated aqueous $NaHCO_3$ (2x), water (1x), saturated aqueous NaCl (1x), and dried over $MgSO_4$. Concentration and through drying under vacuum yielded 85% of **1b** as a yellow oil. (Reference： J. S. Oakdale, L. Kwisnek and V. V. Fokin, *Macromolecules*, 2016, **49**, 4473-4479.)

Procedure C for the synthesis of compound 1c. A 250 mL two-necked flask equipped with a stir bar and a condenser coil was charged with anhydrous potassium carbonate (5 mmol, 691 mg), methyl 2-(4-hydroxyphenyl)-2-(picolinamido) acetate (**1e**; 1.43 g, 5 mmol), acetone (30 mL), and (2-bromoethyl)benzene (5 mmol, 855 mg) were added. The mixture was refluxed until TLC analysis indicated complete consumption of the (2-bromoethyl)benzene. The reaction mixture was cooled to room temperature and filtered. The filtrate was evaporated and dissolved in ethyl acetate (30 mL). The resulting solution was washed with 5% aqueous solution of potassium hydroxide (30 mL) and brine (30 mL) and dried over anhydrous sodium sulfate. The solvent was evaporated, and the residue was purified by column chromatography (hexane/ethyl acetate 10:1) to give **1c** as a yellow solid yielded 83 %. (Reference： W. B. Wu and J. M. Huang, *J. Org. Chem.*, 2014, **79**, 10189-10195.)

2. General procedure for the scale up experiment on gram scale

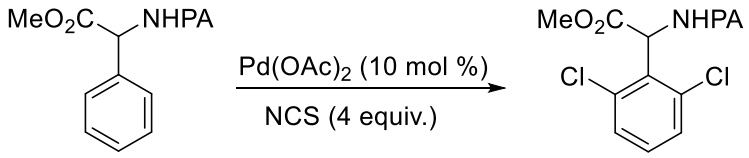
To a 100 mL round bottom flask, **1a** (1.08 g, 4 mmol), 4-Bromo-1-iodobenzene (**2g**; 5.66 g, 20 mmol), AgOAc (1.67 g, 10 mol), $Pd(OAc)_2$ (89.6 mg, 0.4 mmol) and *t*-AmylOH (30 mL) were added, reflux at 130 ^oC in oil bath for 48 h. After cooling to room temperature, The mixture was filtered through a celite pad and concentrated in vacuo. The residue was purified by column chromatography on silica gel (eluent: petroleum ether /EtOAc) to afford the desired arylated product **3g** as a white solid.

3. Removal of directing group

To a 15 mL Schlenk tube, **3g** (58 mg, 0.1 mmol) and MeOH (1 mL) were added under atmospheric air. The reaction solution was stirred at room temperature and $BF_3{\cdot}Et_2O$ (0.126 mL, 1 mmol) was added dropwise to the stirred solution. The tube was sealed with a teflon-coated cap and the mixture was stirred at 130 ℃ for 48 h. After being cooled to ambient temperature, the mixture was quenched by slow addition of saturated Na_2CO_3 solution. The aqueous phase was extracted with ethyl acetate (3 × 10 mL). The combined organic layers were next washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo. The residue was dissolved in DCM (1 mL). Et_3N (0.027 mL, 0.2. mmol) and Boc_2O (43.6 mg, 0.2 mmol) were then added. The solution was stirred 6 h at room temperature. After concentration, the mixture was purified by column chromatography using petroleum ether/EtOAc as the eluent, and the product **5** was obtained as white solid (84 %). (Reference: K. Li, Q. Wu, J. Lan and J. You, *Nat. Commun.*, 2015, **6**, 8404.)

4. Optimization tables for chlorination, alkylation and acyloxylation

Table S1 Optimization of the reaction conditions for di-chlorination of phenylglycine[a]

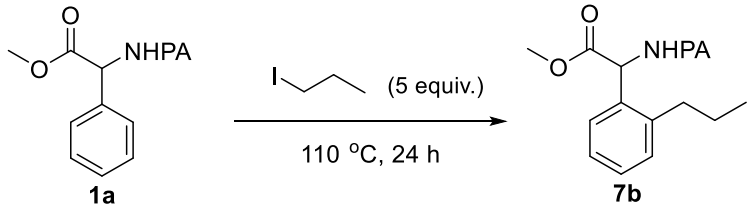


1a **6a**

Entry	Solvent	Oxidant (eq.)	T (^{o}C)	Time (h)	Yield[b] (%)
1	DCE	no	100	24	0
2	CH_3CN	no	100	24	0
3	toluene	no	110	24	trace
4	*p*-xylene	no	110	24	7
5	*t*-AmylOH	no	110	24	0
6	toluene	Ag_2O (3)	110	24	21
7	toluene	AgOAc (3)	110	24	trace
8	toluene	$PhI(OAc)_2$ (3)	110	24	0
9	DCE	$PhI(OAc)_2$ (3)	110	24	0
10	*p*-xylene	Ag_2O (3)	110	36	52
11	*p*-xylene	AgF (3)	110	36	77
12	*p*-xylene	Ag_2CO_3 (3)	110	36	10
13	*p*-xylene	AgOAc (3)	110	36	5

[a]Conditions: **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol %), NCS (4 equiv.), oxidant (3 equiv.), solvent (1mL). [b]Isolated yield.

Table S2 Optimization of the reaction conditions for mono-alkylation of phenylglycine[a]

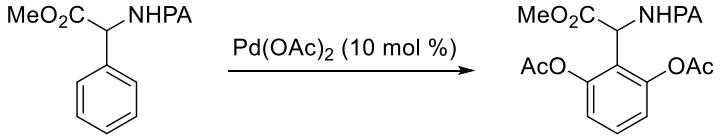


1a **7b**

Entry	Cat. (10 mol %)	Base (2 eq.)	Additive (2 eq.)	Solvent	Yield[b] (%)
1	$Pd(OAc)_2$	$NaHCO_3$	NaTFA	*t*-AmylOH	12
2	$Pd(OAc)_2$	$NaHCO_3$	NaOTf	*t*-AmylOH	10
3	$Pd(OAc)_2$	$NaHCO_3$	PivOH	*t*-AmylOH	0
4	$Pd(OAc)_2$	$NaHCO_3$	no	*t*-AmylOH	27
5	$PdCl_2$	$NaHCO_3$	no	*t*-AmylOH	0
6	$Pd(TFA)_2$	$NaHCO_3$	no	*t*-AmylOH	10
7	$Pd(OAc)_2$	K_2HPO_4	no	*t*-AmylOH	25
8	$Pd(OAc)_2$	Cs_2CO_3	no	*t*-AmylOH	27
9	$Pd(OAc)_2$	KOAc	no	*t*-AmylOH	23
10	$Pd(OAc)_2$	Na_2CO_3	no	*t*-AmylOH	24
11	$Pd(OAc)_2$	NaOAc	no	*t*-AmylOH	20
12	$Pd(OAc)_2$	$NaHCO_3$	no	toluene	15
13	$Pd(OAc)_2$	$NaHCO_3$	no	DCE	30
14[c]	$Pd(OAc)_2$	$NaHCO_3$	no	DCE	45

[a]Conditions: **1a** (0.1 mmol), Pd catalyst (10 mol %), RI (5 equiv.), base (2 equiv.), additives (2 equiv.), solvent (1 mL). [b]Isolated yield. [c]Oxygen atmosphere.

Table S3 Optimization of the reaction conditions for di-acyloxylation of phenylglycine



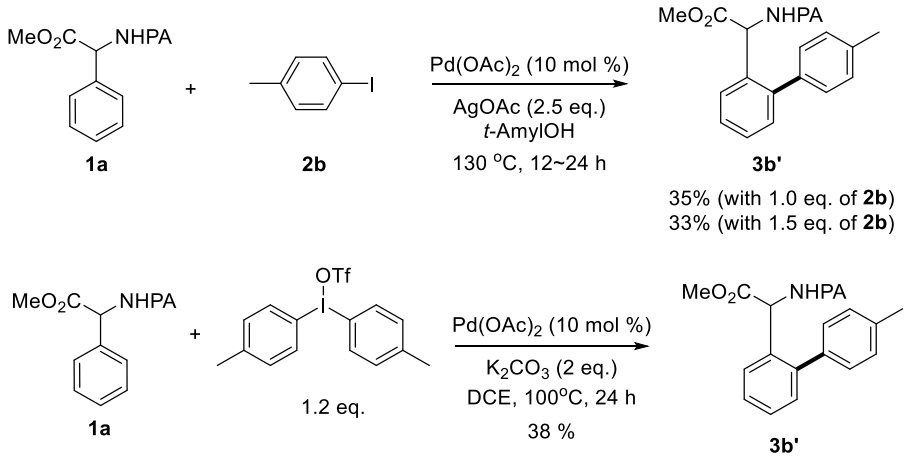
1a **10a**

Entry	Oxcidant (eq.)	Ac_2O (eq.)	Base (eq.)	Solvent	Yield[b] (%)
1	$PhI(OAc)_2$ (5)	no	no	toluene	22
2	$PhI(OAc)_2$ (5)	no	no	p-xylene	20
3	$PhI(OAc)_2$ (5)	no	no	Ac_2O	0
4	$PhI(OAc)_2$ (5)	no	no	Ac_2O:AcOH (1:1)	0
5	$PhI(OAc)_2$ (5)	no	no	DCE	3
6	$PhI(OAc)_2$ (5)	no	Ag_2CO_3 (3)	toluene	40
7	$PhI(OAc)_2$ (5)	no	Li_2CO_3 (3)	toluene	0
8	$PhI(OAc)_2$ (5)	no	Na_2CO_3 (3)	toluene	0
9	$PhI(OAc)_2$ (5)	no	Ag_2CO_3 (1)	toluene	40
10	$PhI(OAc)_2$ (4)	no	Ag_2CO_3 (1)	toluene	42
11	$PhI(OAc)_2$ (5)	10	Ag_2CO_3 (1)	toluene	65
12	$PhI(OAc)_2$ (4)	10	Ag_2CO_3 (1)	toluene	69
13	$PhI(OAc)_2$ (3)	10	Ag_2CO_3 (1)	toluene	59
14	$K_2S_2O_8$ (3)	10	Ag_2CO_3 (1)	toluene	0

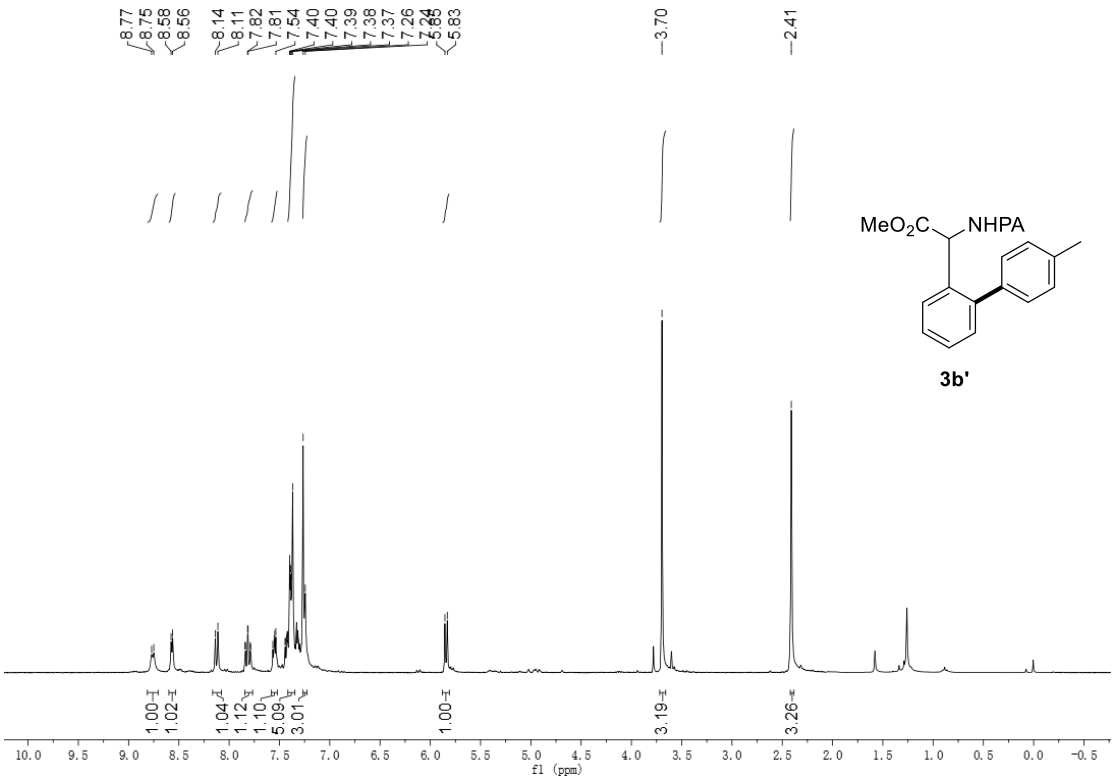
[a]Conditions: **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol %), base, solvent (1mL). [b]Isolated yield.

5. The attempts and result of mono-arylation of α-phenylglycine

One of the referees commented about the mono-arylated product. It would be of great value if mono-arylation could be developed to access mono-arylated α-phenylglycine derivatives. A lot of efforts were made to optimize the yield of the mono-arylated product *via* reducing the equivalents of aryl iodide to 1~1.5 equiv, however both the conversion of the reactions and the yields of mono-arylated product are low. Other efforts to achieve mono-arylation reaction included: solvent screening (DCE, toluene, dioxane, etc), additive screening ($AgCO_3$, AgF, AgO, K_2CO_3, Li_2CO_3, $NaHCO_3$, etc), and different arylation reagents ($PhB(OH)_2$, diaryliodonium triflate, and diaryliodonium tetrafluoroborate). However, the yield of the mono-arylated product is always below 40%. The following are our inferior results of mono-arylation of α-phenylglycine.



Compound 3b': ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.76 (d, *J* = 6.5 Hz, 1H), 8.57 (d, *J* = 4.7 Hz, 1H), 8.12 (d, *J* = 7.8 Hz, 1H), 7.81 (td, *J* = 7.7, 1.6 Hz, 1H), 7.55 (dd, *J* = 5.4, 3.6 Hz, 1H), 7.45 – 7.35 (m, 5H), 7.25 (d, *J* = 6.5 Hz, 3H), 5.84 (d, *J* = 7.0 Hz, 1H), 3.70 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.55, 163.64, 149.40, 148.26, 142.75, 137.33, 137.25, 137.20, 134.32, 130.96, 129.41, 129.07, 128.43, 128.02, 127.01, 126.38, 122.34, 53.73, 52.73, 21.29. HRMS (ESI) calcd. for $C_{22}H_{20}N_2O_3$: 360.1474. Found 360.1476.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8.14
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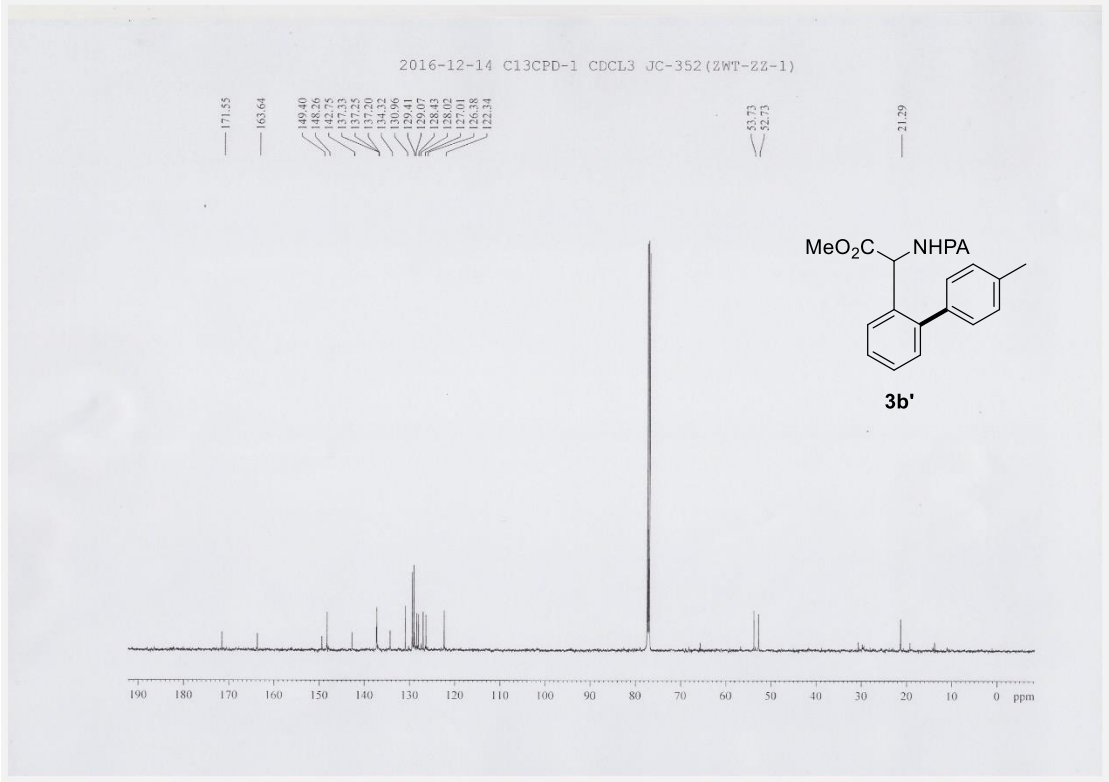
7.82
7.81
7.54
7.40
7.40
7.39
7.38
7.37
7.26
5.85
5.83
3.70
2.41
MeO2C
NHPA
3b'
1.00
1.02
1.04
1.12
1.10
5.09
3.01
1.00
3.19
3.26
f1 (ppm)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142.75
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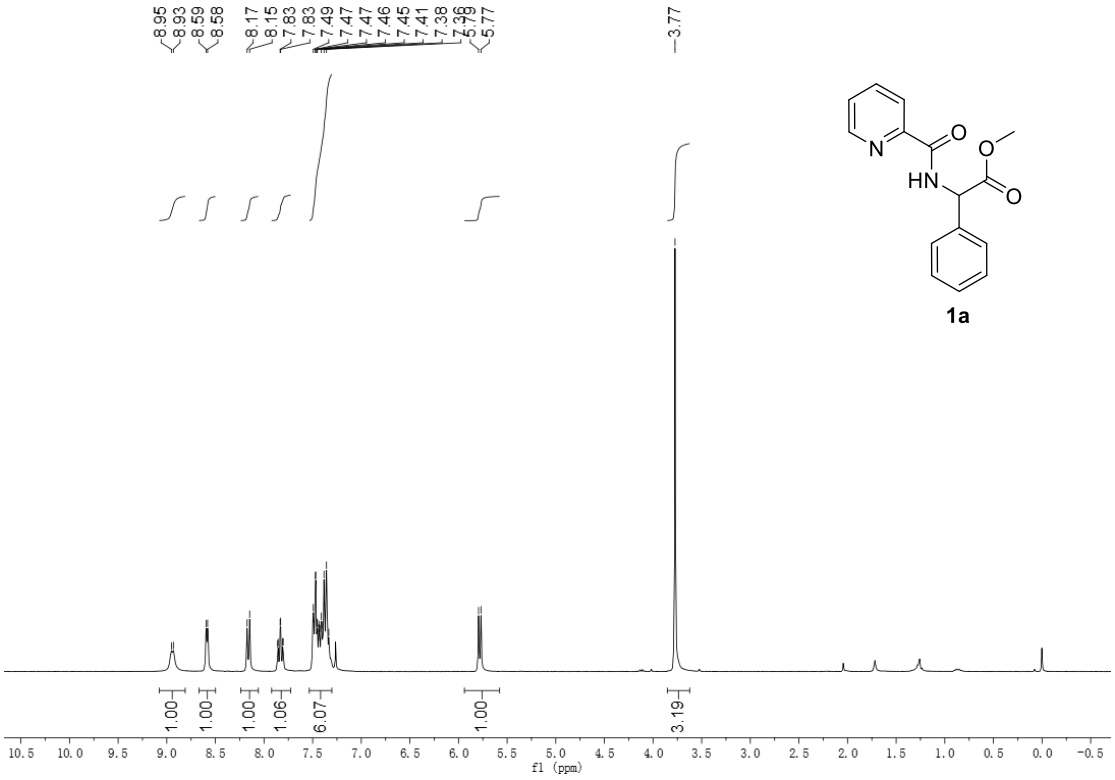
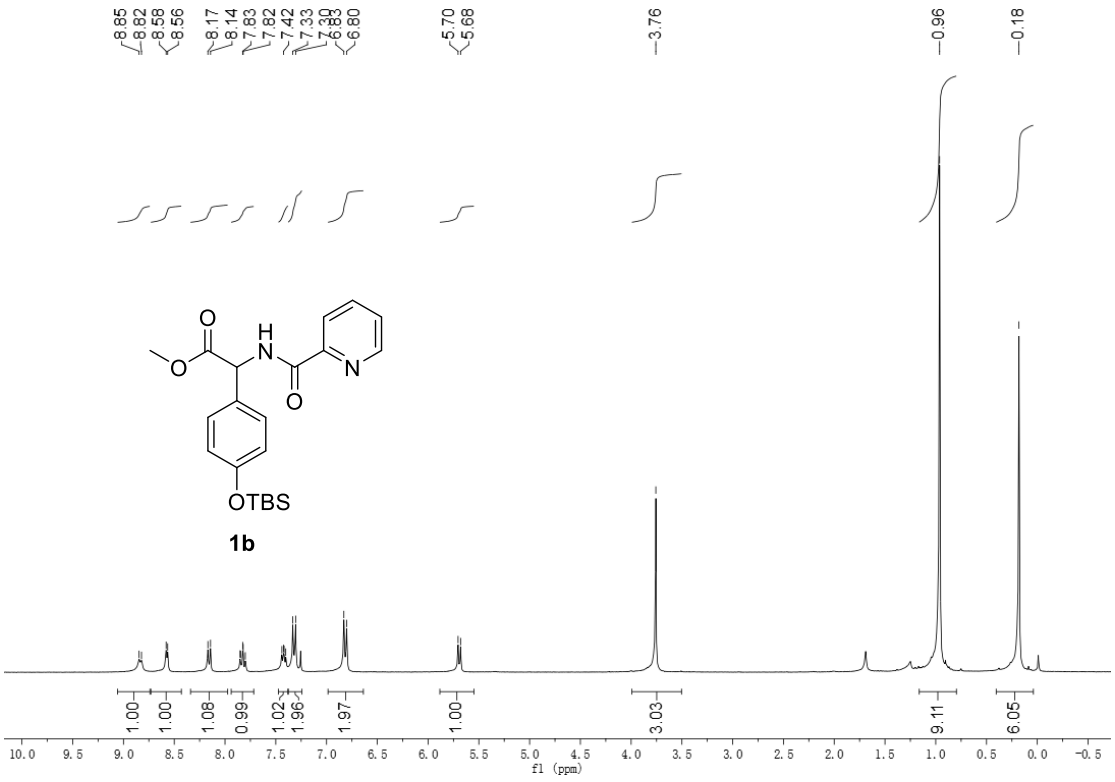
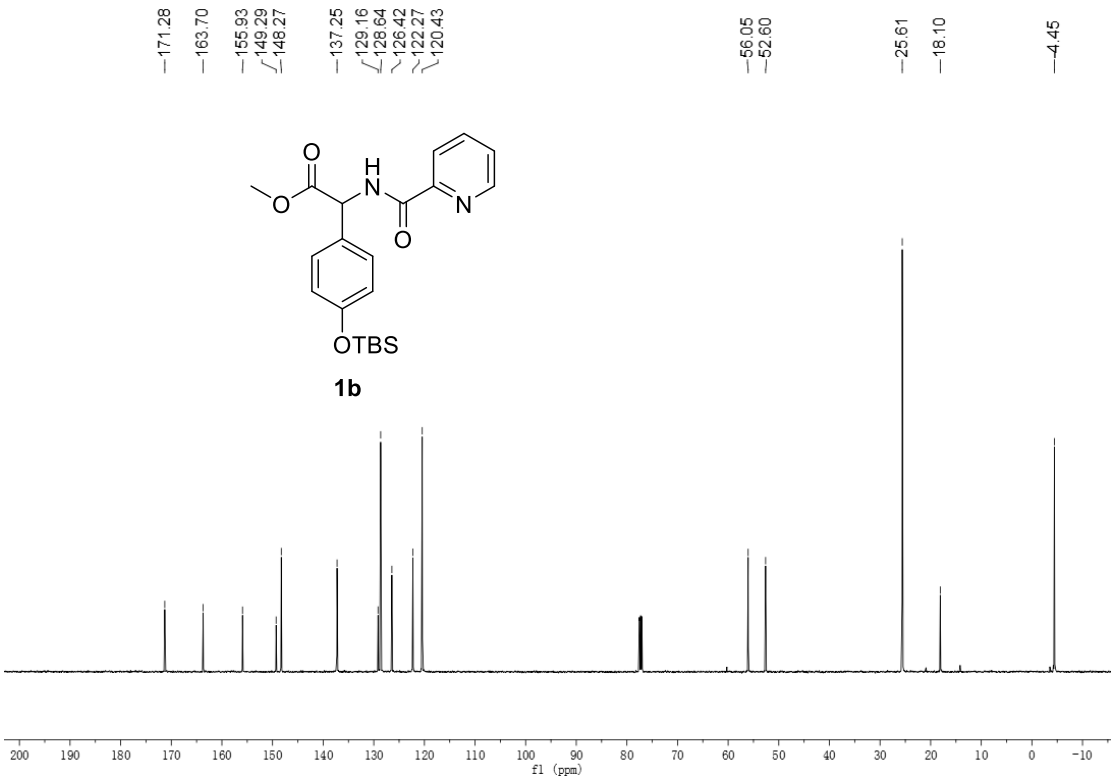
137.20
134.32
130.96
129.41
129.07
128.43
128.02
127.01
126.38
122.34
53.73
52.73
21.29
MeO2C
NHPA
3b'
ppm

6. ^{1}H and ^{13}C NMR spectra of compounds



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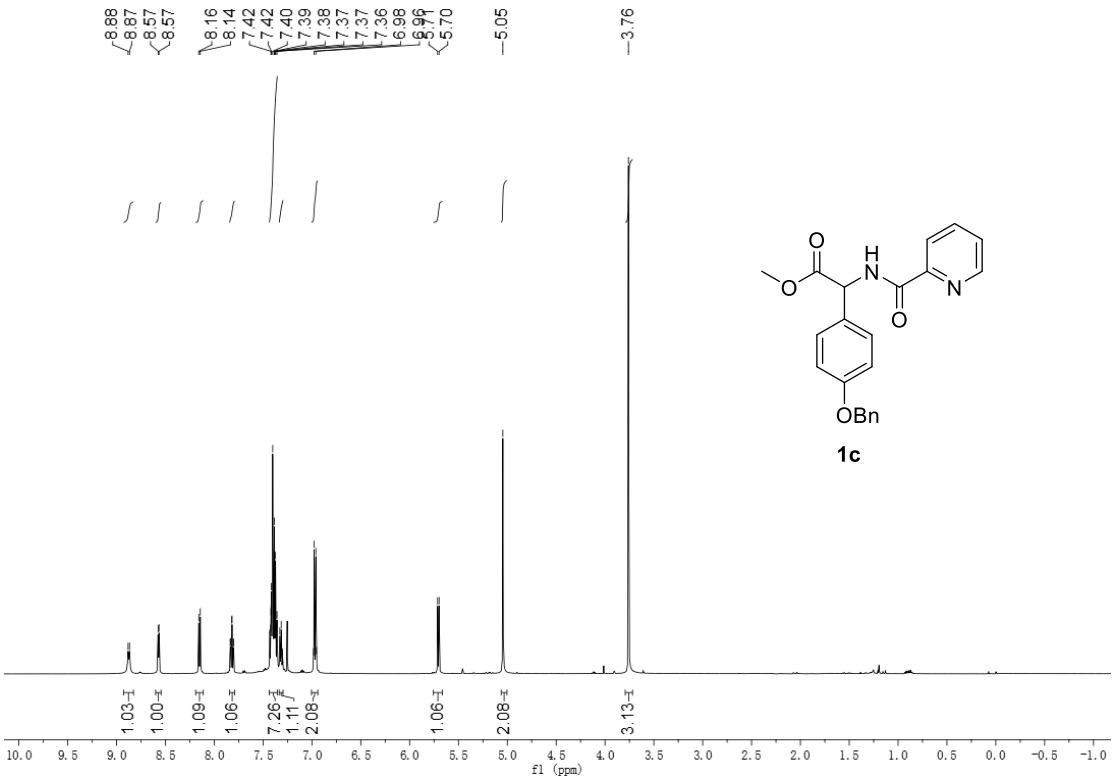
25.61
18.10
-4.45
OTBS
1b
f1 (ppm)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7.37
7.36
6.98
5.71
5.70
5.05
3.76
OBn
1c
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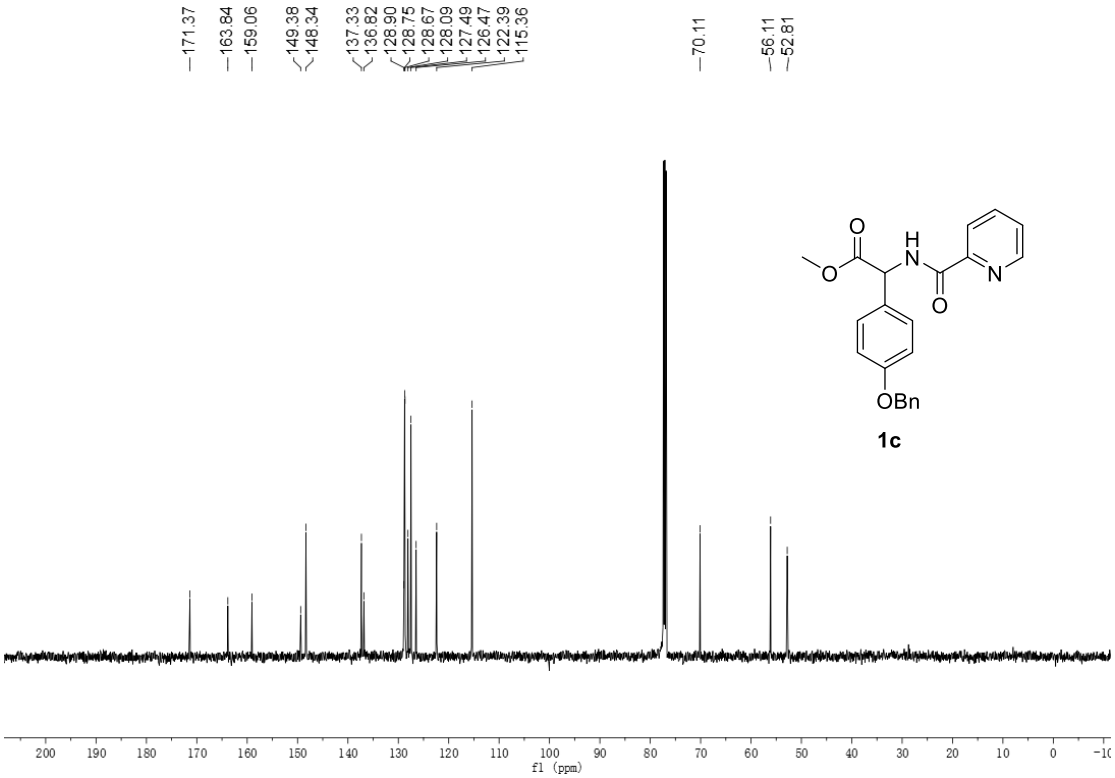
1.09
1.06
7.26
1.11
2.08
1.06
2.08
3.13
f1 (ppm)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70.11
56.11
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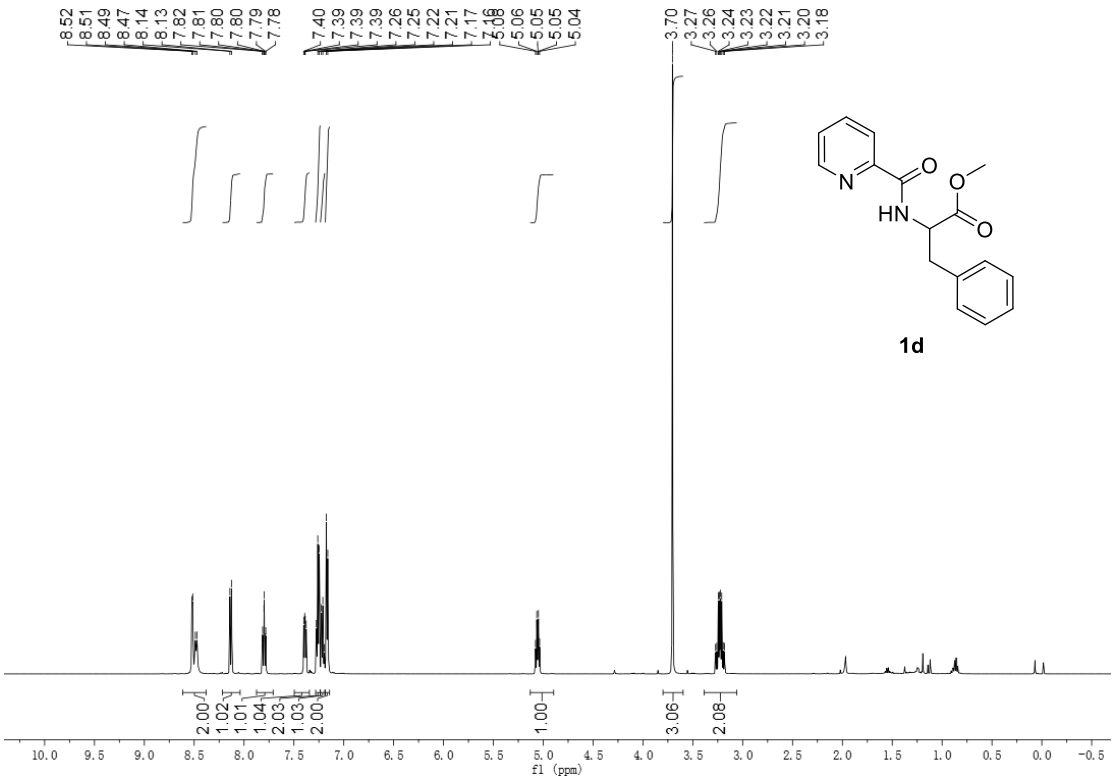
OBn
1c
f1 (ppm)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7.81
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7.39
7.39
7.39
7.26
7.25
7.22
7.21
7.17
7.16
5.08
5.06
5.05
5.05
5.04
3.70
3.27
3.26
3.24
3.23
3.22
3.21
3.20
3.18
1d
2.00
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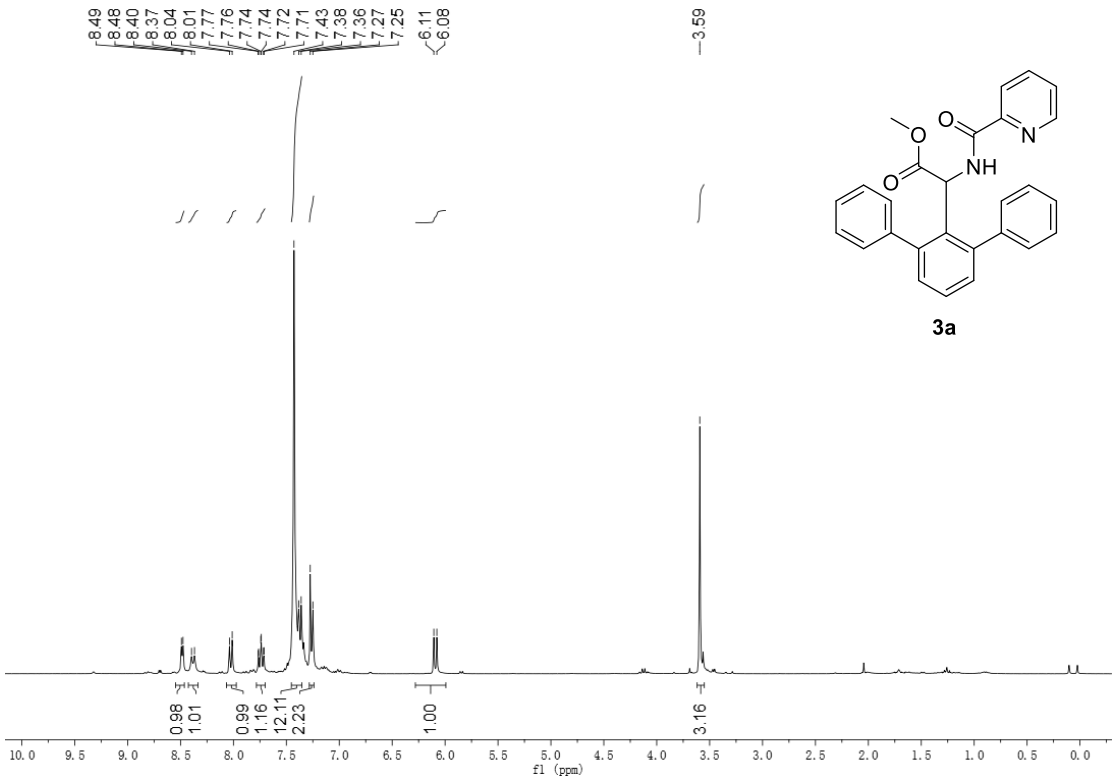
1.01
1.04
2.03
1.03
2.00
1.00
3.06
2.08
f1 (ppm)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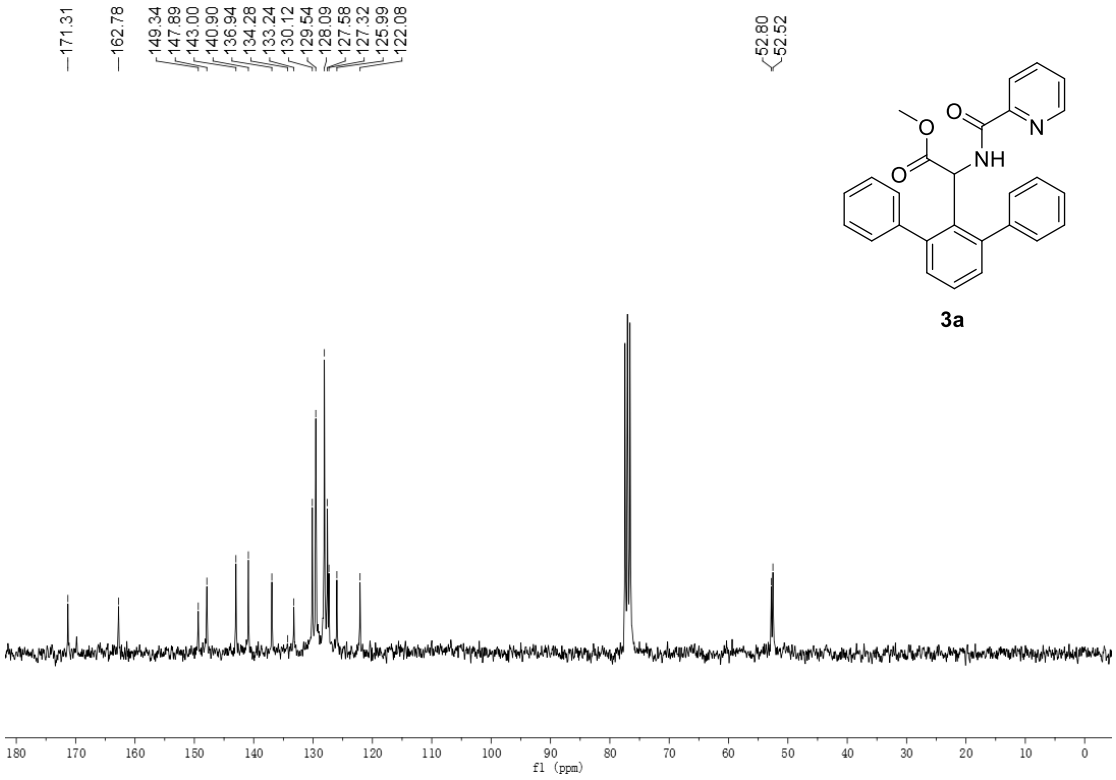
7.38
7.36
7.27
7.25
6.11
6.08
3.59
O
N
NH
O
O
0.98
1.01
0.99
1.16
12.11
2.23
1.00
3.16
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3a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127.32
125.99
122.08
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O
N
NH
O
O
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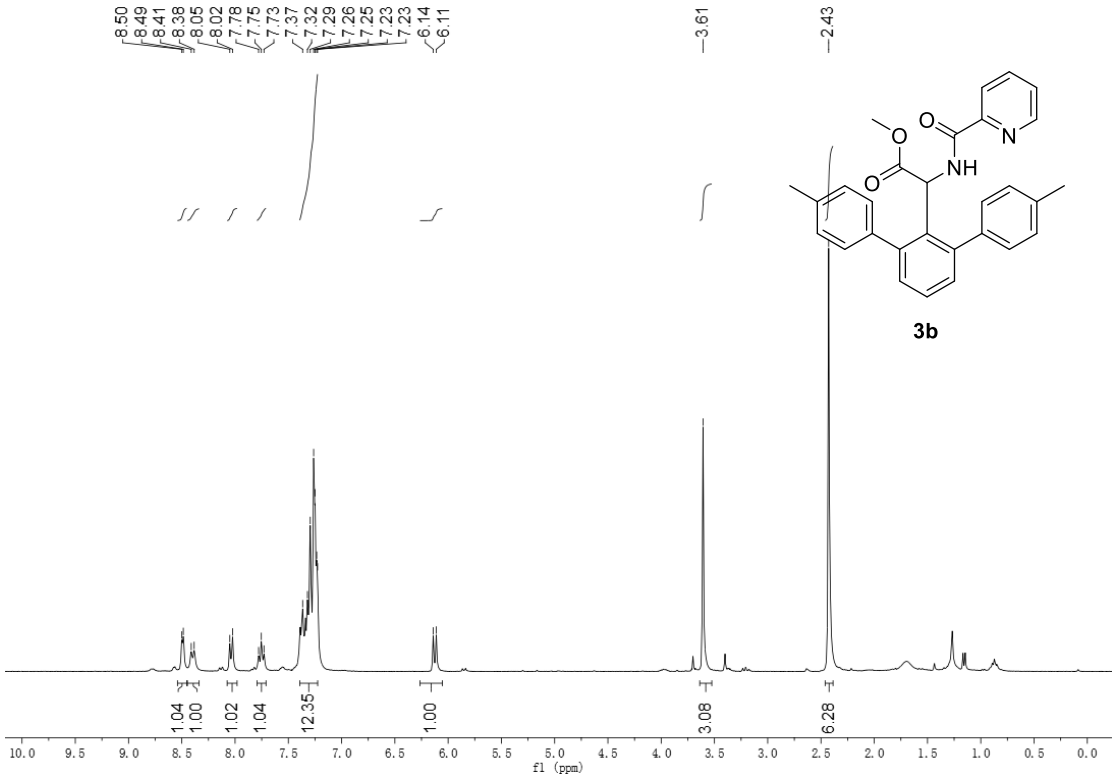
f1 (ppm)

3a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7.26
7.25
7.23
7.23
6.14
6.11
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1.02
1.04
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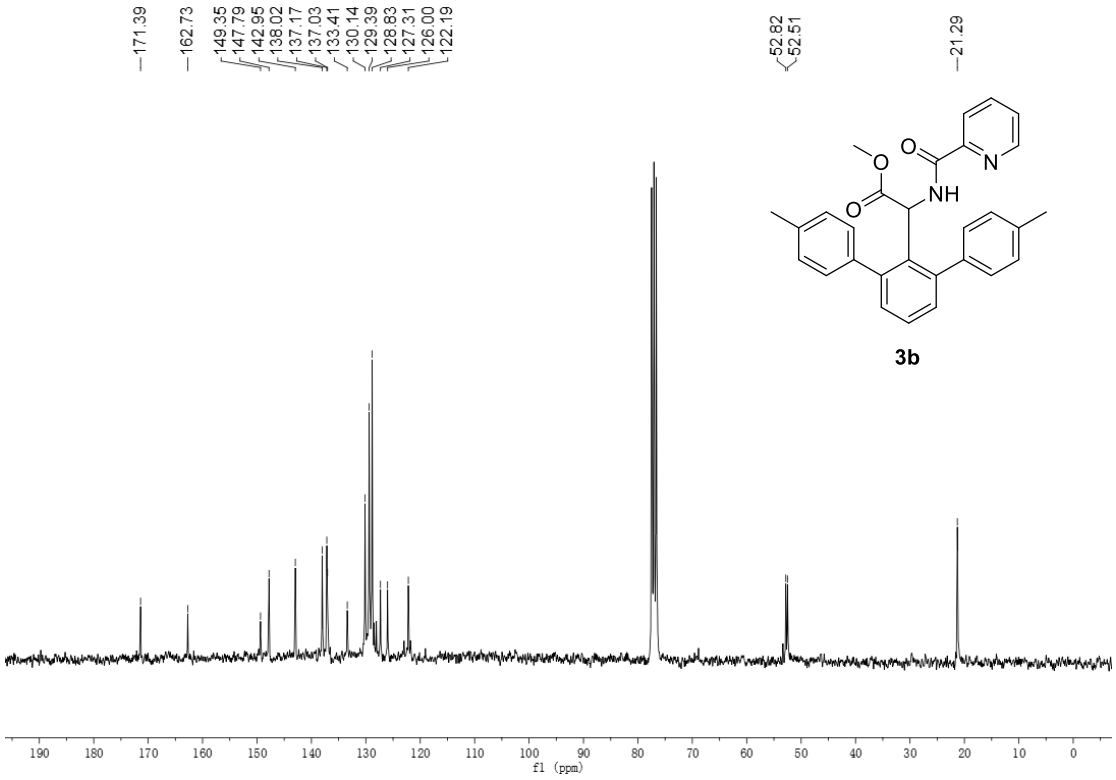
1.00
3.08
6.28
f1 (ppm)

3b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137.03
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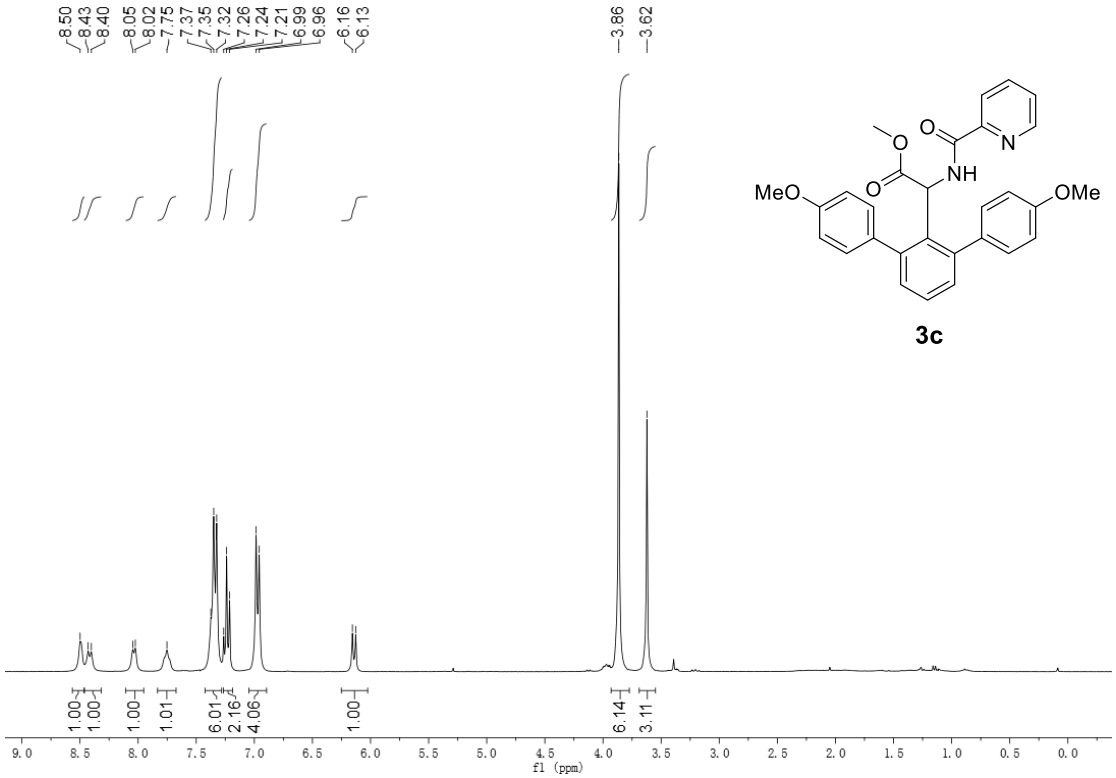
52.51
21.29
f1 (ppm)

3b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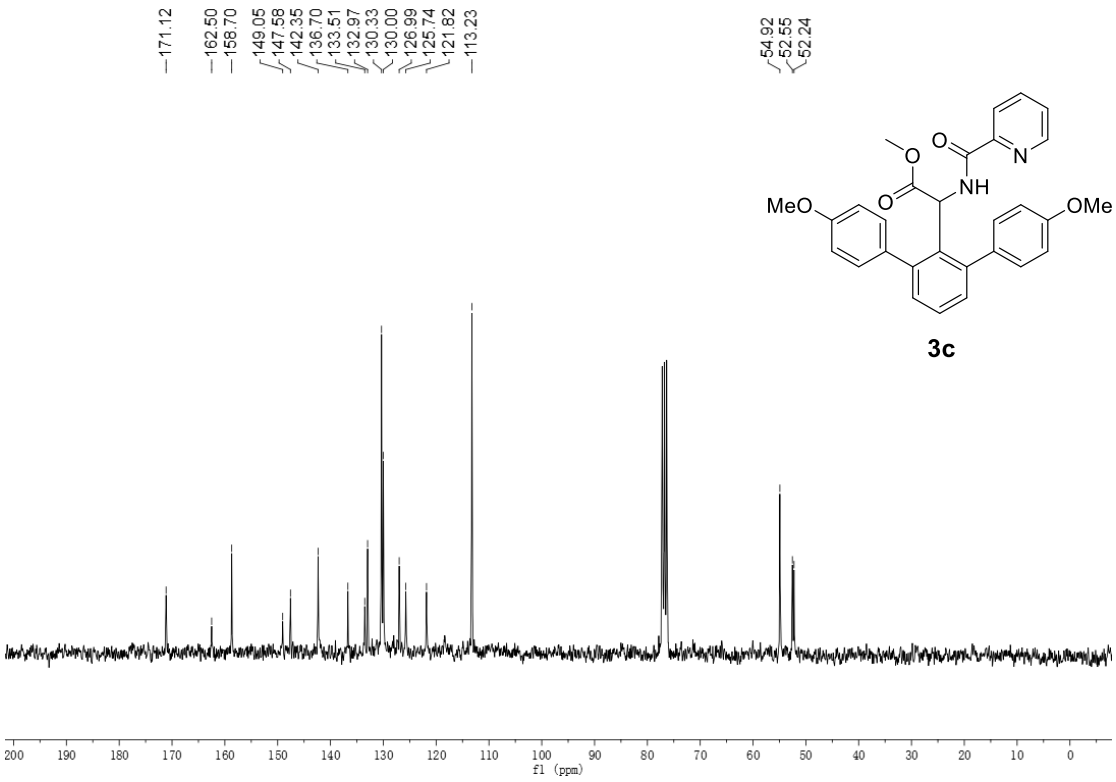
OMe
O
N
NH

3c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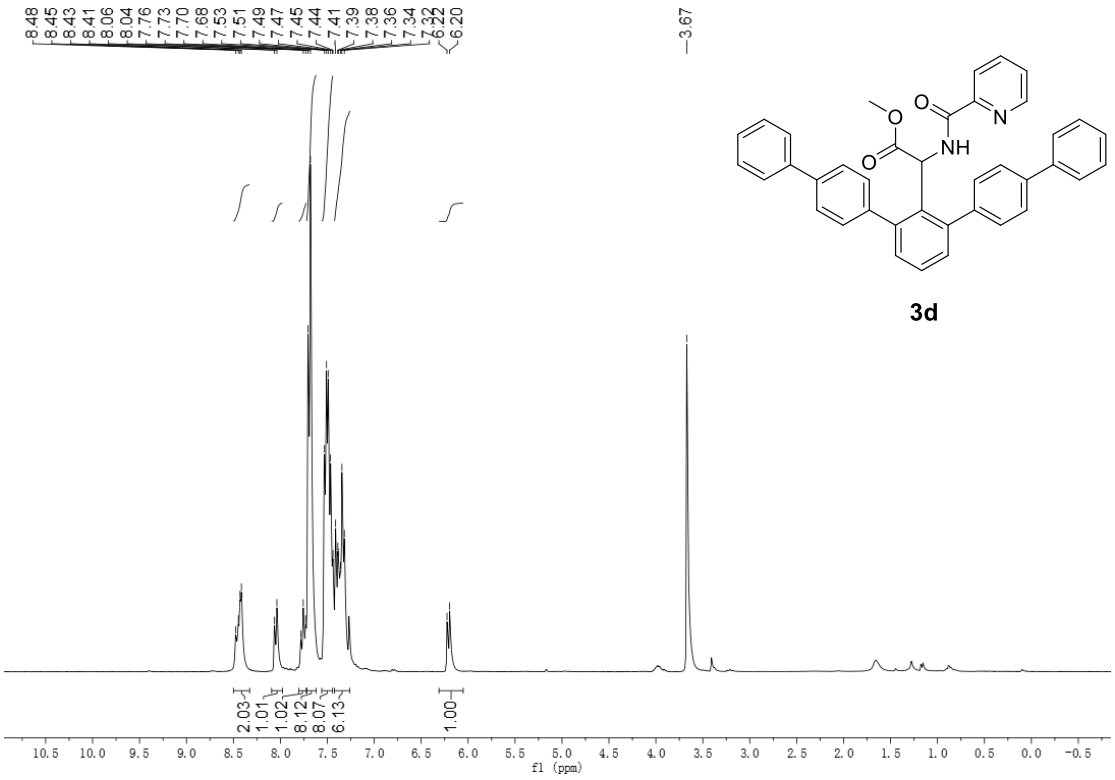
f1 (ppm)
MeO
OMe
O
N
NH

3c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7.76
7.73
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7.68
7.53
7.51
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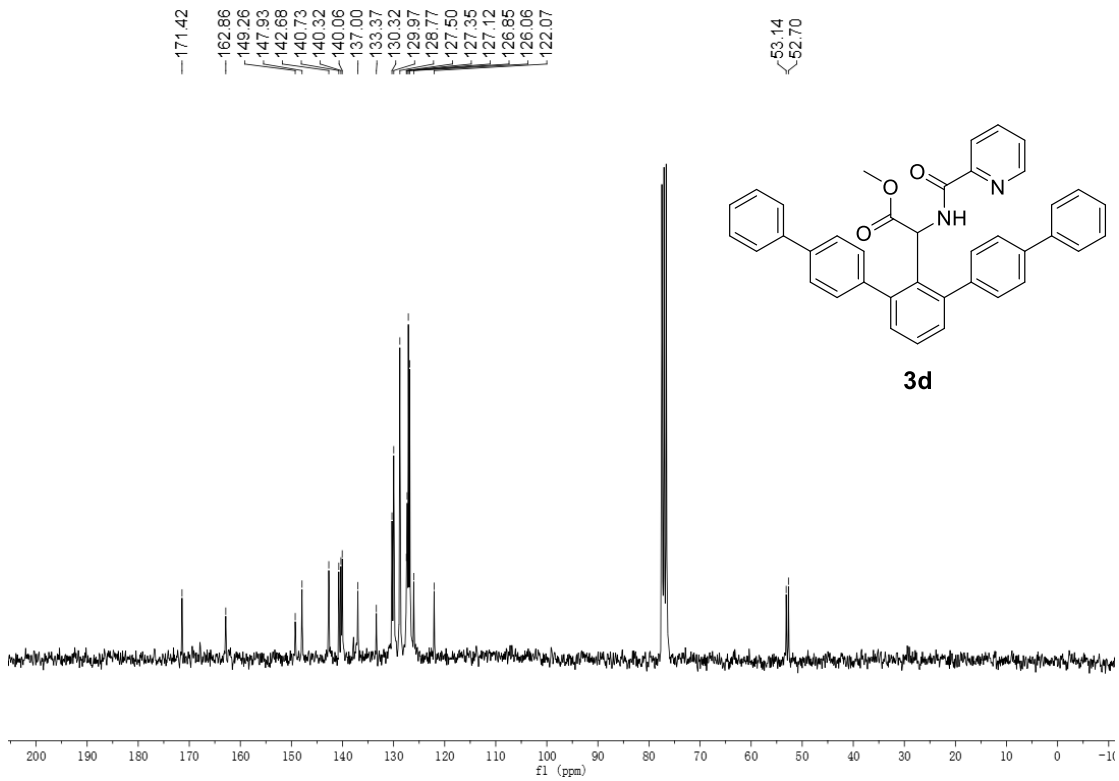
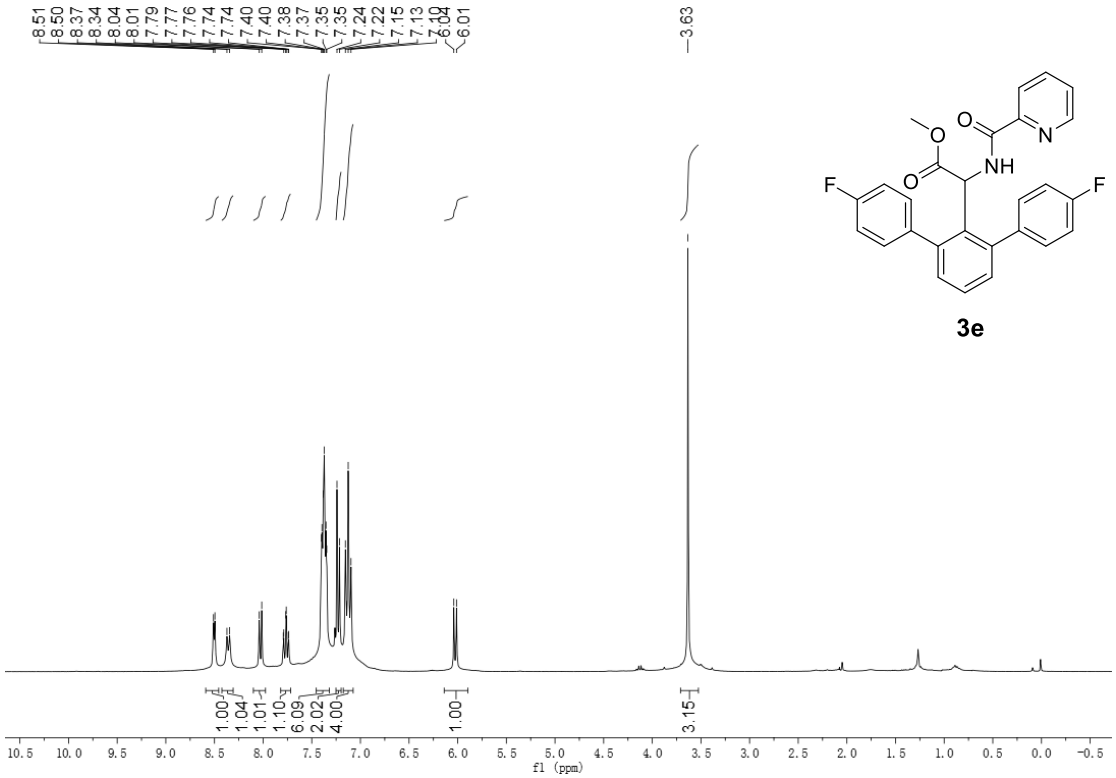
7.47
7.45
7.44
7.41
7.39
7.38
7.36
7.34
7.32
6.22
6.20
3.67
2.03
1.01
1.02
8.12
8.07
6.13
1.00
f1 (ppm)

3d

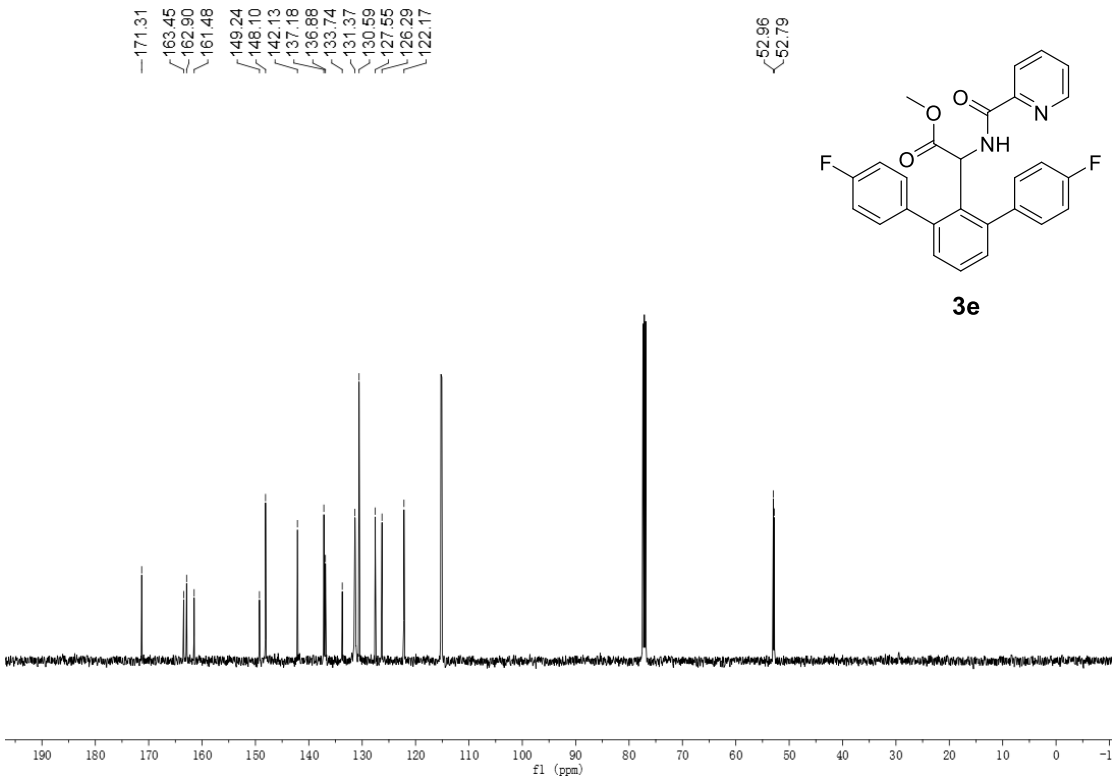

171.42
162.86
149.26
147.93
142.68
140.73
140.32
140.06
137.00
133.37
130.32
129.97
128.77
127.50
127.35
127.12
126.85
126.06
122.07
53.14
52.70
f1 (ppm)

3d


8.51
8.50
8.37
8.34
8.04
8.01
7.79
7.77
7.76
7.74
7.74
7.40
7.40
7.38
7.37
7.35
7.35
7.24
7.22
7.15
7.13
6.04
6.01
3.63
1.00
1.04
1.01
1.10
6.09
2.02
4.00
1.00
3.15
f1 (ppm)

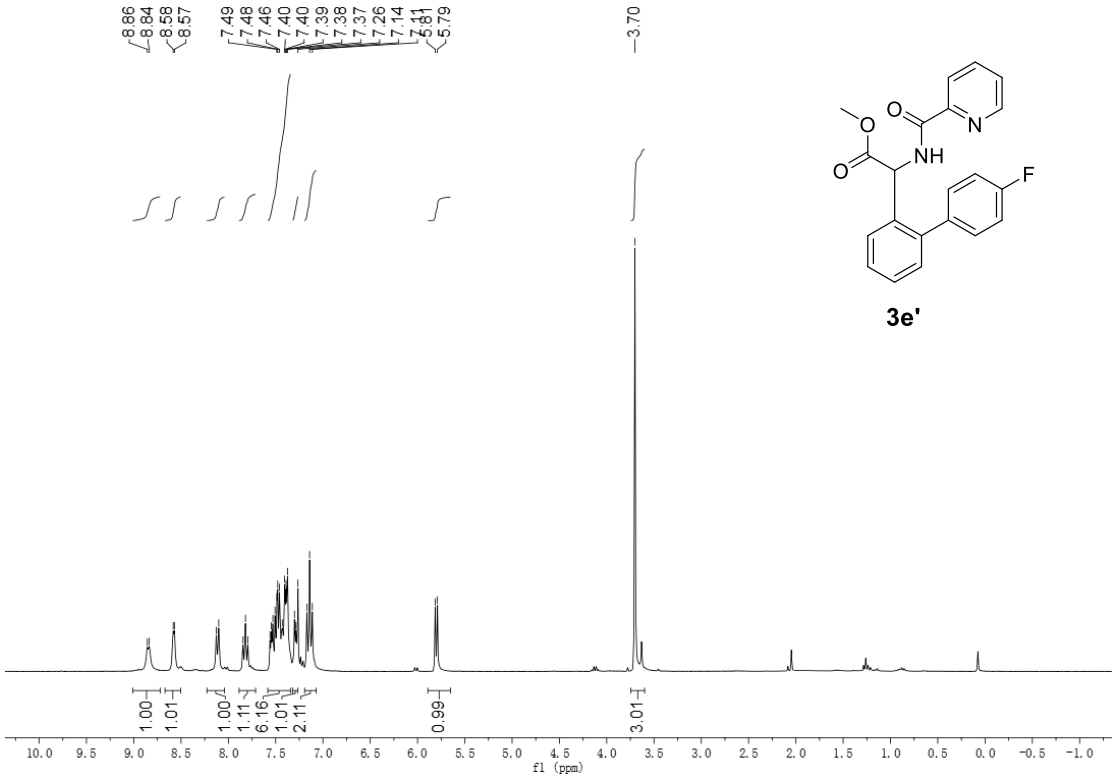
O
N
O
NH
O
F
F

3e

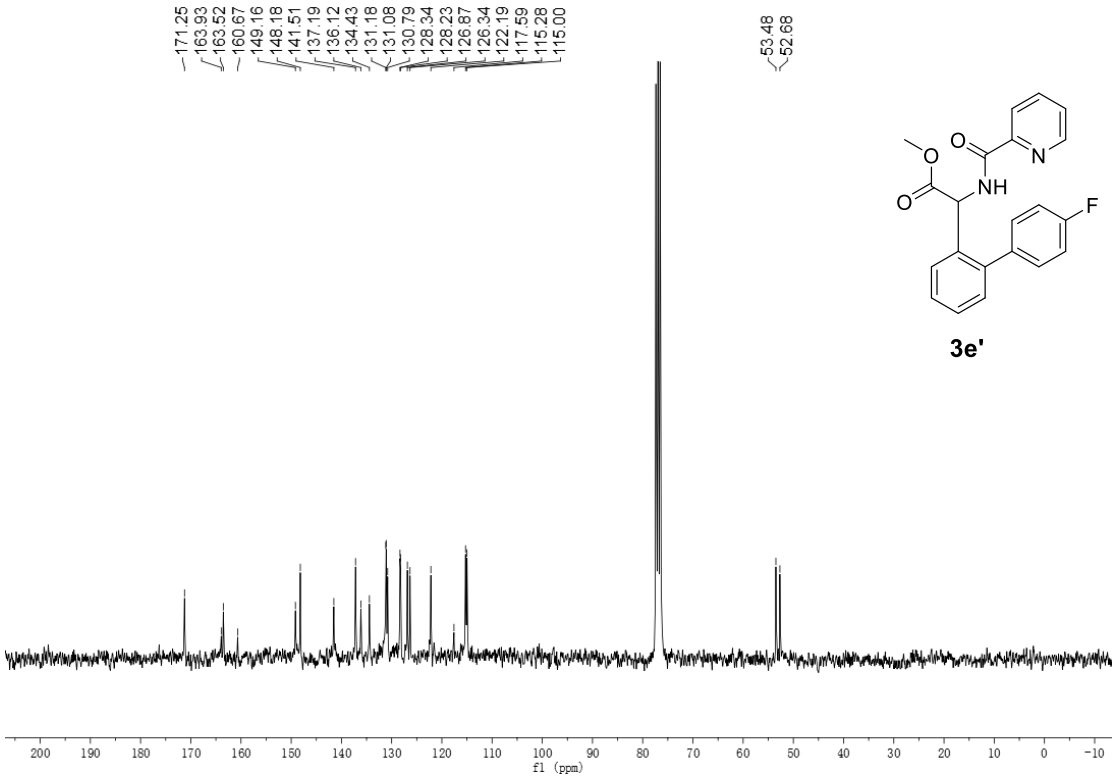

171.31
163.45
162.90
161.48
149.24
148.10
142.13
137.18
136.88
133.74
131.37
130.59
127.55
126.29
122.17
52.96
52.79
f1 (ppm)

O
N
O
NH
O
F
F

3e


8.86
8.84
8.58
8.57
7.49
7.48
7.46
7.40
7.40
7.39
7.38
7.37
7.26
7.14
5.81
5.79
3.70
1.00
1.01
1.00
1.11
6.16
1.01
2.11
0.99
3.01
f1 (ppm)

3e'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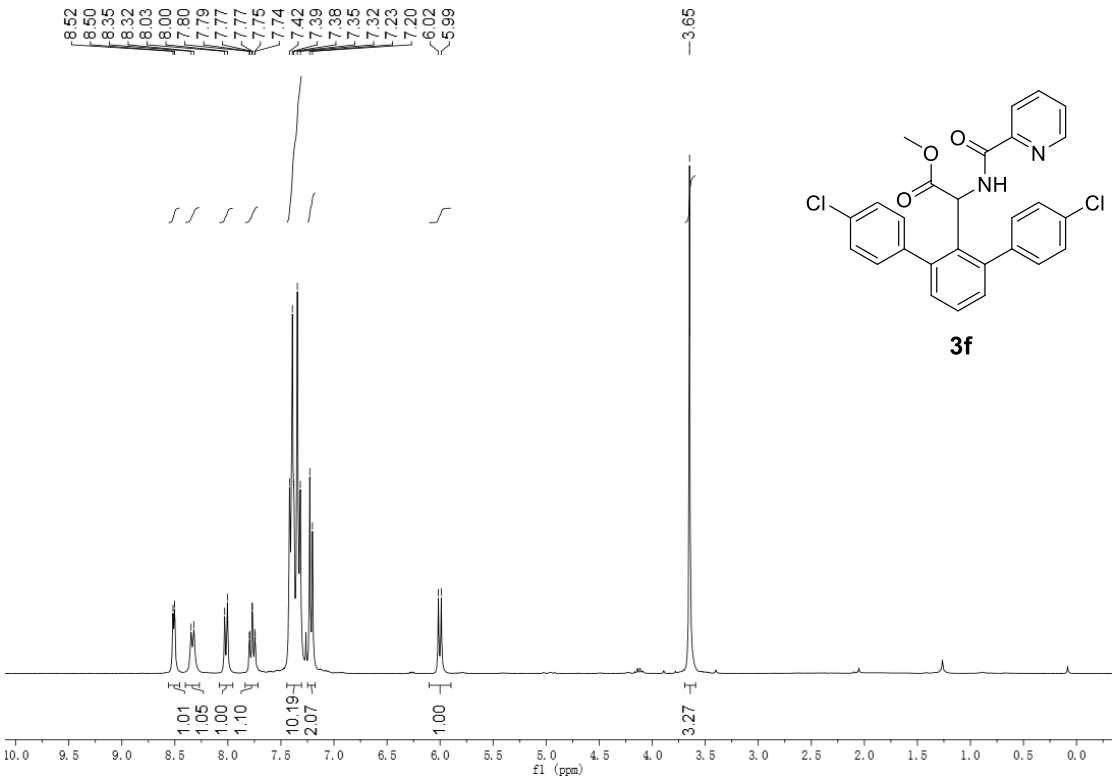
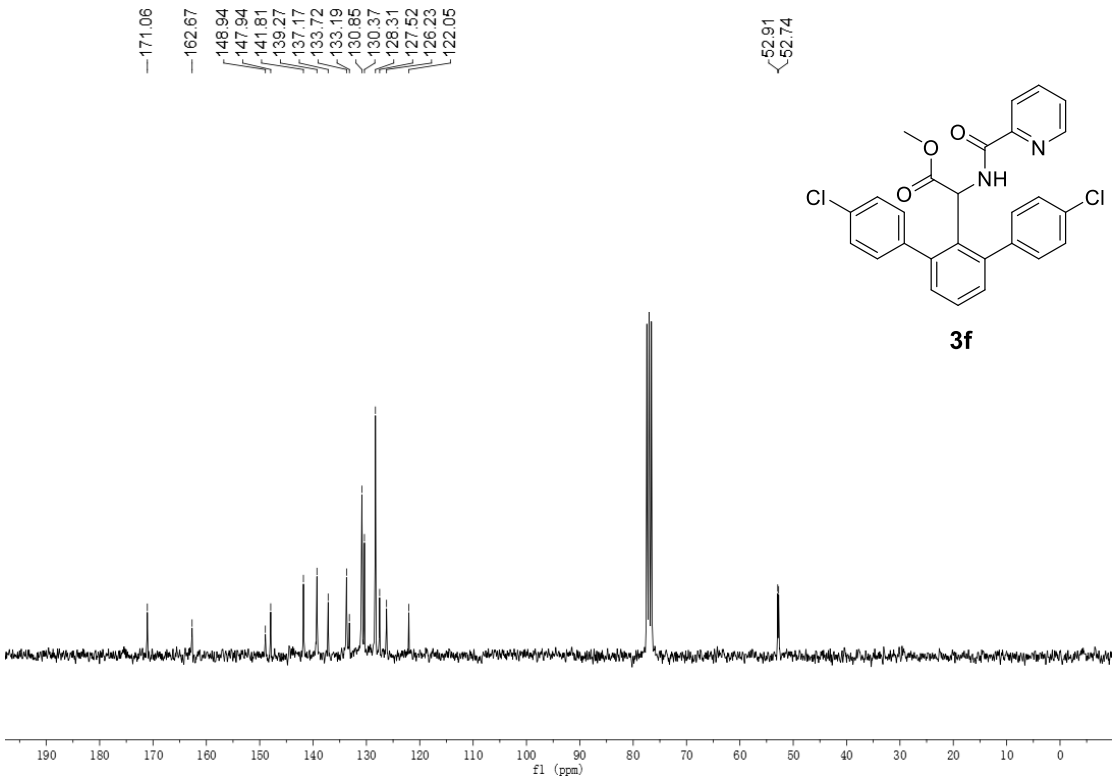
149.16
148.18
141.51
137.19
136.12
134.43
131.18
131.08
130.79
128.34
128.23
126.87
126.34
122.19
117.59
115.28
115.00
53.48
52.68
f1 (ppm)

3e'


8.52
8.50
8.35
8.32
8.03
8.00
7.80
7.79
7.77
7.77
7.75
7.74
7.42
7.39
7.38
7.35
7.32
7.23
7.20
6.02
5.99
3.65
1.01
1.05
1.00
1.10
10.19
2.07
1.00
3.27
f1 (ppm)

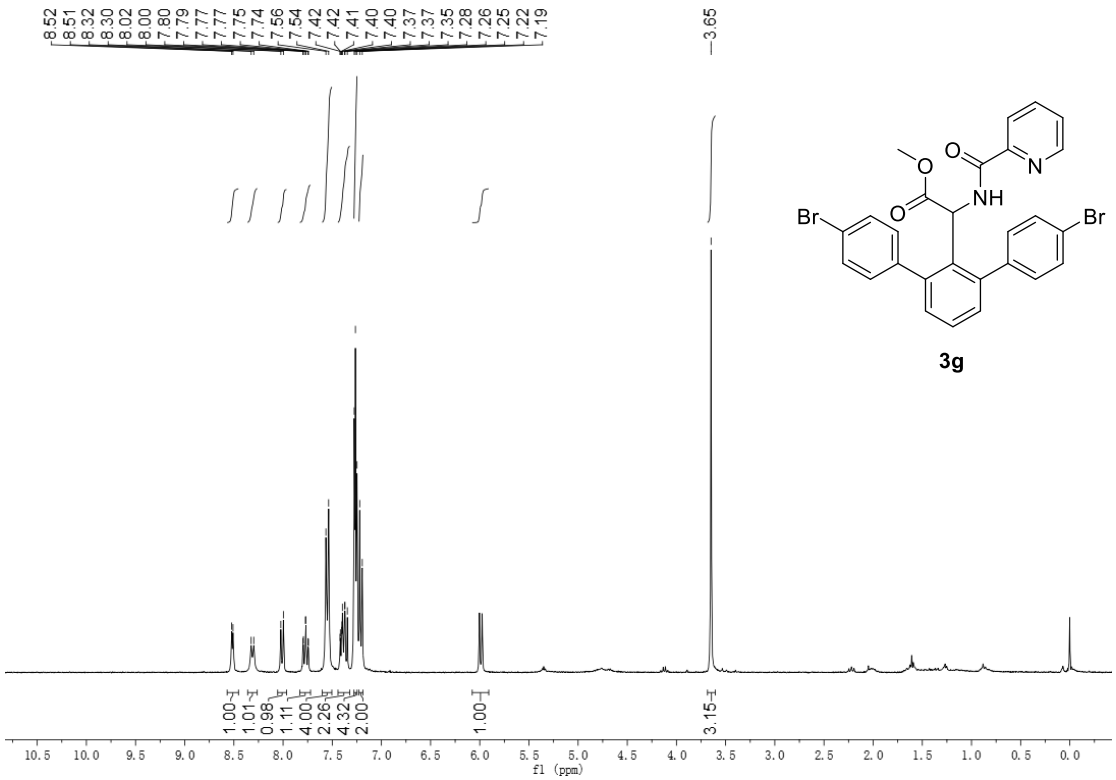
O
N
O
NH
O
Cl
Cl

3f


171.06
162.67
148.94
147.94
141.81
139.27
137.17
133.72
133.19
130.85
130.37
128.31
127.52
126.23
122.05
52.91
52.74
f1 (ppm)

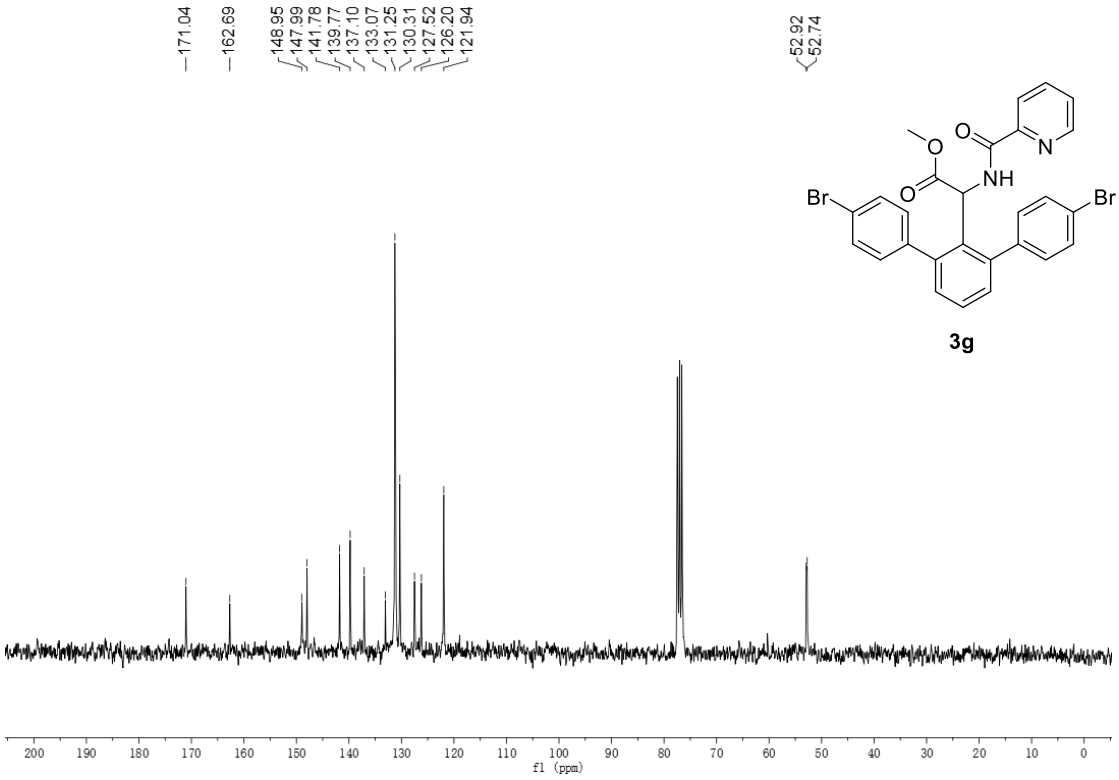
O
N
O
NH
O
Cl
Cl

3f


8.52
8.51
8.32
8.30
8.02
8.00
7.80
7.79
7.77
7.77
7.75
7.74
7.56
7.54
7.42
7.42
7.41
7.40
7.40
7.37
7.37
7.35
7.28
7.26
7.25
7.22
7.19
3.65
1.00
1.01
0.98
1.11
4.00
2.26
4.32
2.00
1.00
3.15
f1 (ppm)

O
N
O
NH
O
Br
Br

3g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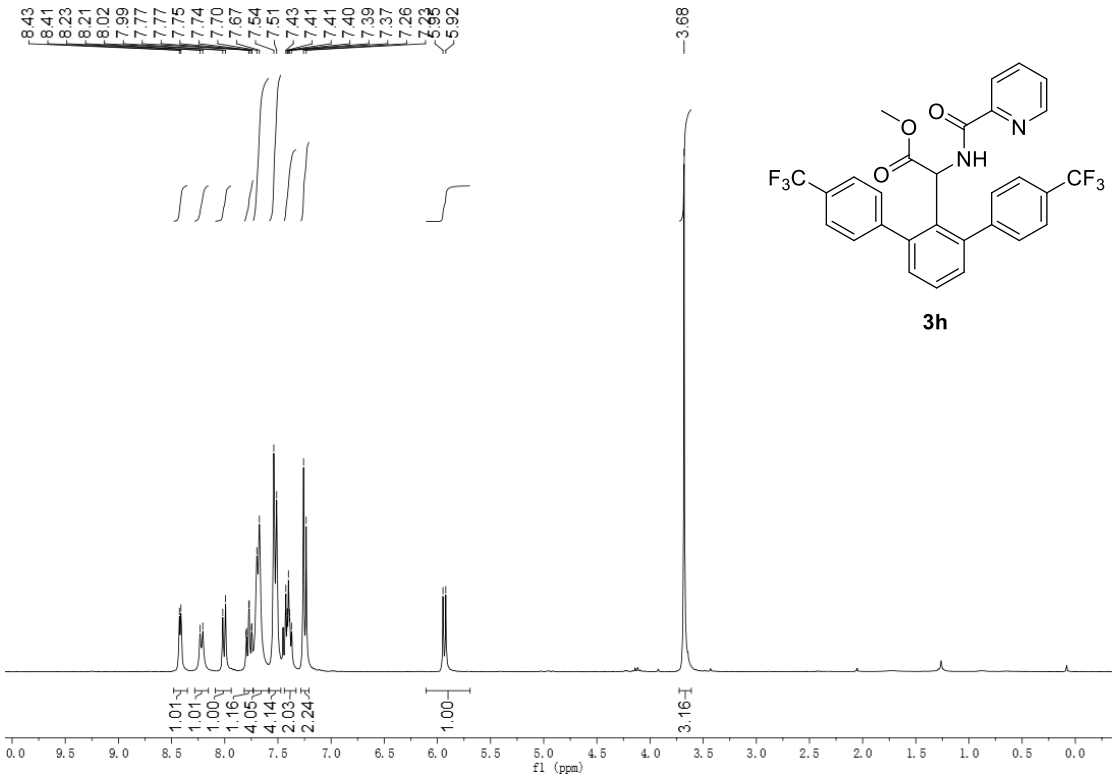
162.69
148.95
147.99
141.78
139.77
137.10
133.07
131.25
130.31
127.52
126.20
121.94
52.92
52.74
f1 (ppm)

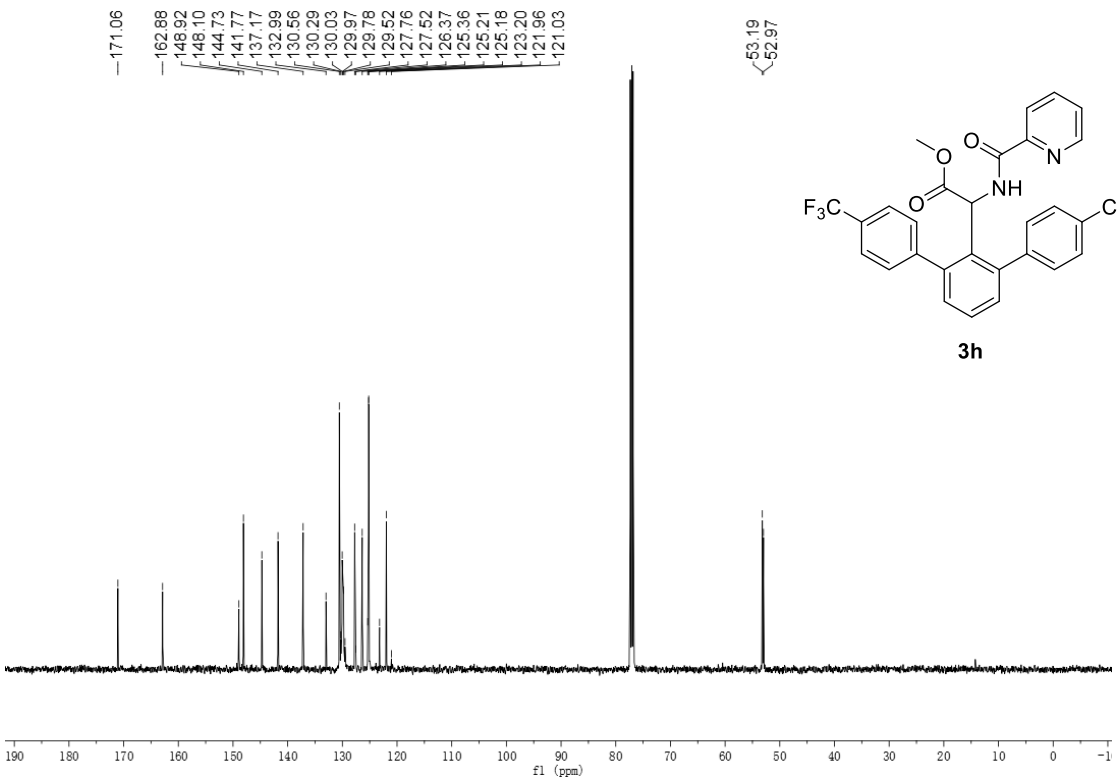
O
N
O
NH
O
Br
Br

3g


8.43
8.41
8.23
8.21
8.02
7.99
7.77
7.75
7.74
7.70
7.67
7.54
7.51
7.43
7.41
7.40
7.39
7.37
7.26
5.95
5.92
3.68
1.01
1.01
1.00
1.16
4.05
4.14
2.03
2.24
1.00
3.16
f1 (ppm)

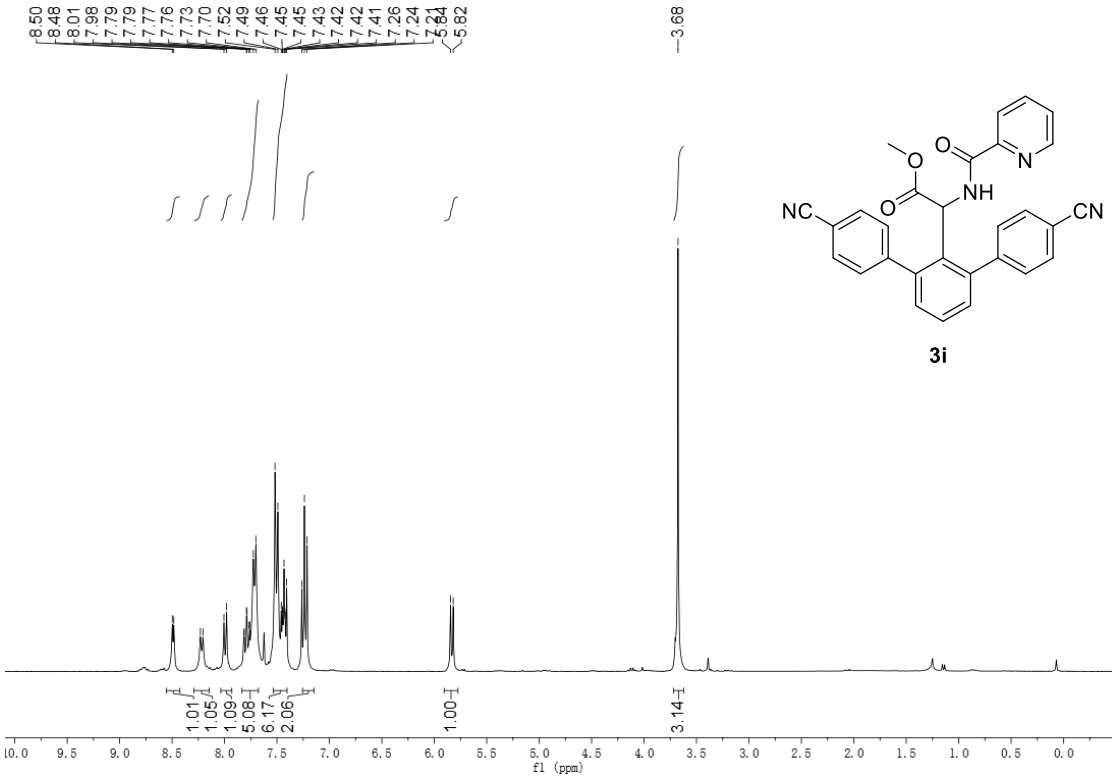
O
N
O
NH
O
F3C
CF3

3h


171.06
162.88
148.92
148.10
144.73
141.77
137.17
132.99
130.56
130.29
130.03
129.97
129.78
129.52
127.76
127.52
126.37
125.36
125.21
125.18
123.20
121.96
121.03
53.19
52.97
f1 (ppm)

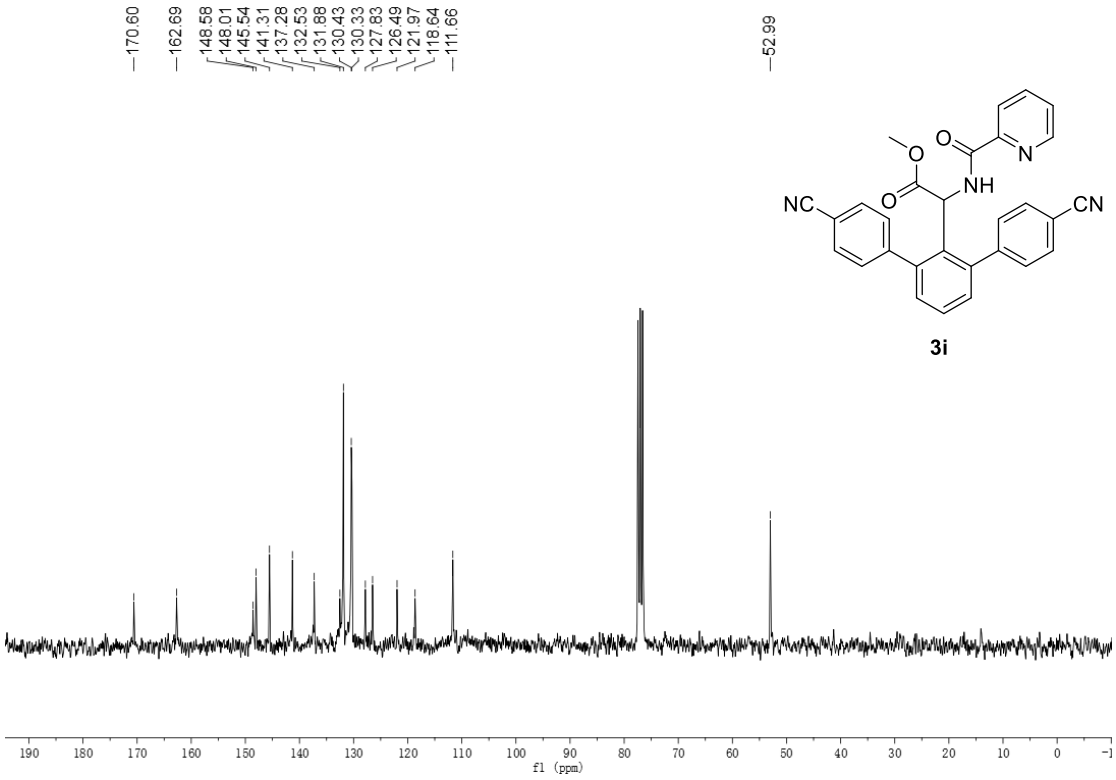
O
N
O
NH
O
F3C
CF3

3h


8.50
8.48
8.01
7.98
7.79
7.79
7.77
7.76
7.73
7.70
7.52
7.49
7.46
7.45
7.45
7.43
7.42
7.42
7.41
7.26
7.24
7.21
5.84
5.82
3.68
1.01
1.05
1.09
5.08
6.17
2.06
1.00
3.14
f1 (ppm)

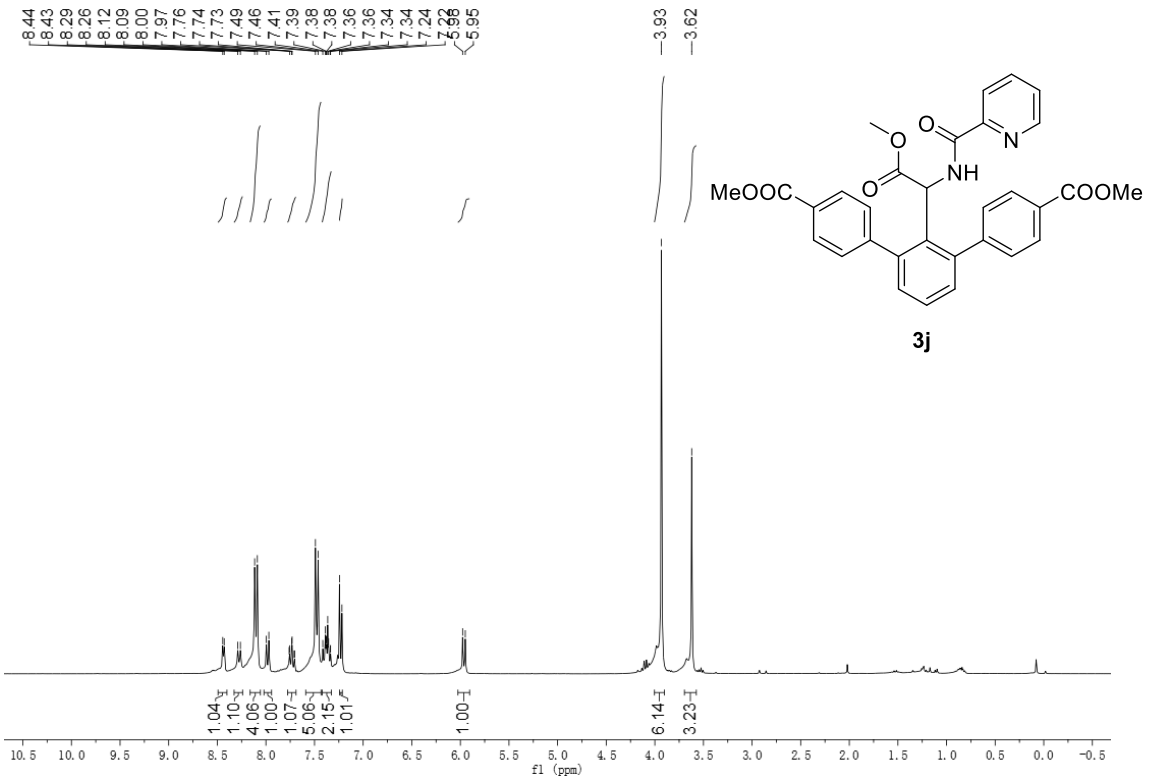
O
N
O
NH
O
NC
CN

3i


170.60
162.69
148.58
148.01
145.54
141.31
137.28
132.53
131.88
130.43
130.33
127.83
126.49
121.97
118.64
111.66
52.99
f1 (ppm)

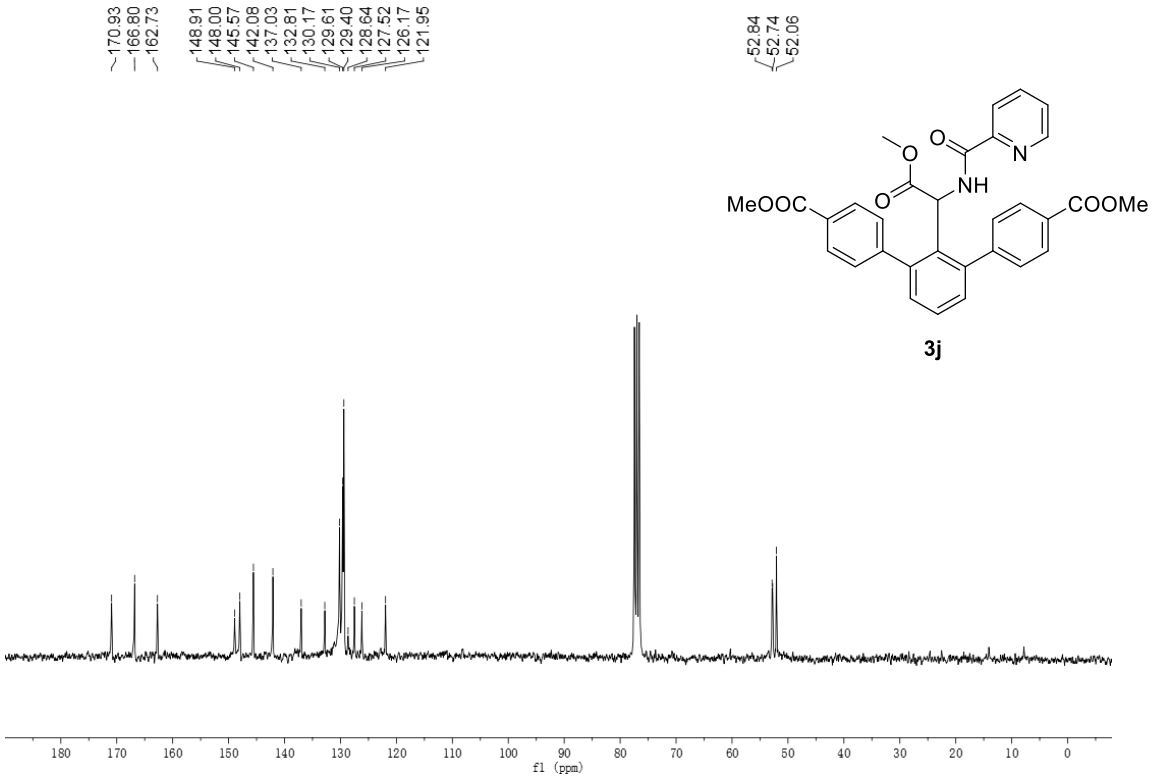
O
N
O
NH
O
NC
CN

3i


8.44
8.43
8.29
8.26
8.12
8.09
8.00
7.97
7.76
7.74
7.73
7.49
7.46
7.41
7.39
7.38
7.38
7.36
7.36
7.34
7.34
7.24
7.22
5.98
5.95
3.93
3.62
1.04
1.10
4.06
1.00
1.07
5.06
2.15
1.01
1.00
6.14
3.23
f1 (ppm)

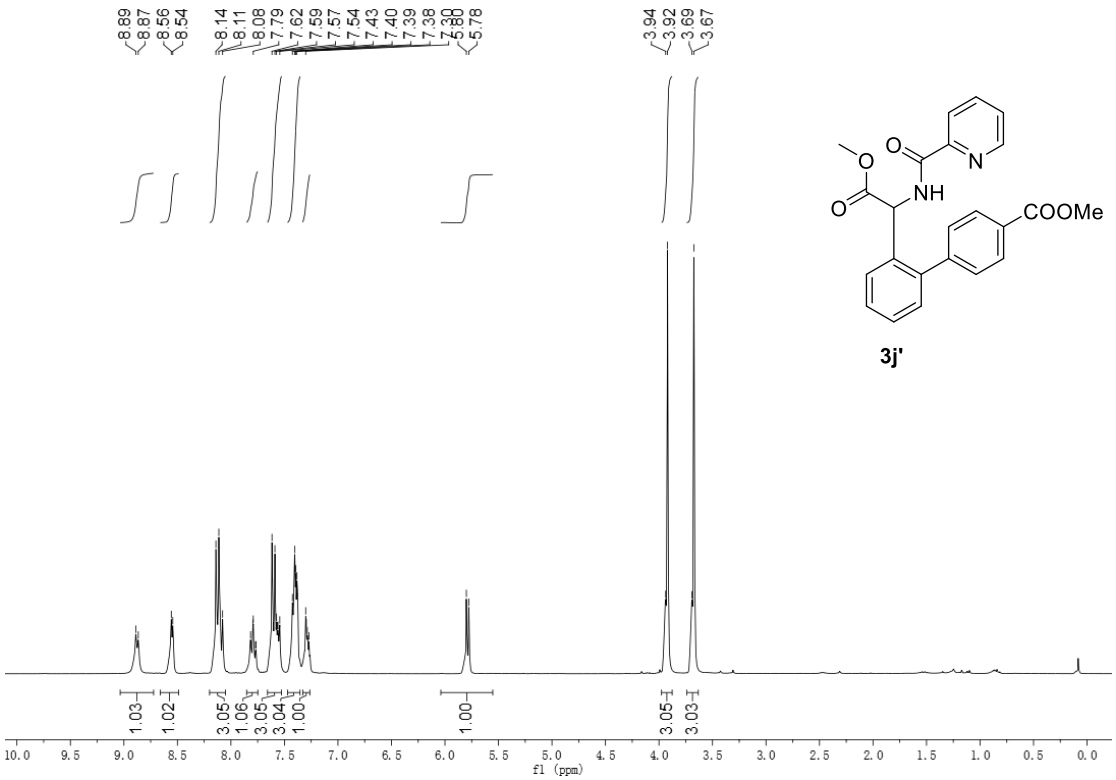
O
N
O
NH
O
MeOOC
COOMe

3j


170.93
166.80
162.73
148.91
148.00
145.57
142.08
137.03
132.81
130.17
129.61
129.40
128.64
127.52
126.17
121.95
52.84
52.74
52.06
f1 (ppm)

O
N
O
NH
O
MeOOC
COOMe

3j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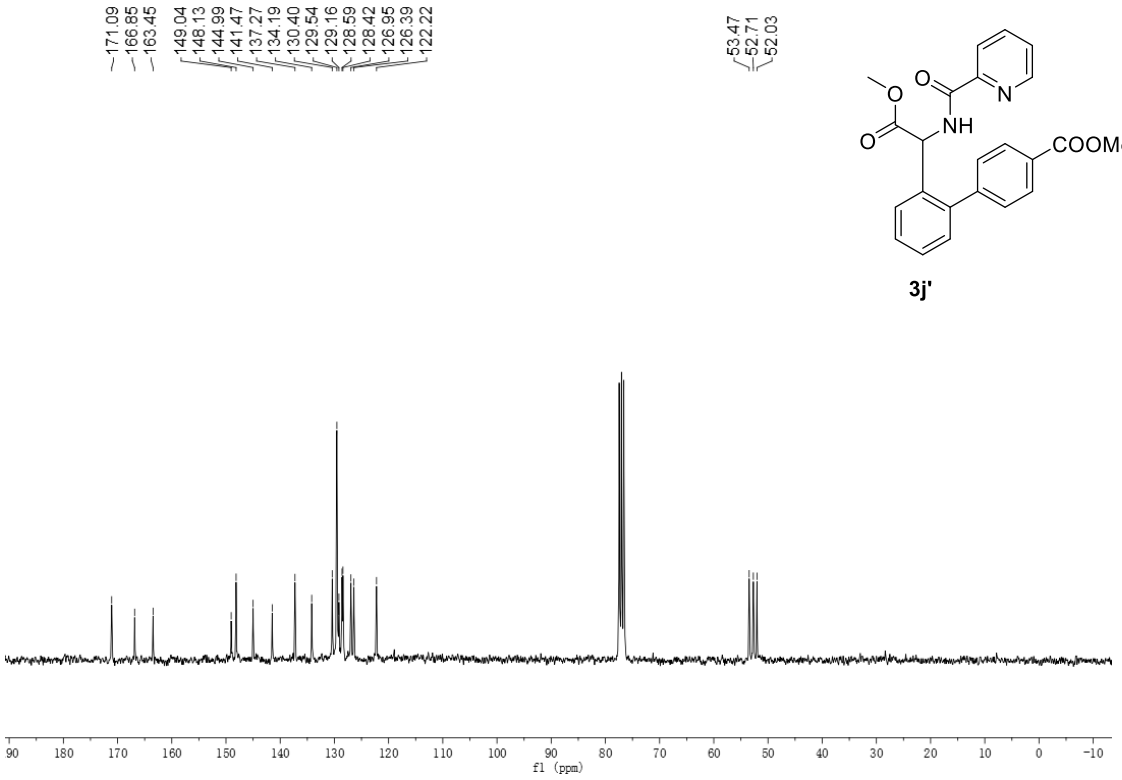
8.87
8.56
8.54
8.14
8.11
8.08
7.79
7.62
7.59
7.57
7.54
7.43
7.40
7.39
7.38
5.80
5.78
3.94
3.92
3.69
3.67
1.03
1.02
3.05
1.06
3.05
3.04
1.00
1.00
3.05
3.03
f1 (ppm)

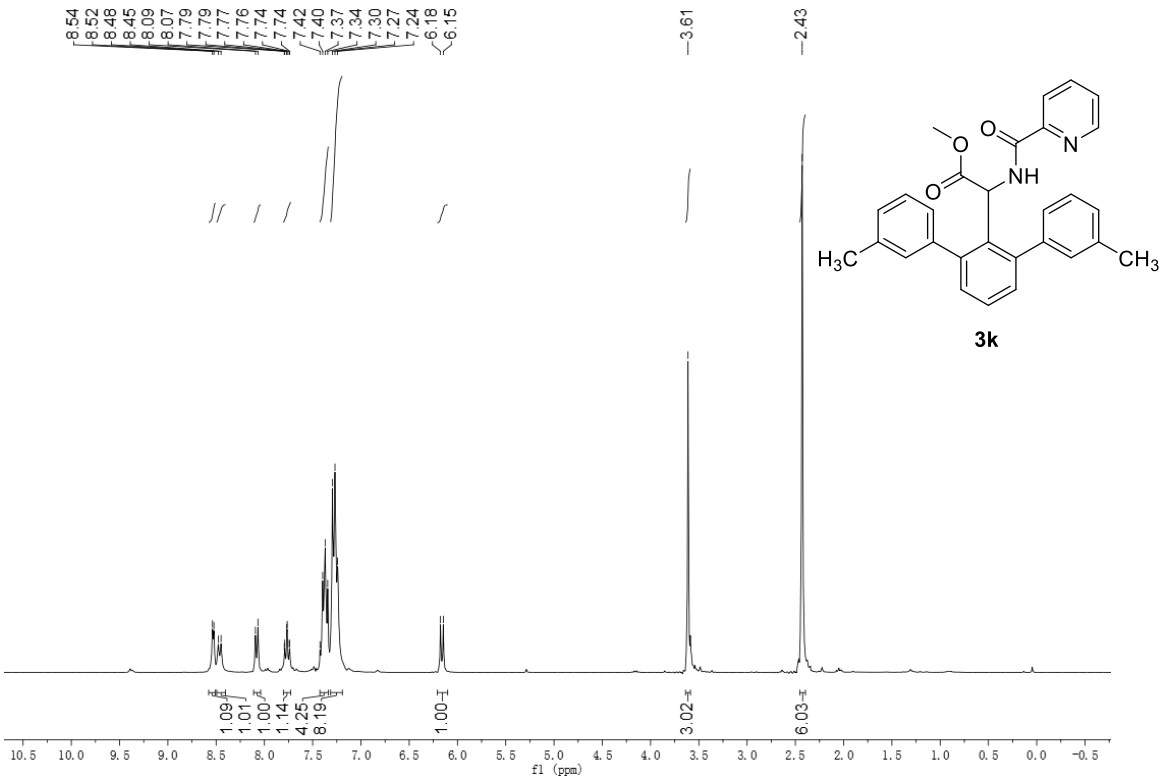
O
N
O
NH
O
COOMe

3j'


171.09
166.85
163.45
149.04
148.13
144.99
141.47
137.27
134.19
130.40
129.54
129.16
128.59
128.42
126.95
126.39
122.22
53.47
52.71
52.03
f1 (ppm)

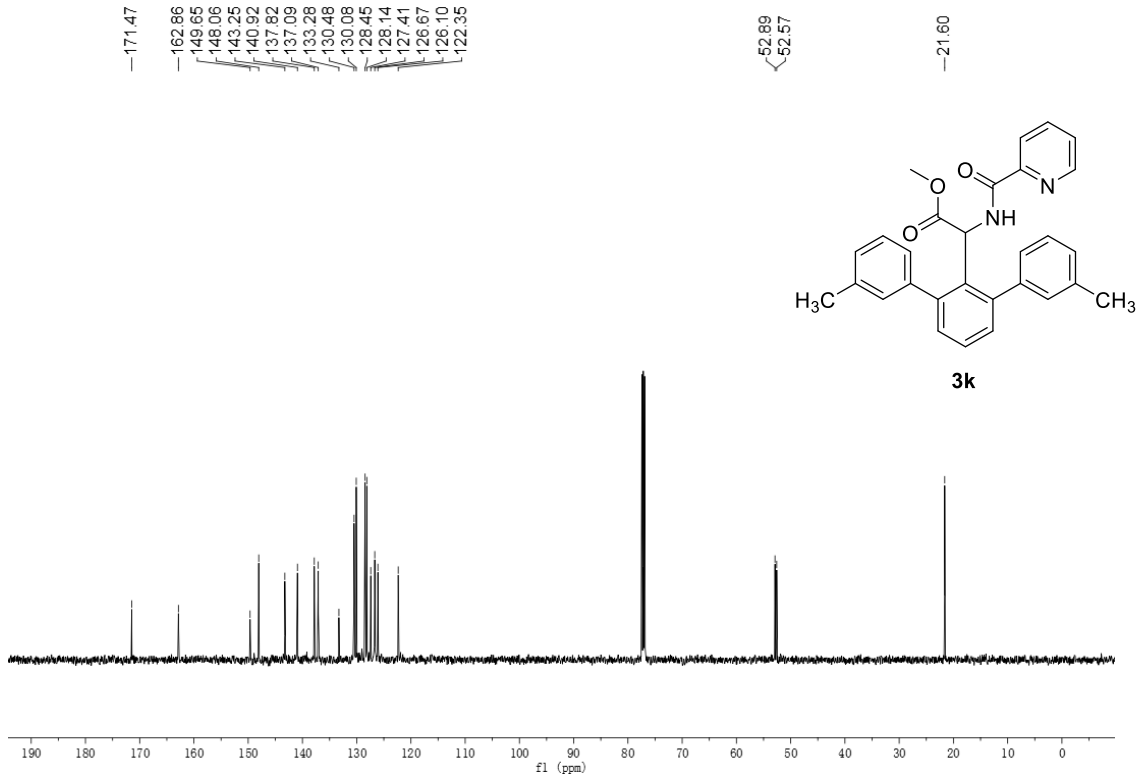
O
N
O
NH
O
COOMe

3j'


8.54
8.52
8.48
8.45
8.09
8.07
7.79
7.79
7.77
7.76
7.74
7.74
7.42
7.40
7.37
7.34
7.30
7.27
7.24
6.18
6.15
3.61
2.43
1.09
1.01
1.00
1.14
4.25
8.19
1.00
3.02
6.03
f1 (ppm)

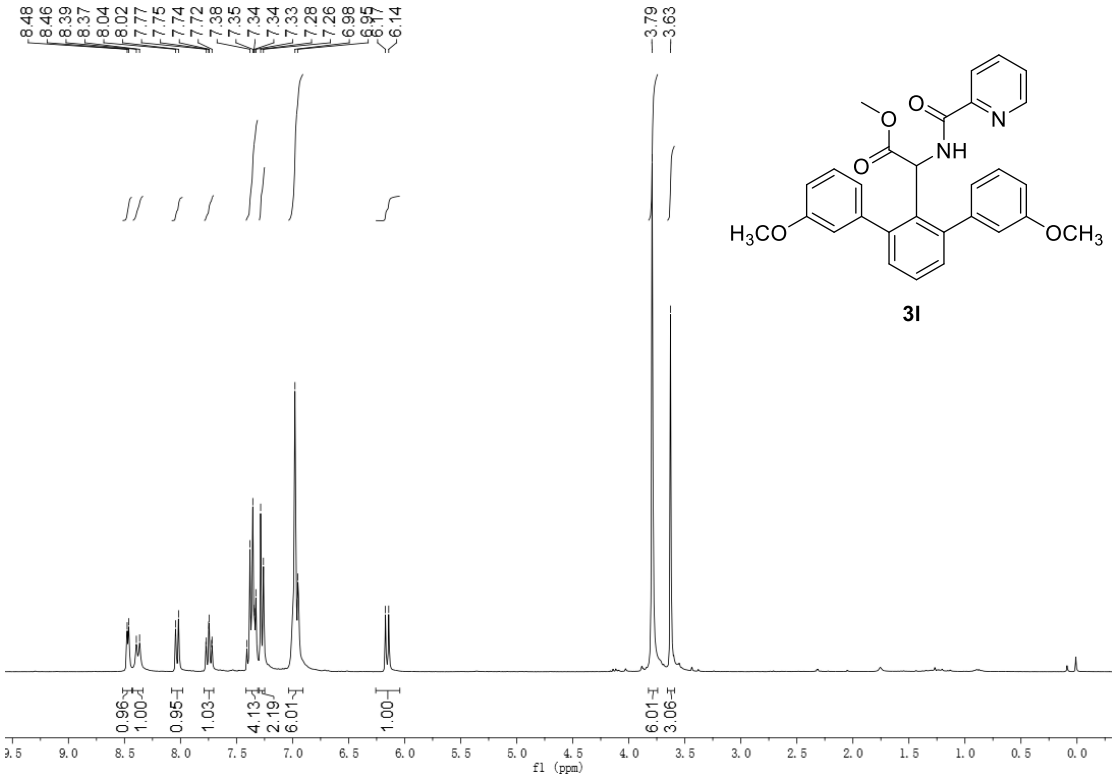
O
N
O
NH
O
H3C
CH3

3k


171.47
162.86
149.65
148.06
143.25
140.92
137.82
137.09
133.28
130.48
130.08
128.45
128.14
127.41
126.67
126.10
122.35
52.89
52.57
21.60
f1 (ppm)

O
N
O
NH
O
H3C
CH3

3k


8.48
8.46
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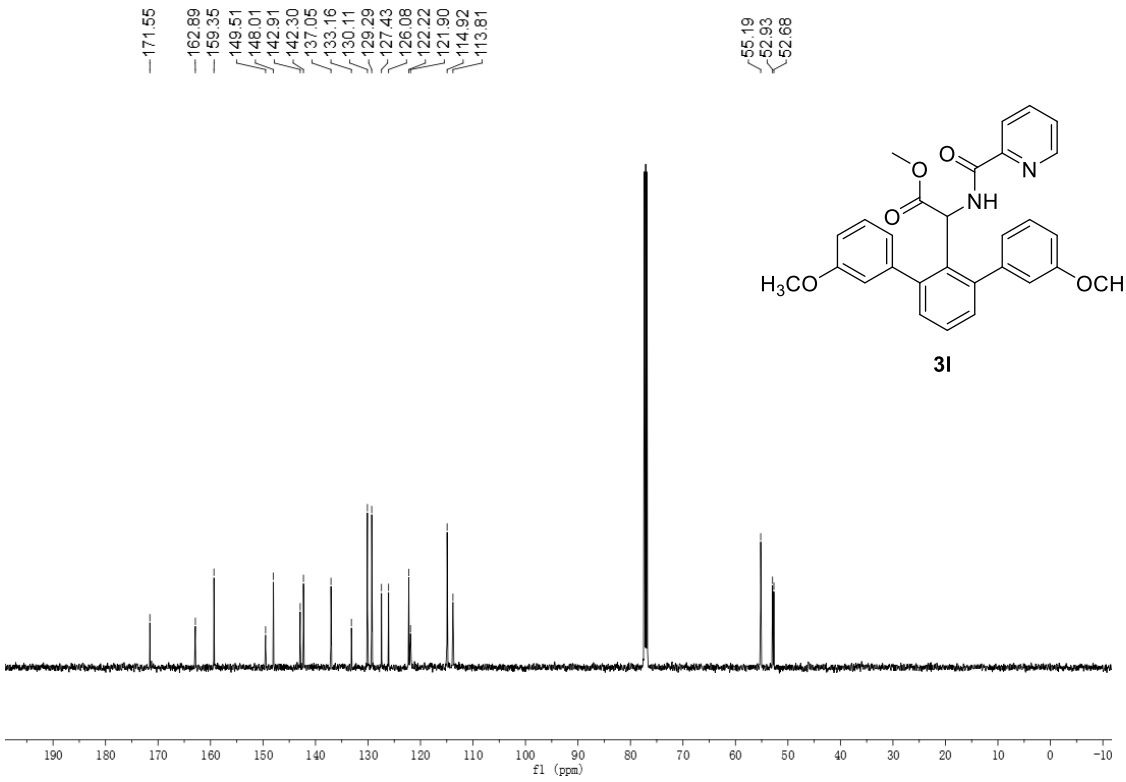
8.37
8.04
8.02
7.77
7.75
7.74
7.72
7.38
7.35
7.34
7.34
7.33
7.28
7.26
6.98
6.95
6.17
6.14
3.79
3.63
0.96
1.00
0.95
1.03
4.13
2.19
6.01
1.00
6.01
3.06
f1 (ppm)

O
N
O
NH
O
H3CO
OCH3

3l


171.55
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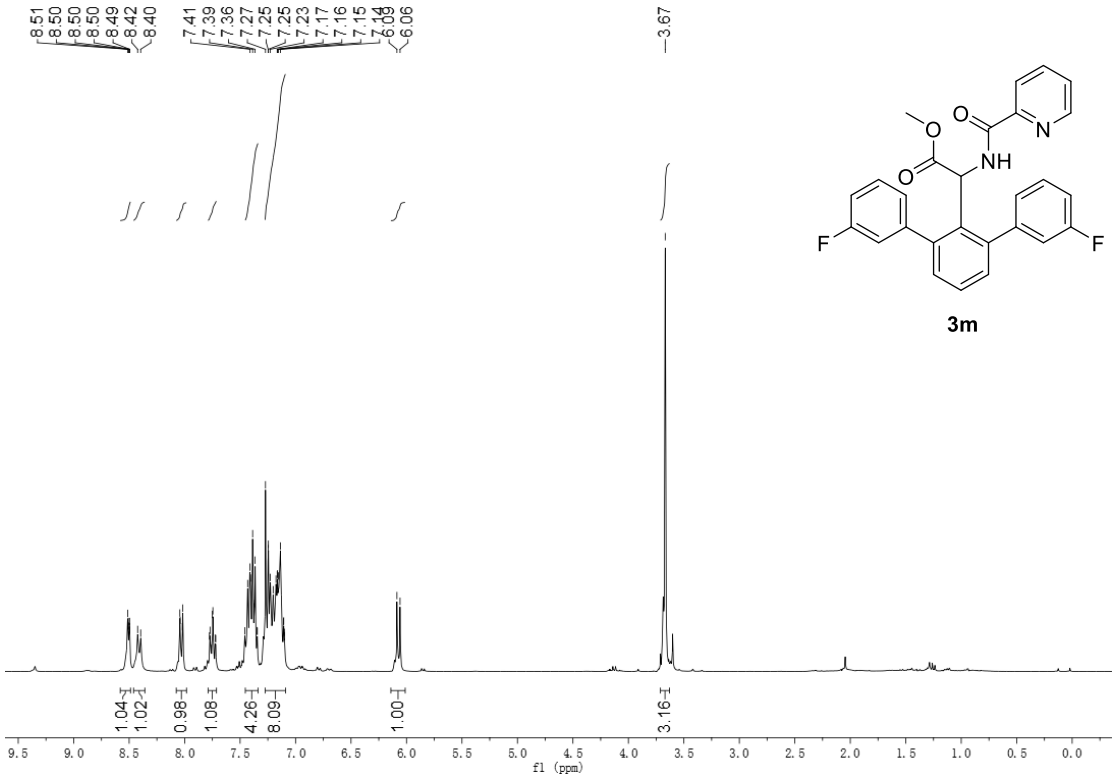
159.35
149.51
148.01
142.91
142.30
137.05
133.16
130.11
129.29
127.43
126.08
122.22
121.90
114.92
113.81
55.19
52.93
52.68
f1 (ppm)

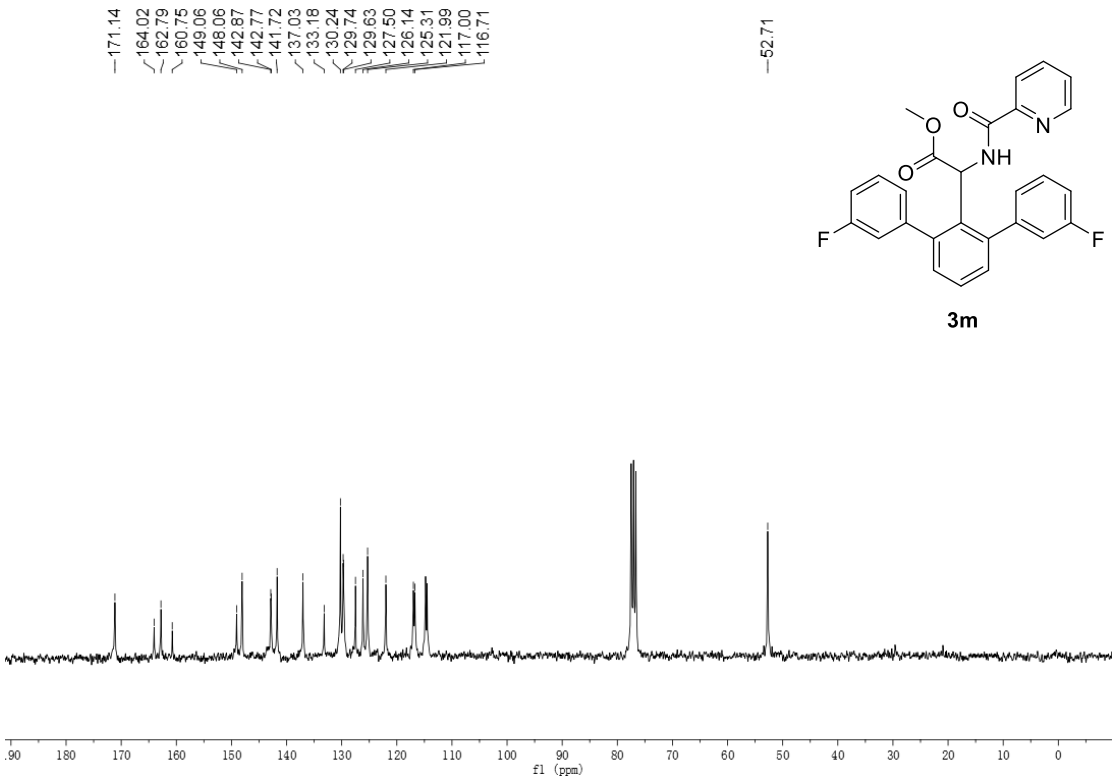
O
N
O
NH
O
H3CO
OCH3

3l


8.51
8.50
8.50
8.50
8.49
8.42
8.40
7.41
7.39
7.36
7.27
7.25
7.25
7.23
7.17
7.16
7.15
7.14
6.08
6.06
3.67
1.04
1.02
0.98
1.08
4.26
8.09
1.00
3.16
f1 (ppm)

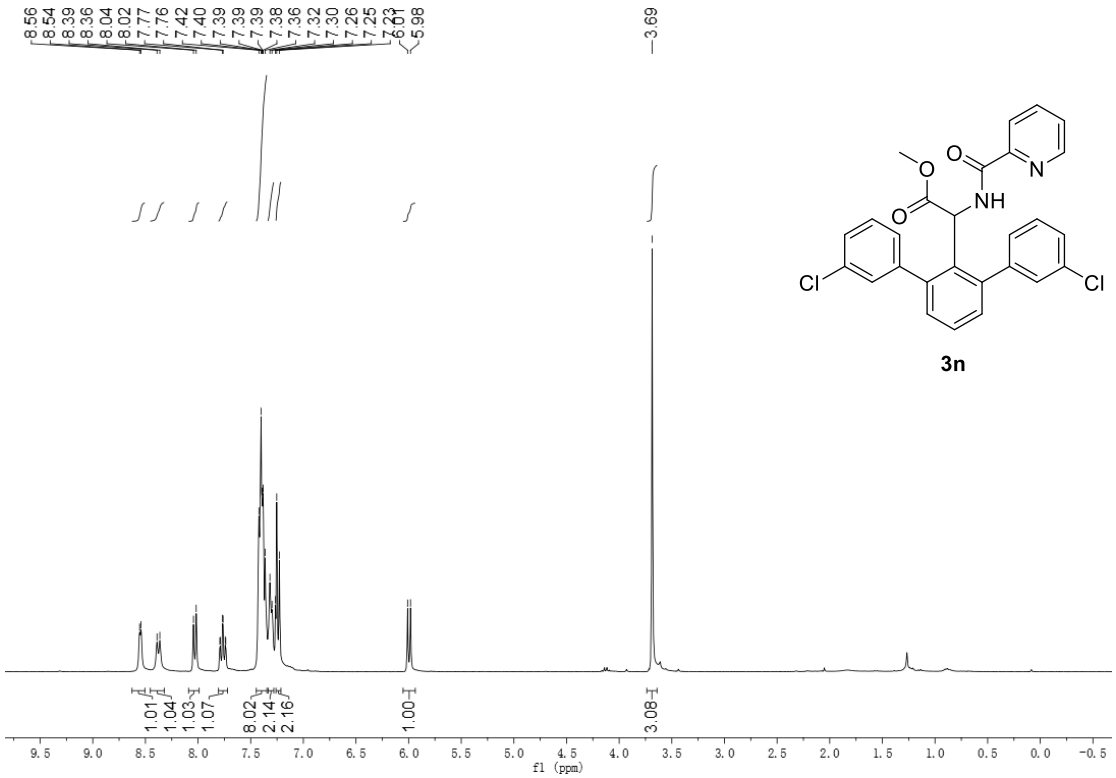
O
N
O
NH
O
F
F

3m


171.14
164.02
162.79
160.75
149.06
148.06
142.87
142.77
141.72
137.03
133.18
130.24
129.74
129.63
127.50
126.14
125.31
121.99
117.00
116.71
52.71
f1 (ppm)

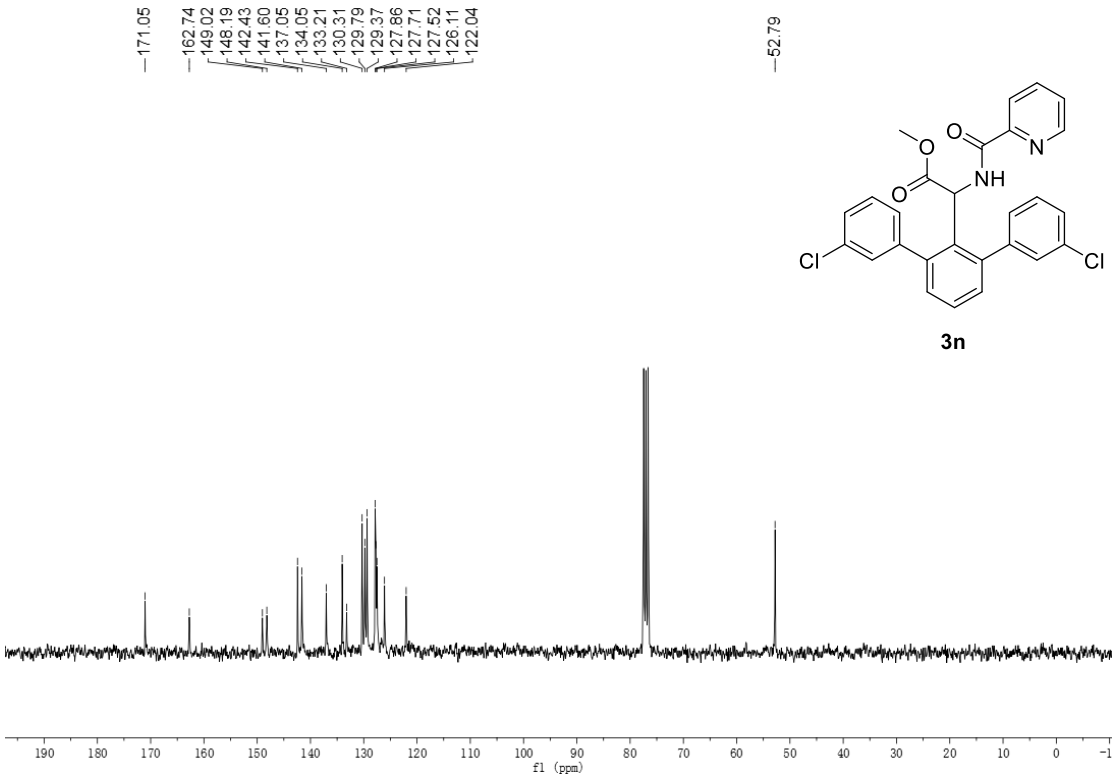
O
N
O
NH
O
F
F

3m


8.56
8.54
8.39
8.36
8.04
8.02
7.77
7.76
7.42
7.40
7.39
7.39
7.39
7.38
7.36
7.32
7.30
7.26
7.25
7.23
6.01
5.98
3.69
1.01
1.04
1.03
1.07
8.02
2.14
2.16
1.00
3.08
f1 (ppm)

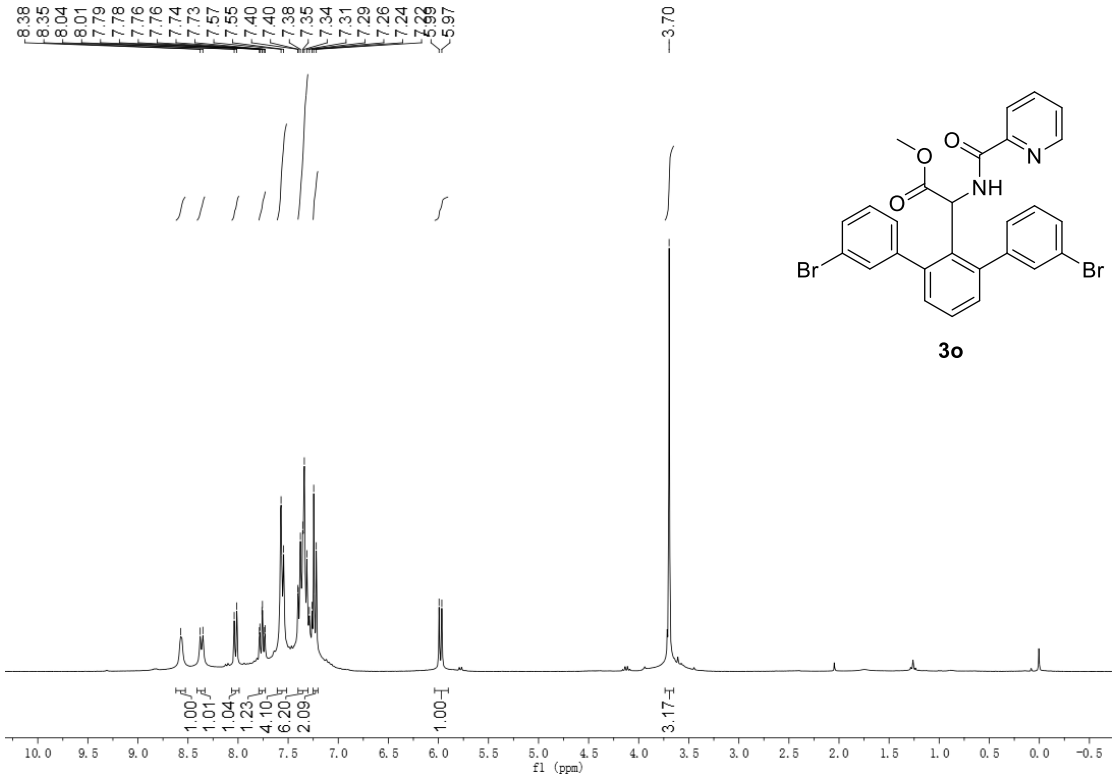
O
N
O
NH
O
Cl
Cl

3n


171.05
162.74
149.02
148.19
142.43
141.60
137.05
134.05
133.21
130.31
129.79
129.37
127.86
127.71
127.52
126.11
122.04
52.79
f1 (ppm)

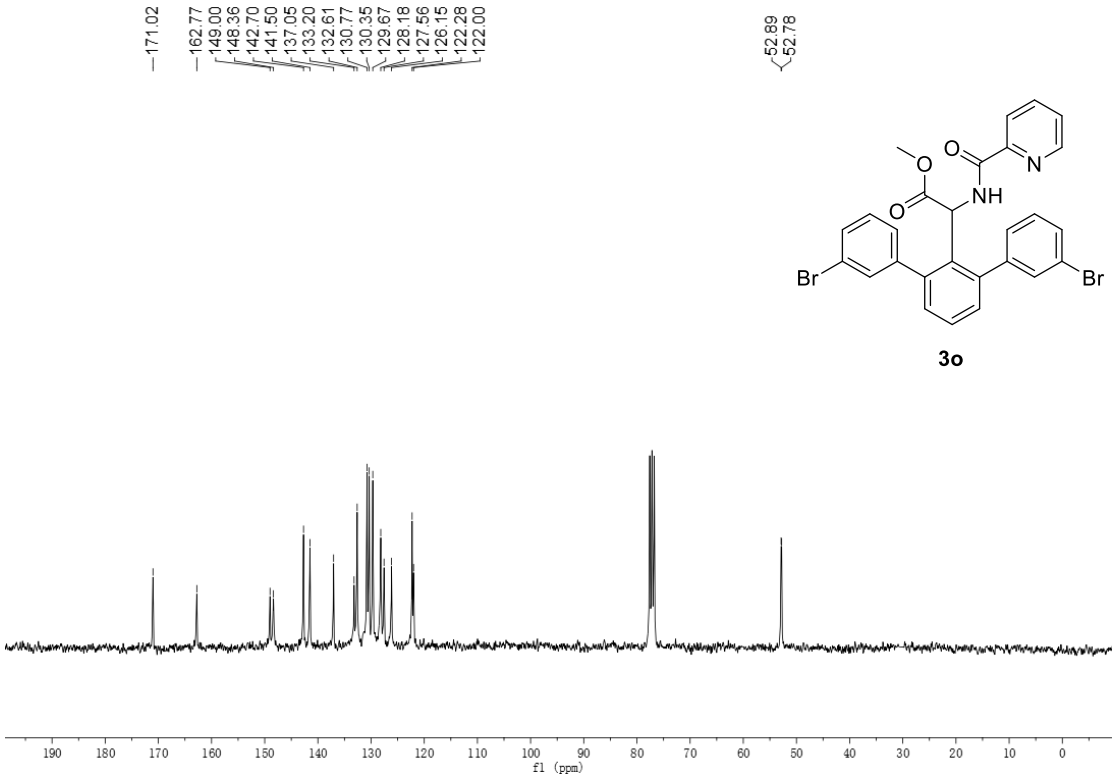
O
N
O
NH
O
Cl
Cl

3n


8.38
8.35
8.04
8.01
7.79
7.78
7.76
7.76
7.74
7.73
7.57
7.55
7.40
7.40
7.38
7.35
7.34
7.31
7.29
7.26
7.24
5.97
3.70
1.00
1.01
1.04
1.23
4.10
6.20
2.09
1.00
3.17
f1 (ppm)

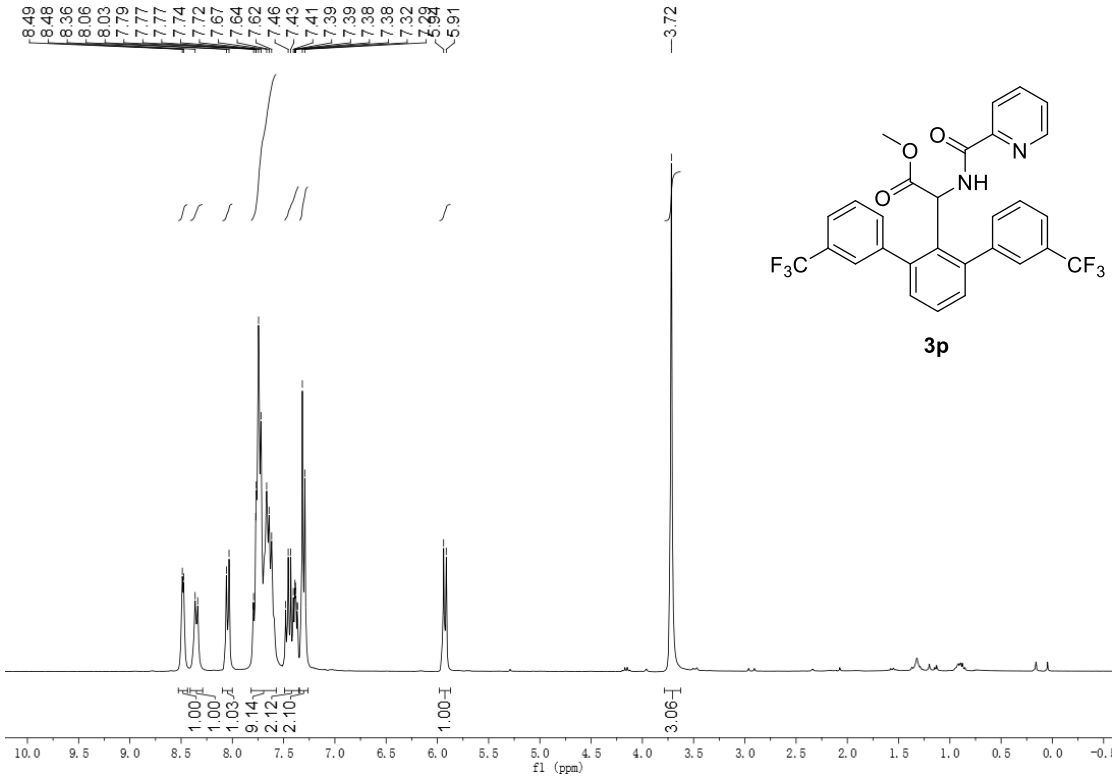
O
N
O
NH
O
Br
Br

3o


171.02
162.77
149.00
148.36
142.70
141.50
137.05
133.20
132.61
130.77
130.35
129.67
128.18
127.56
126.15
122.28
122.00
52.89
52.78
f1 (ppm)

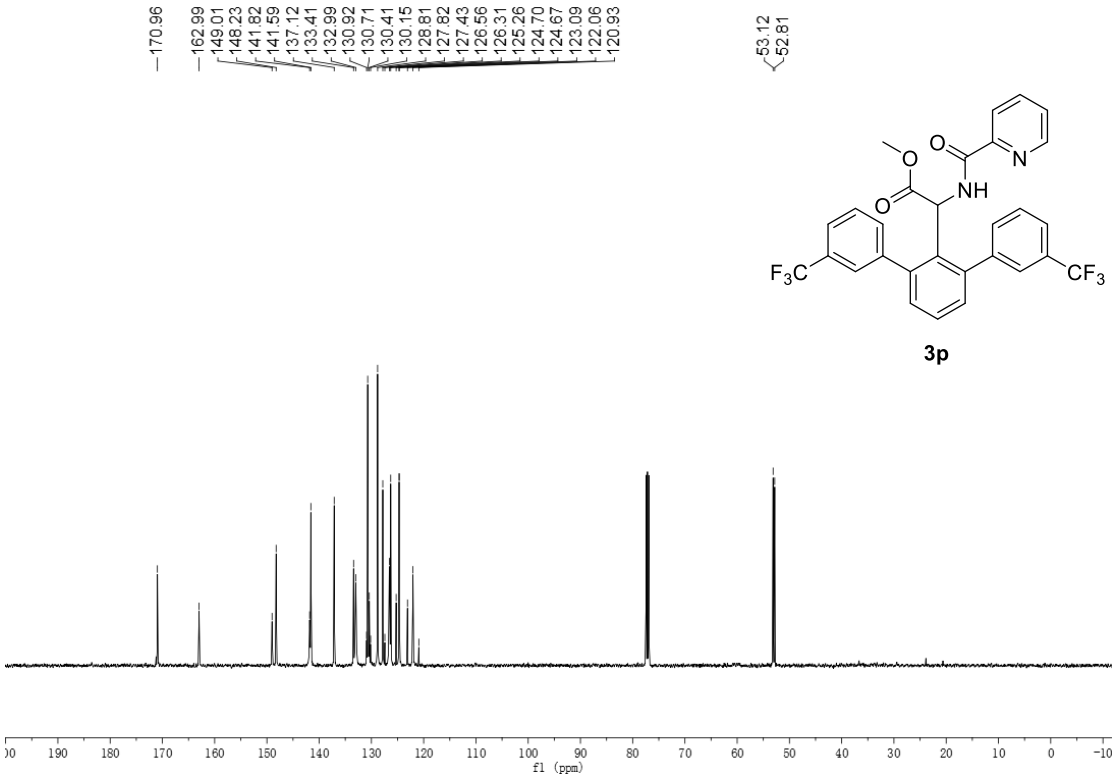
O
N
O
NH
O
Br
Br

3o


8.49
8.48
8.36
8.06
8.03
7.79
7.77
7.77
7.74
7.72
7.67
7.64
7.62
7.46
7.43
7.41
7.39
7.39
7.38
7.38
7.32
7.29
5.94
5.91
3.72
1.00
1.00
1.03
9.14
2.12
2.10
1.00
3.06
f1 (ppm)

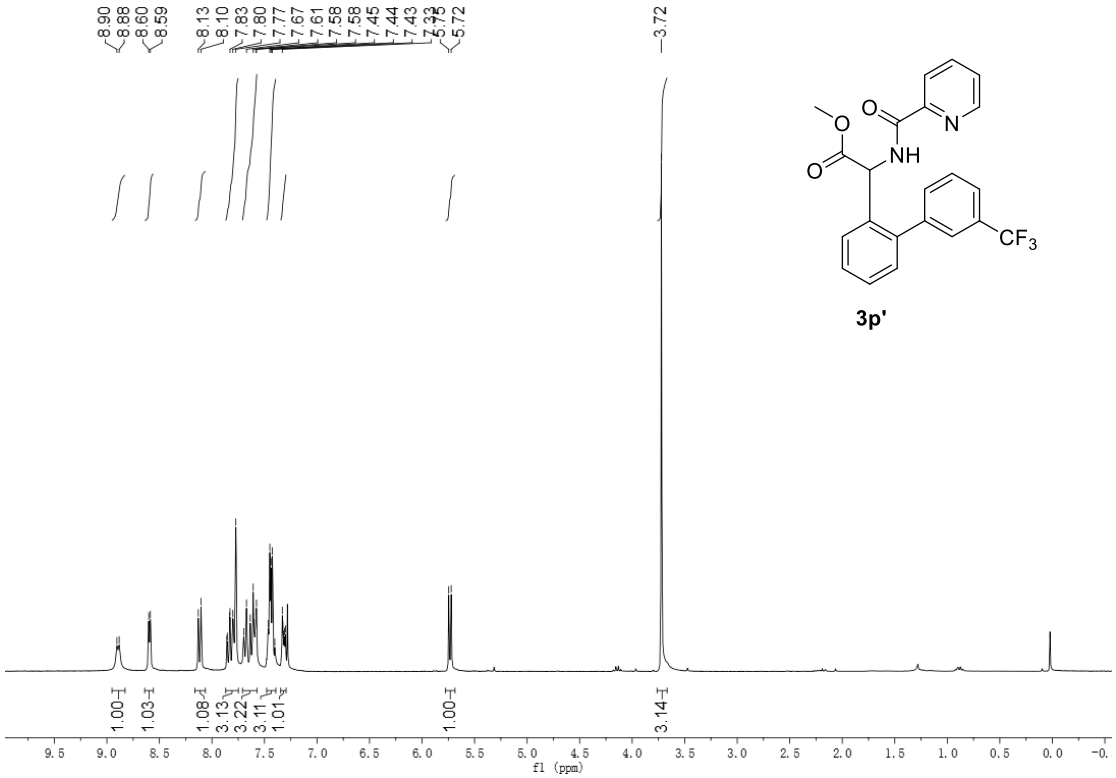
O
N
O
NH
O
F3C
CF3

3p


170.96
162.99
149.01
148.23
141.82
141.59
137.12
133.41
132.99
130.92
130.71
130.41
130.15
128.81
127.82
127.43
126.56
126.31
125.26
124.70
124.67
123.09
122.06
120.93
53.12
52.81
f1 (ppm)

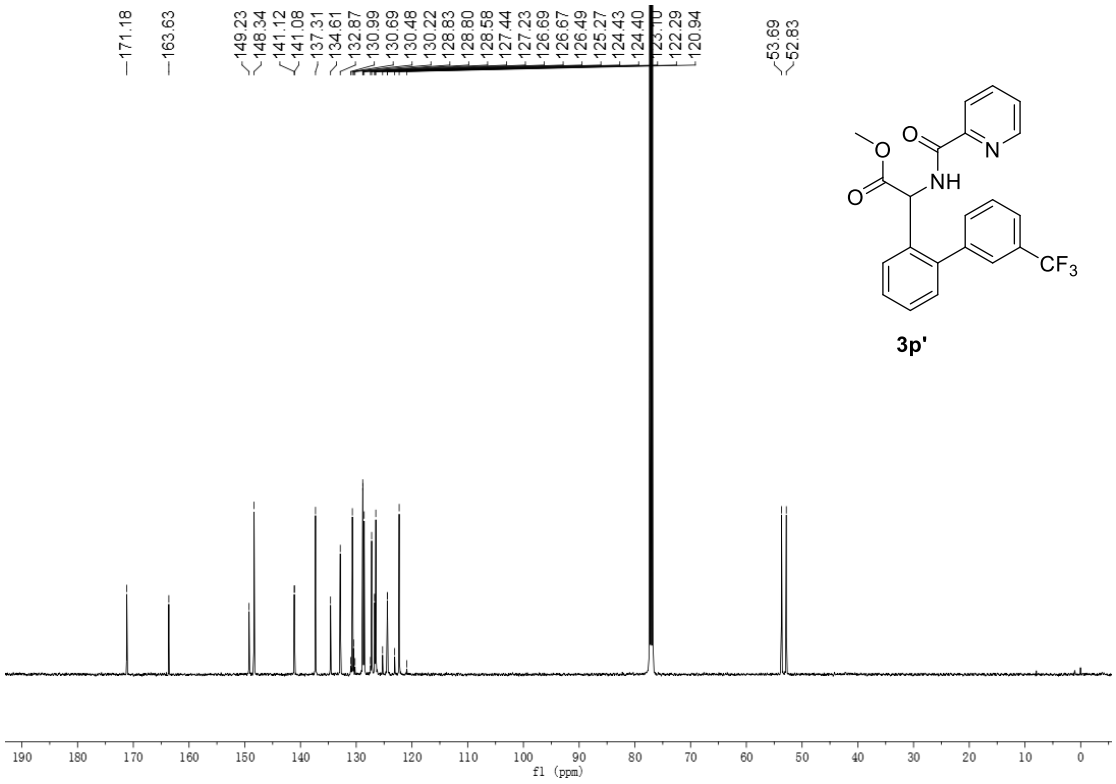
O
N
O
NH
O
F3C
CF3

3p


8.90
8.88
8.60
8.59
8.13
8.10
7.83
7.80
7.77
7.67
7.61
7.58
7.58
7.45
7.44
7.43
7.33
5.75
5.72
3.72
1.00
1.03
1.08
3.13
3.22
3.11
1.01
1.00
3.14
f1 (ppm)

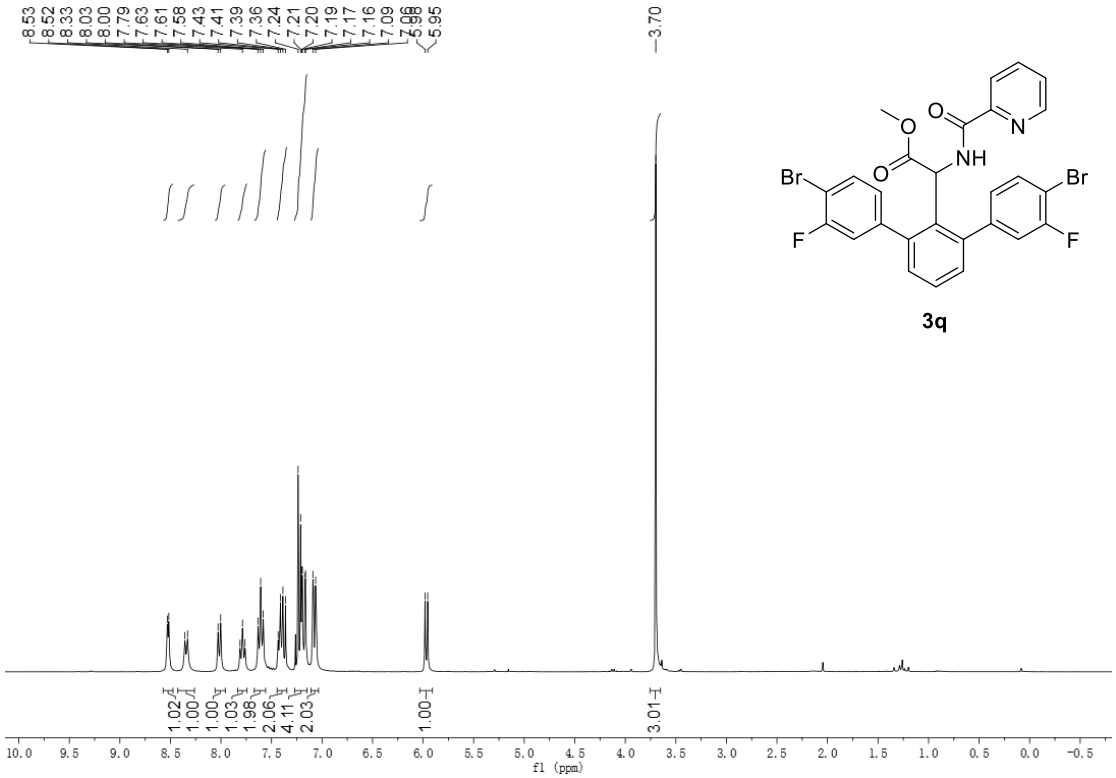
O
N
O
NH
O
CF3

3p'

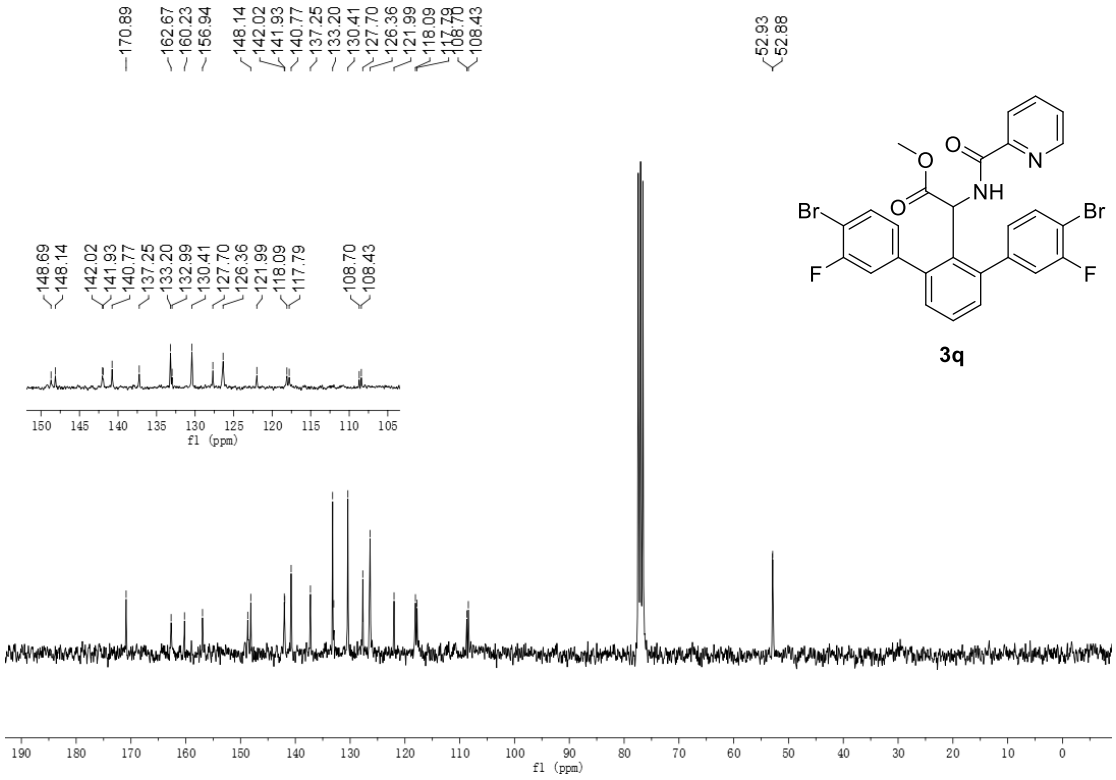

171.18
163.63
149.23
148.34
141.12
141.08
137.31
134.61
132.87
130.99
130.69
130.48
130.22
128.83
128.80
128.58
127.44
127.23
126.69
126.67
126.49
125.27
124.43
124.40
123.10
122.29
120.94
53.69
52.83
f1 (ppm)

O
N
O
NH
O
CF3

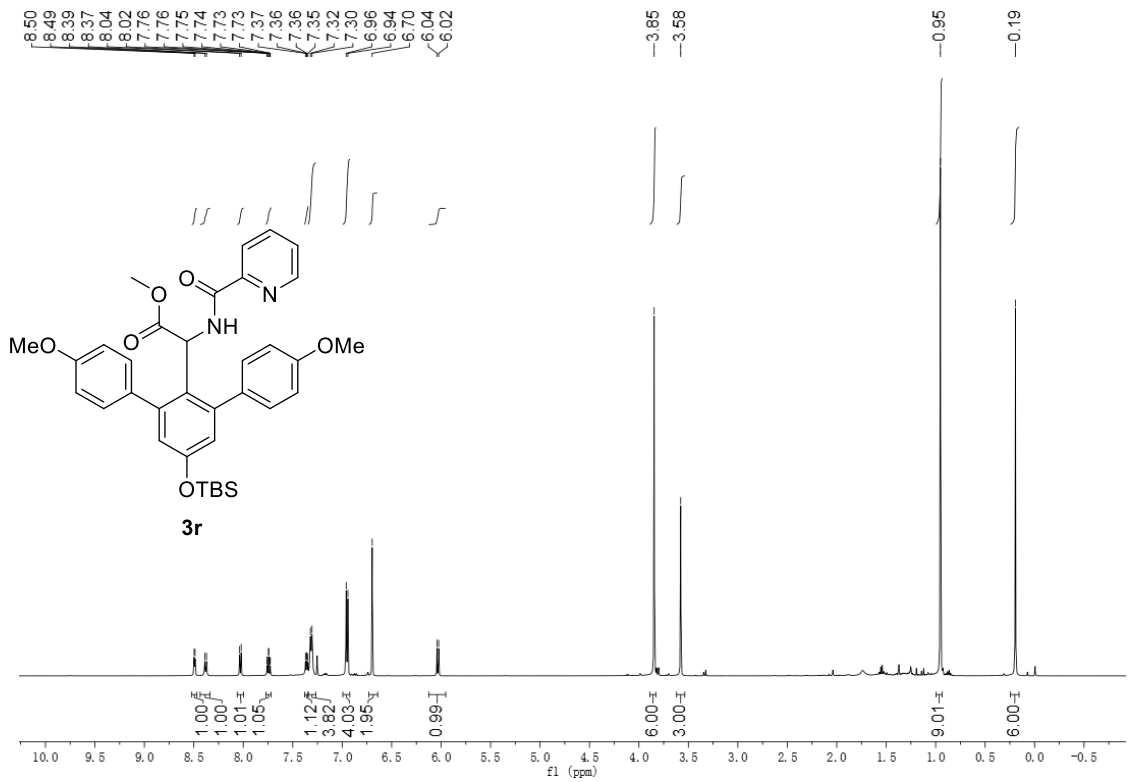
3p'


8.53
8.52
8.33
8.03
8.00
7.79
7.63
7.61
7.58
7.43
7.41
7.39
7.36
7.24
7.21
7.20
7.19
7.17
7.16
7.09
5.98
5.95
3.70
1.02
1.00
1.00
1.03
1.98
2.06
4.11
2.03
1.00
3.01
f1 (ppm)
O
N
O
NH
Br
F
Br
F

3q


170.89
162.67
160.23
156.94
148.14
142.02
141.93
140.77
137.25
133.20
130.41
127.70
126.36
121.99
118.09
117.79
108.70
108.43
52.93
52.88
148.69
148.14
142.02
141.93
140.77
137.25
133.20
132.99
130.41
127.70
126.36
121.99
118.09
117.79
108.70
108.43
f1 (ppm)
O
N
O
NH
Br
F
Br
F

3q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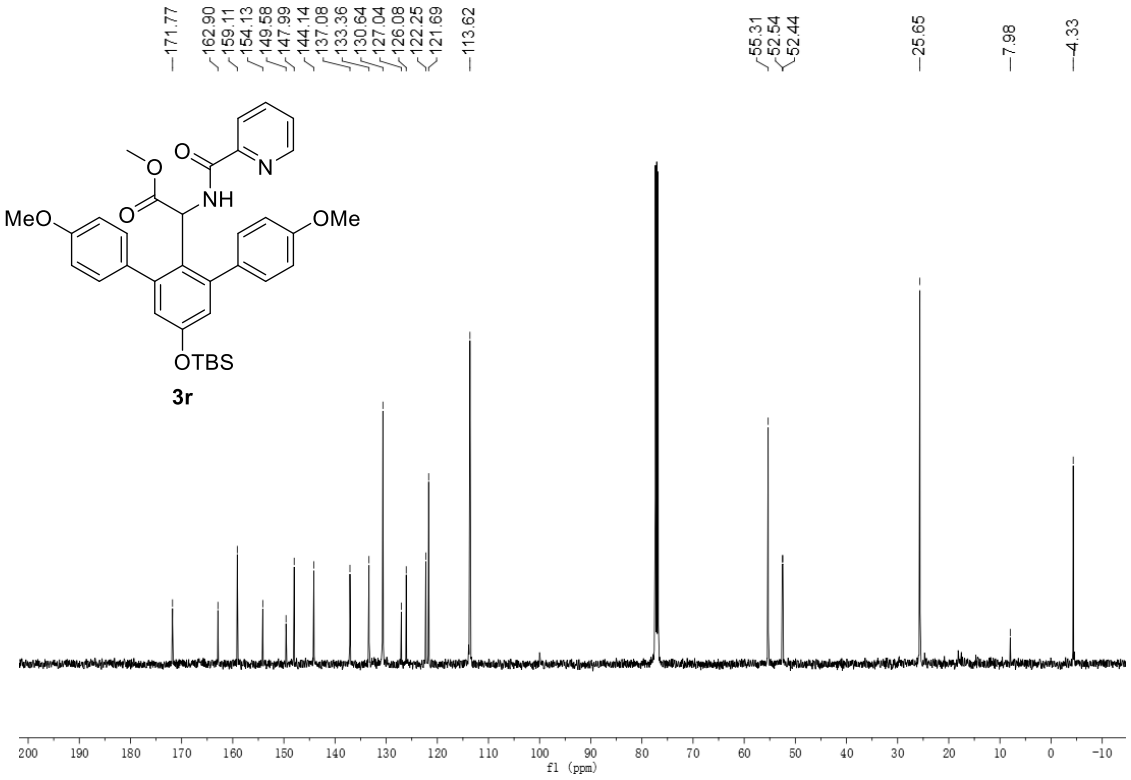
8.49
8.39
8.37
8.04
8.02
7.76
7.76
7.75
7.74
7.73
7.73
7.37
7.36
7.36
7.35
7.32
7.30
6.96
6.94
6.70
6.04
6.02
3.85
3.58
0.95
0.19
MeO
OMe
OTBS
3r
1.00
1.00
1.01
1.05
1.12
3.82
4.03
1.95
0.99
6.00
3.00
9.01
6.00
f1 (ppm)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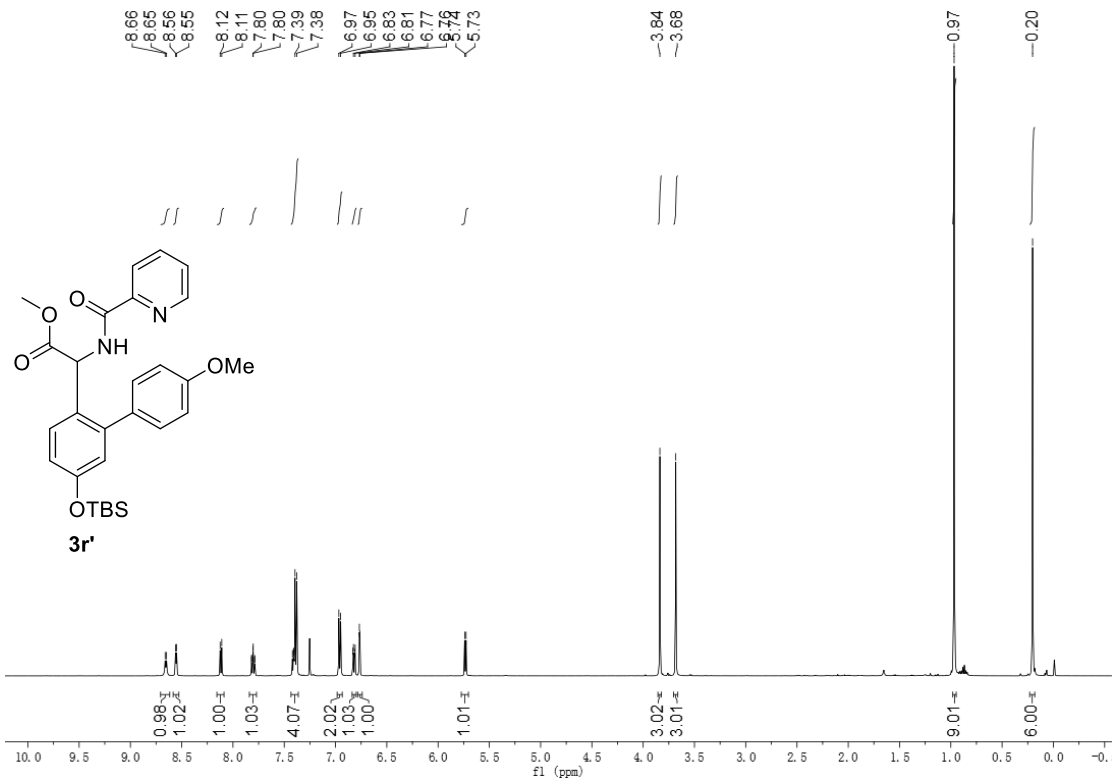
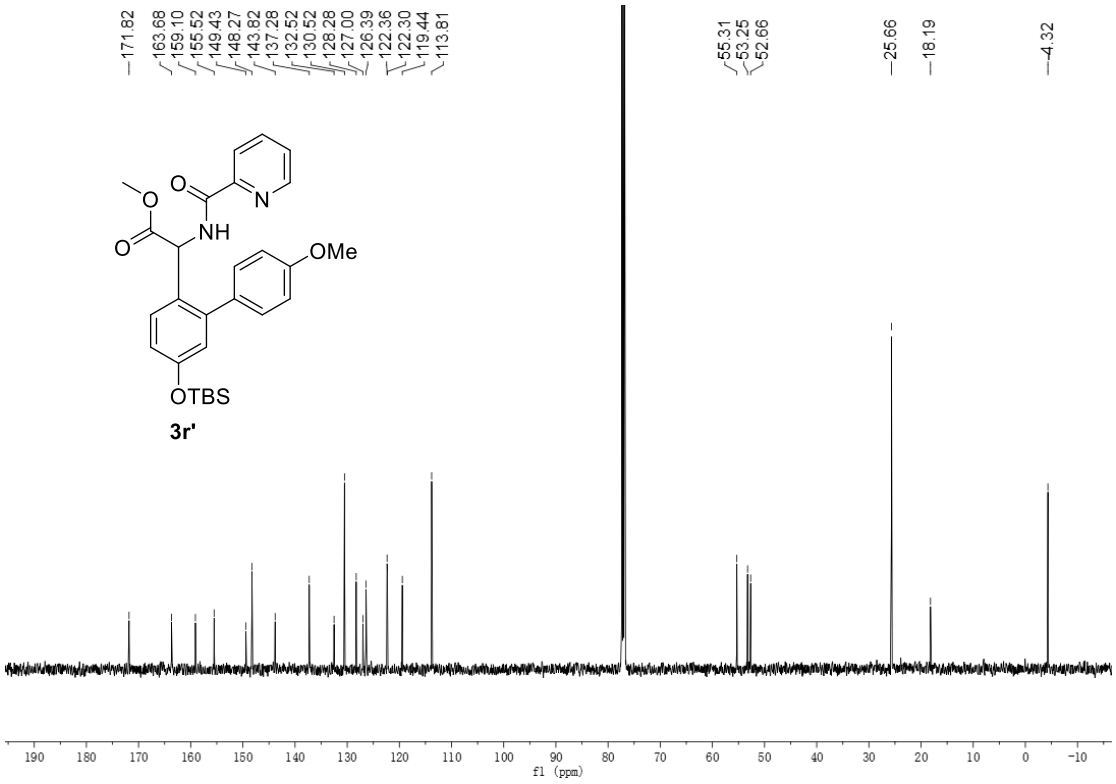
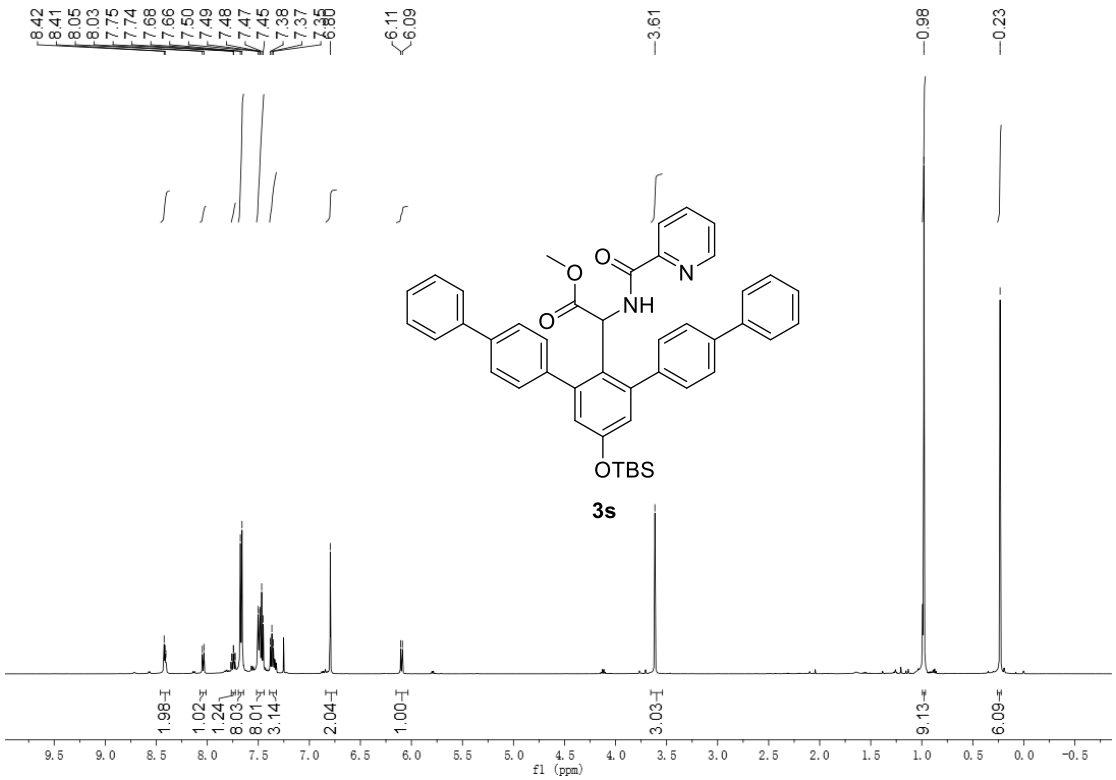
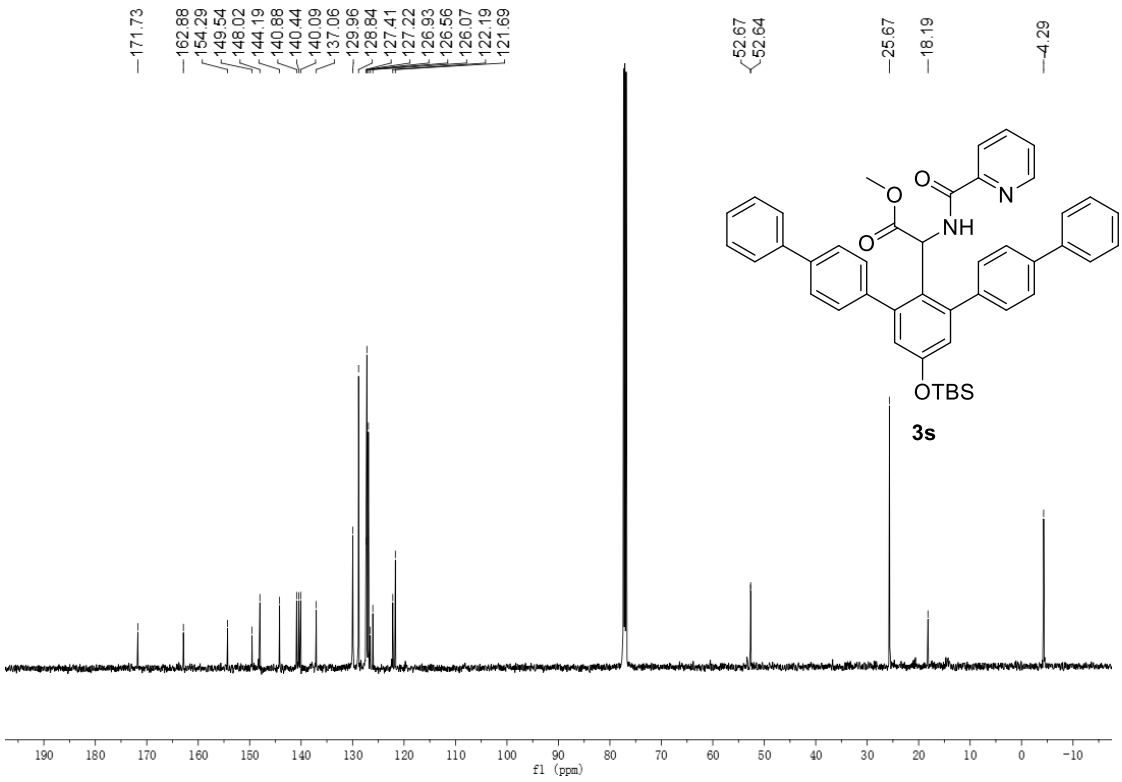
162.90
159.11
154.13
149.58
147.99
144.14
137.08
133.36
130.64
127.04
126.08
122.25
121.69
113.62
55.31
52.54
52.44
25.65
7.98
4.33
MeO
OMe
OTBS
3r
f1 (ppm)


8.66
8.65
8.56
8.55
8.12
8.11
7.80
7.80
7.39
7.38
6.97
6.95
6.83
6.81
6.77
6.76
5.74
5.73
3.84
3.68
0.97
0.20
O
N
O
NH
OMe
OTBS
3r'
0.98
1.02
1.00
1.03
4.07
2.02
1.03
1.00
1.01
3.02
3.01
9.01
6.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)


171.82
163.68
159.10
155.52
149.43
148.27
143.82
137.28
132.52
130.52
128.28
127.00
126.39
122.36
122.30
119.44
113.81
55.31
53.25
52.66
25.66
18.19
4.32
O
N
O
NH
OMe
OTBS
3r'
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.42
8.41
8.05
8.03
7.75
7.74
7.68
7.66
7.50
7.49
7.48
7.47
7.45
7.38
7.37
7.35
6.11
6.09
3.61
0.98
0.23
O
N
NH
O
O
OTBS
3s
1.98
1.02
1.24
8.03
8.01
3.14
2.04
1.00
3.03
9.13
6.09
f1 (ppm)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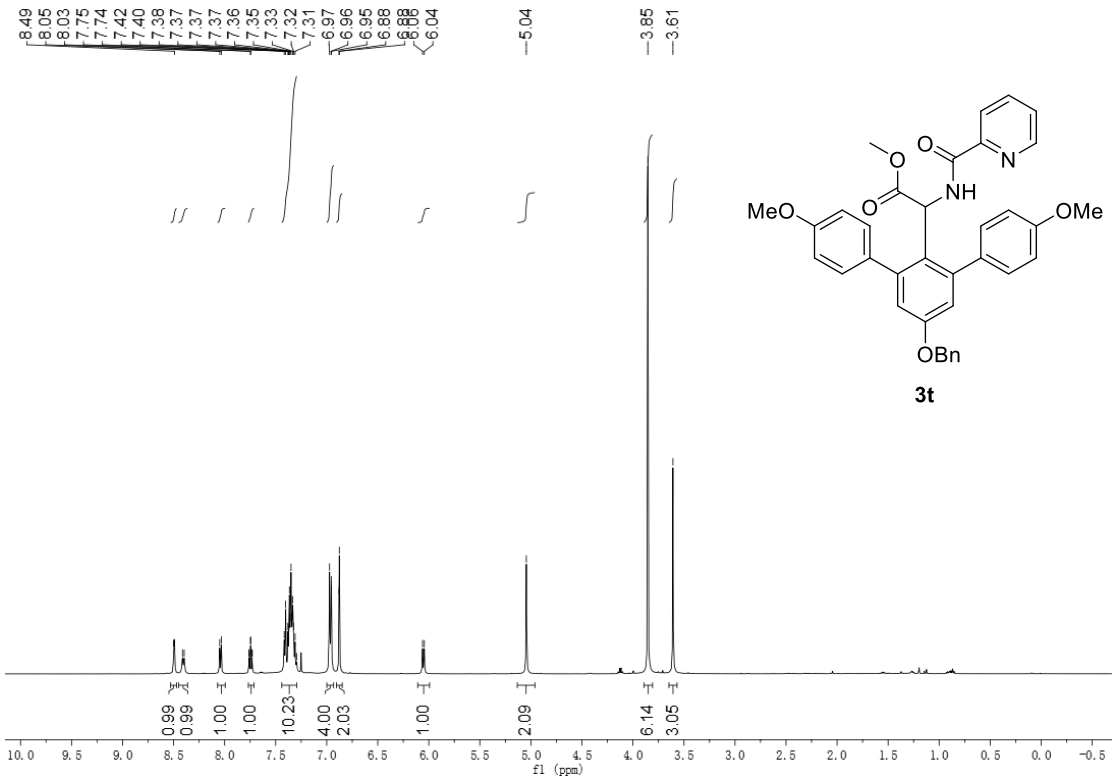
162.88
154.29
149.54
148.02
144.19
140.88
140.44
140.09
137.06
129.96
128.84
127.41
127.22
126.93
126.56
126.07
122.19
121.69
52.67
52.64
25.67
18.19
4.29
O
N
NH
O
O
OTBS
3s
f1 (ppm)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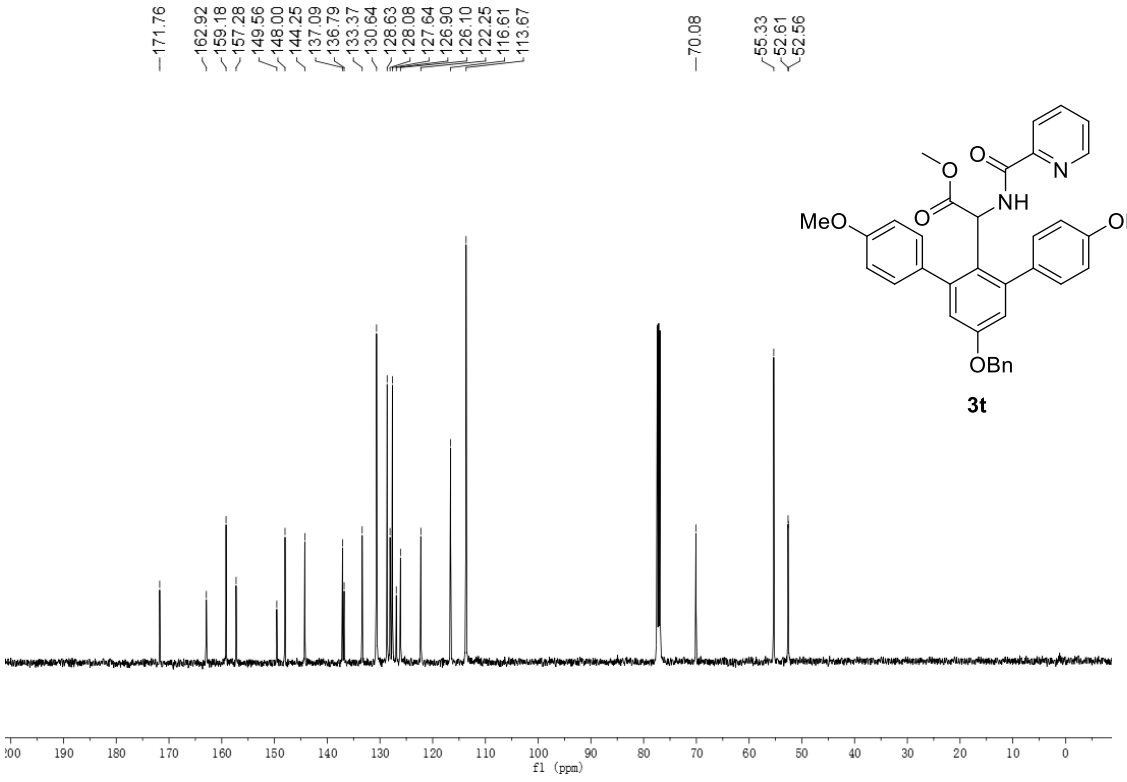
8.05
8.03
7.75
7.74
7.42
7.40
7.38
7.37
7.37
7.37
7.36
7.35
7.33
7.32
7.31
6.97
6.96
6.95
6.88
6.04
5.04
3.85
3.61
MeO
OMe
OBn
0.99
0.99
1.00
1.00
10.23
4.00
2.03
1.00
2.09
6.14
3.05
f1 (ppm)

3t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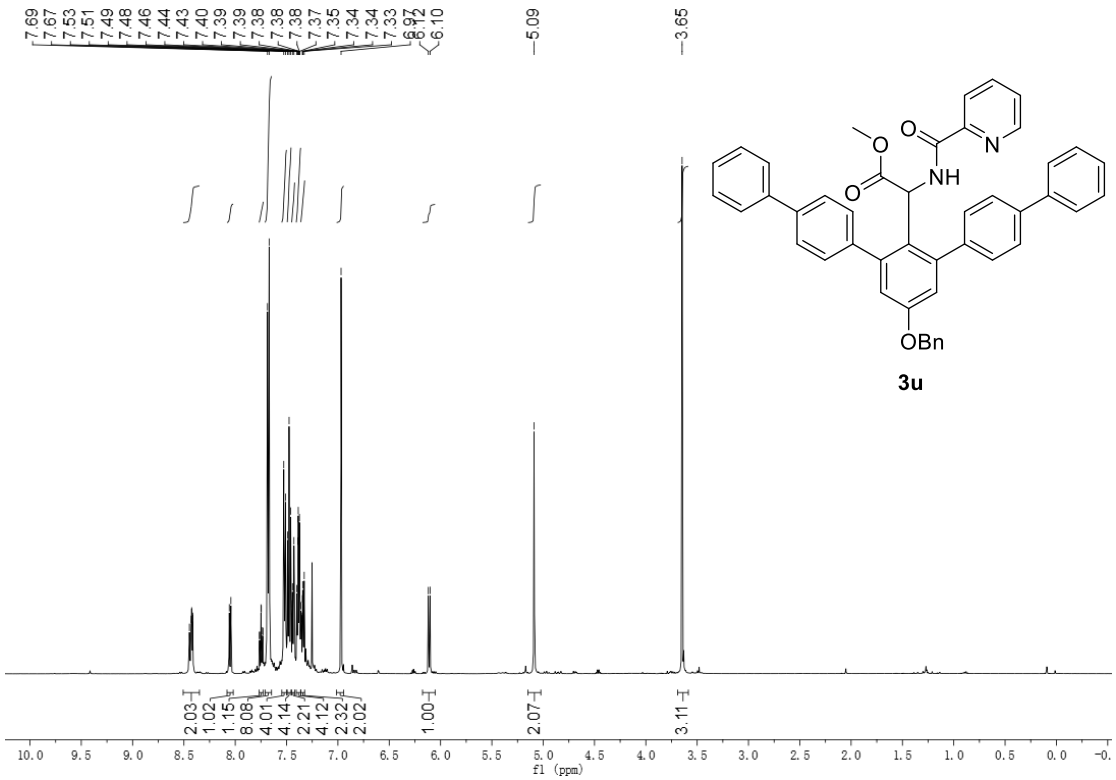
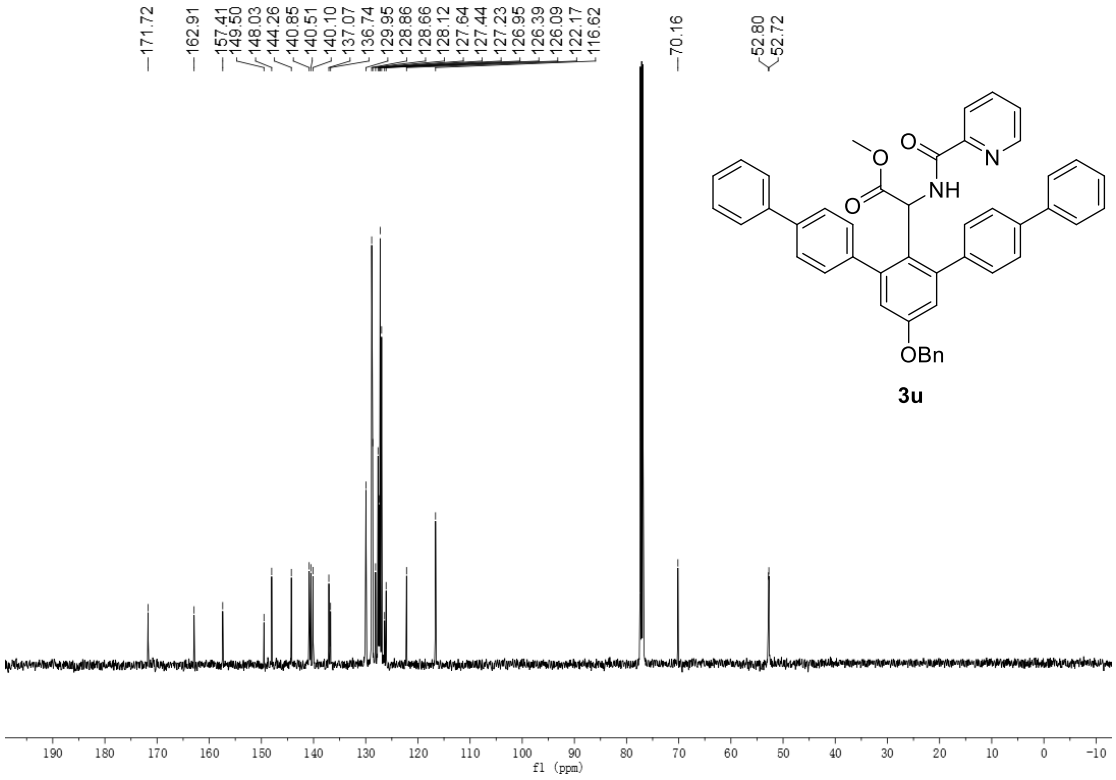
162.92
159.18
157.28
149.56
148.00
144.25
137.09
136.79
133.37
130.64
128.63
128.08
127.64
126.90
126.10
122.25
116.61
113.67
70.08
55.33
52.61
52.56
MeO
OMe
OBn
f1 (ppm)

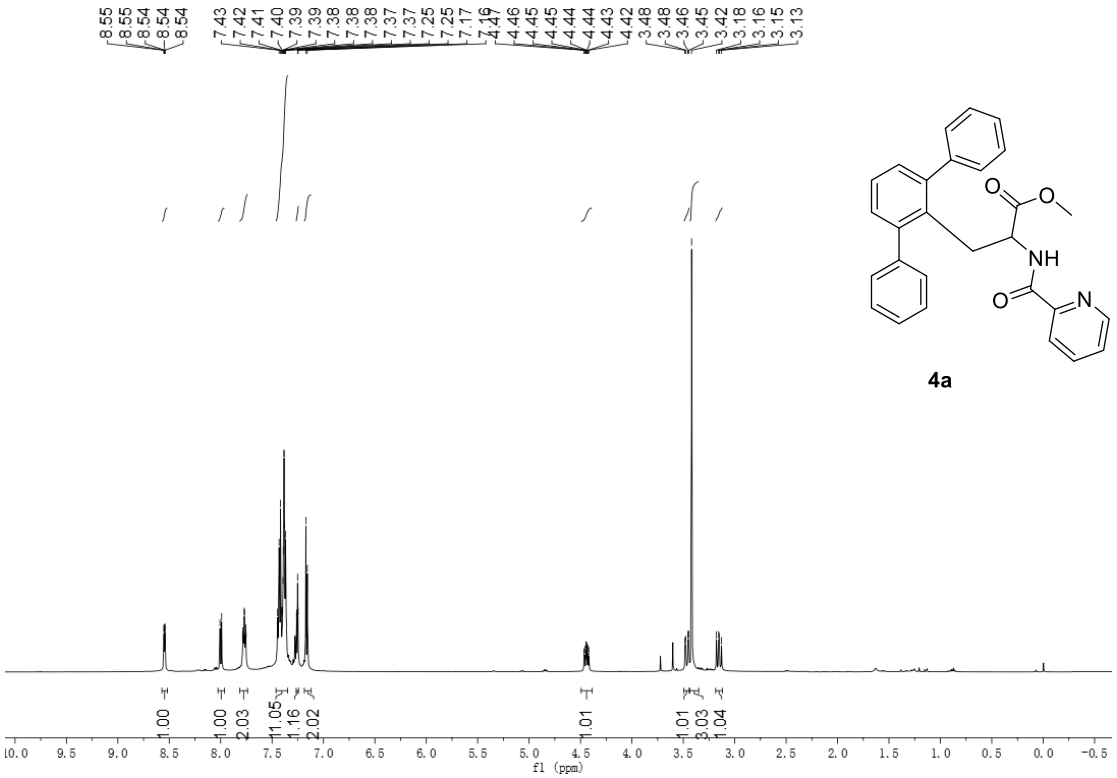
3t


7.69
7.67
7.53
7.51
7.49
7.48
7.46
7.44
7.43
7.40
7.39
7.39
7.38
7.38
7.37
7.35
7.34
7.34
7.33
6.97
6.12
6.10
5.09
3.65
O
N
O
NH
OBn
2.03
1.02
1.15
8.08
4.01
4.14
2.21
4.12
2.32
2.02
1.00
2.07
3.11
f1 (ppm)

3u


171.72
162.91
157.41
149.50
148.03
144.26
140.85
140.51
140.10
137.07
136.74
129.95
128.86
128.66
128.12
127.64
127.44
127.23
126.95
126.39
126.09
122.17
116.62
70.16
52.80
52.72
O
N
O
NH
OBn
f1 (ppm)

3u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8.55
8.54
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8.54
7.43
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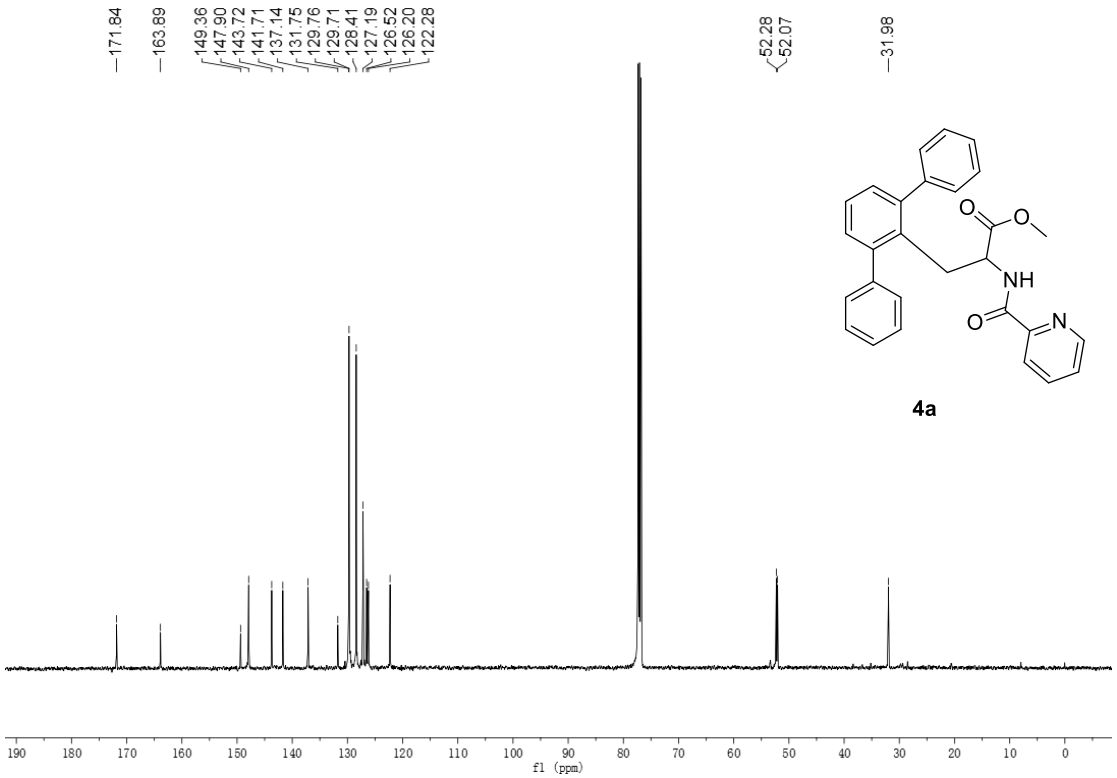
7.41
7.40
7.39
7.39
7.38
7.38
7.38
7.37
7.37
7.25
7.25
7.17
7.16
4.47
4.46
4.45
4.45
4.44
4.44
4.43
4.42
3.48
3.48
3.46
3.45
3.42
3.18
3.16
3.15
3.13
1.00
1.00
2.03
11.05
1.16
2.02
1.01
1.01
3.03
1.04
f1 (ppm)

O
O
NH
O
N

4a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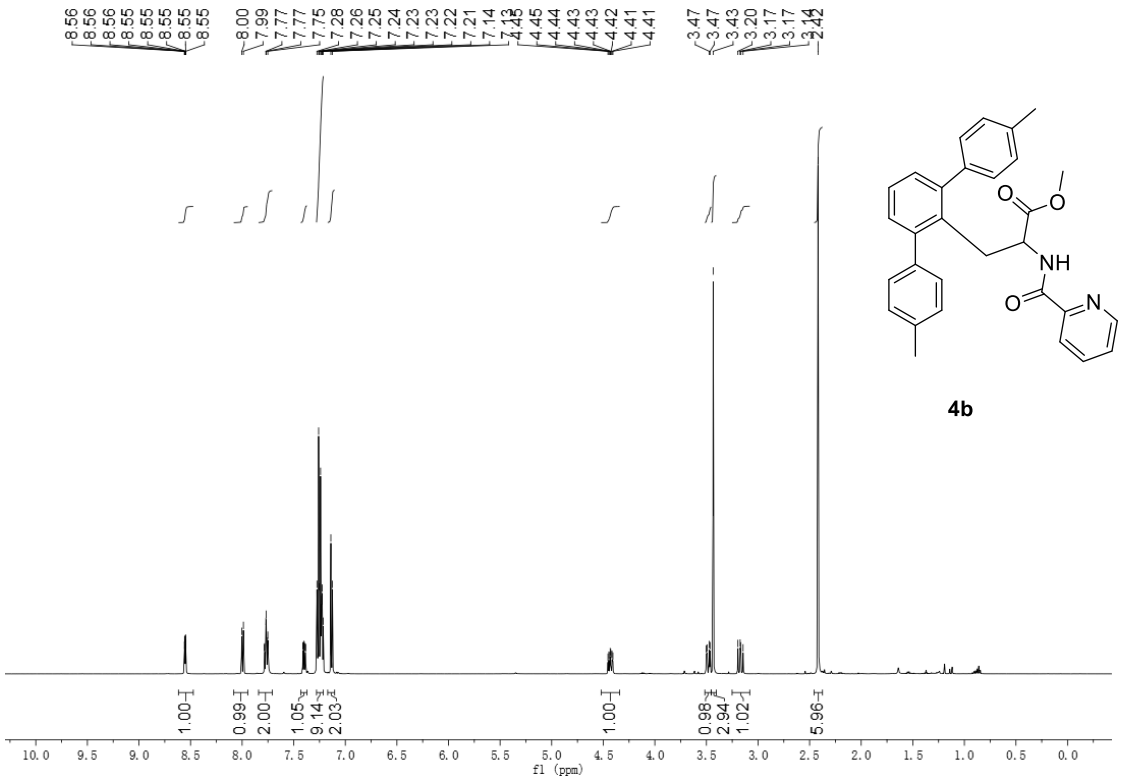
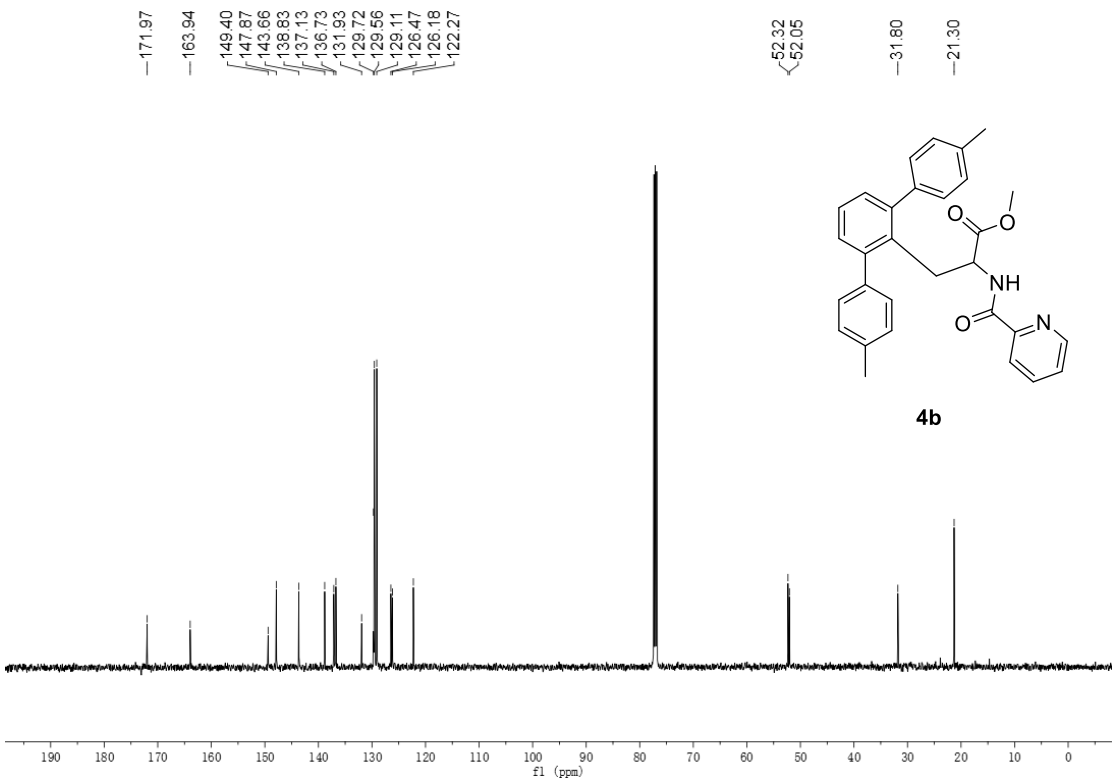
163.89
149.36
147.90
143.72
141.71
137.14
131.75
129.76
129.71
128.41
127.19
126.52
126.20
122.28
52.28
52.07
31.98
f1 (ppm)

O
O
NH
O
N

4a


8.56
8.56
8.56
8.55
8.55
8.55
8.55
8.00
7.99
7.77
7.77
7.75
7.28
7.26
7.25
7.24
7.23
7.23
7.22
7.21
7.14
4.45
4.44
4.43
4.43
4.42
4.41
4.41
3.47
3.47
3.43
3.20
3.17
3.17
2.42
1.00
0.99
2.00
1.05
9.14
2.03
1.00
0.98
2.94
1.02
5.96
f1 (ppm)
O
O
NH
O
N

4b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163.94
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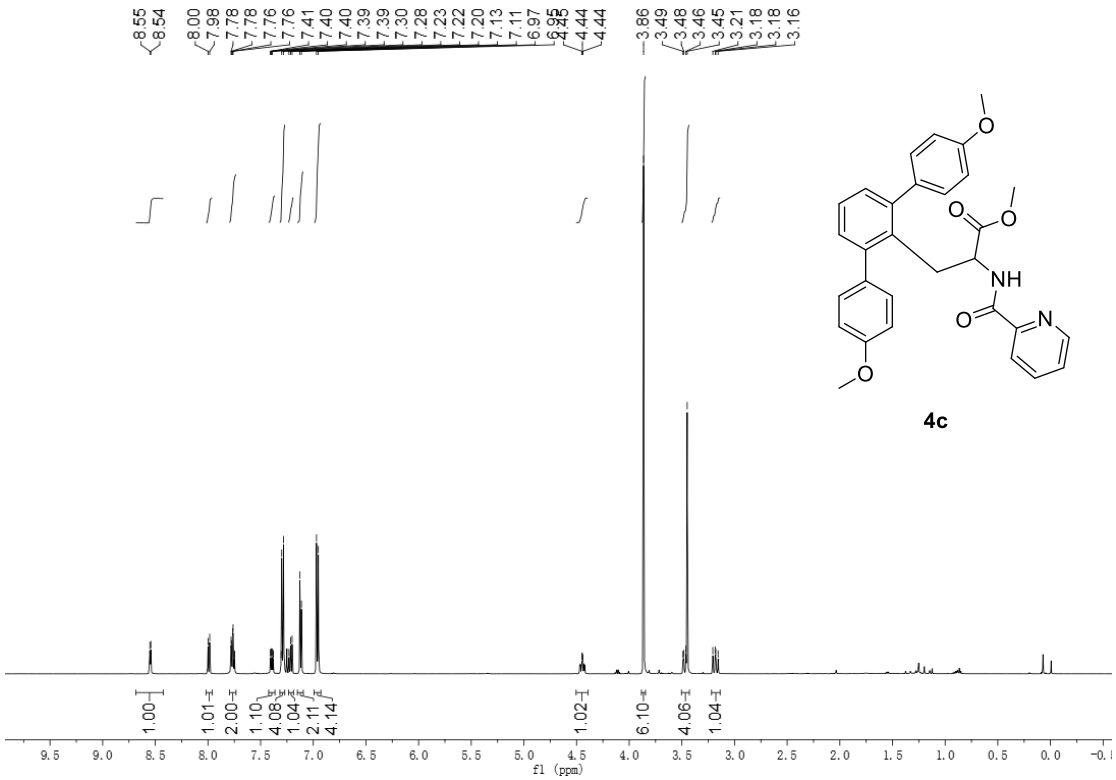
147.87
143.66
138.83
137.13
136.73
131.93
129.72
129.56
129.11
126.47
126.18
122.27
52.32
52.05
31.80
21.30
f1 (ppm)
O
O
NH
O
N

4b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8.54
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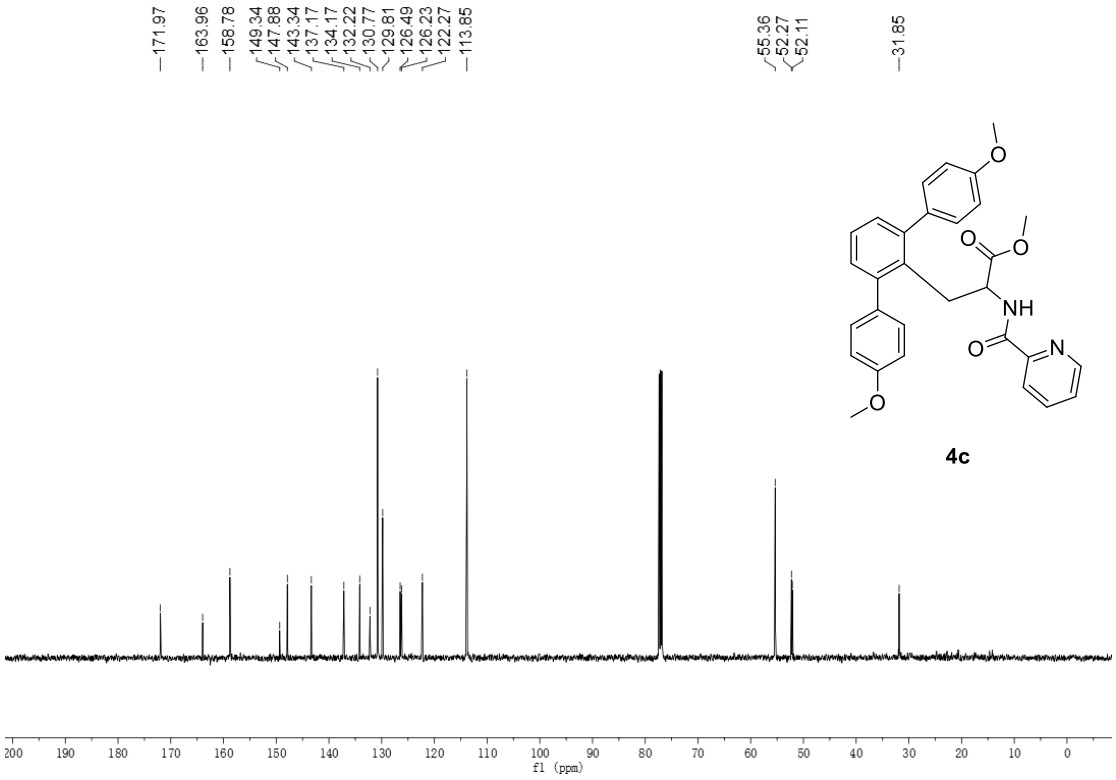
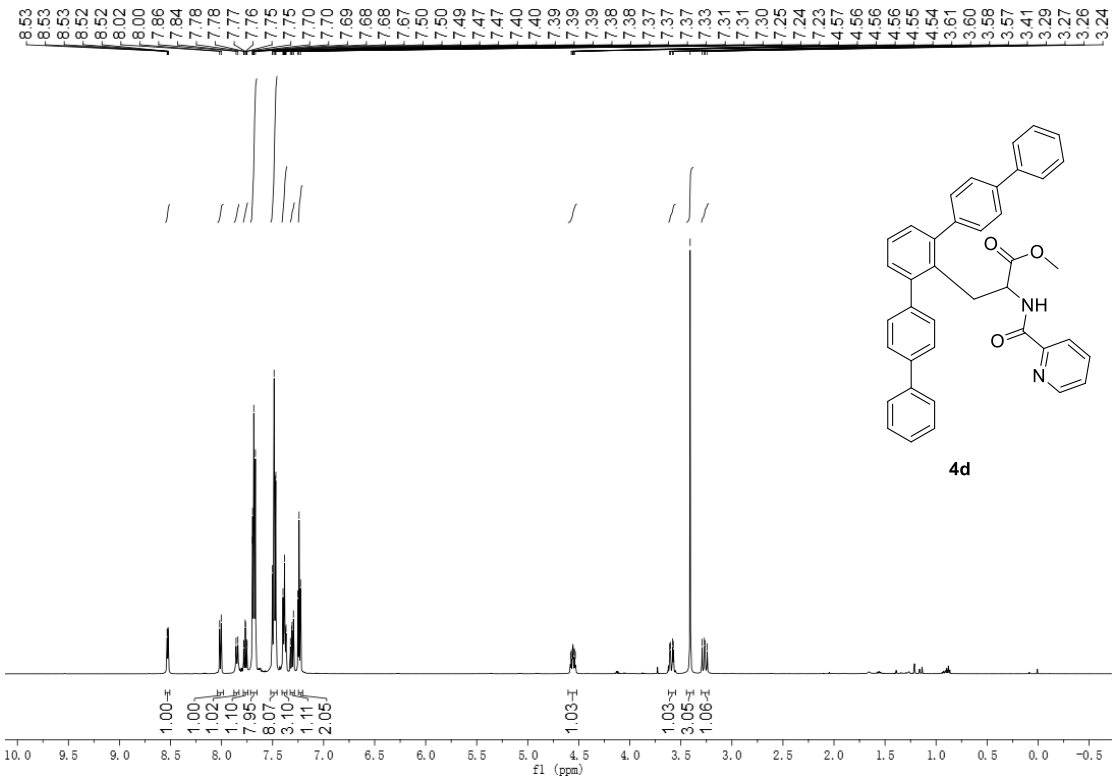
7.98
7.78
7.78
7.76
7.76
7.41
7.40
7.40
7.39
7.39
7.30
7.28
7.23
7.22
7.20
7.13
7.11
6.97
4.44
4.44
3.86
3.49
3.48
3.46
3.45
3.21
3.18
3.18
3.16
1.00
1.01
2.00
1.10
4.08
1.04
2.11
4.14
1.02
6.10
4.06
1.04
f1 (ppm)

4c

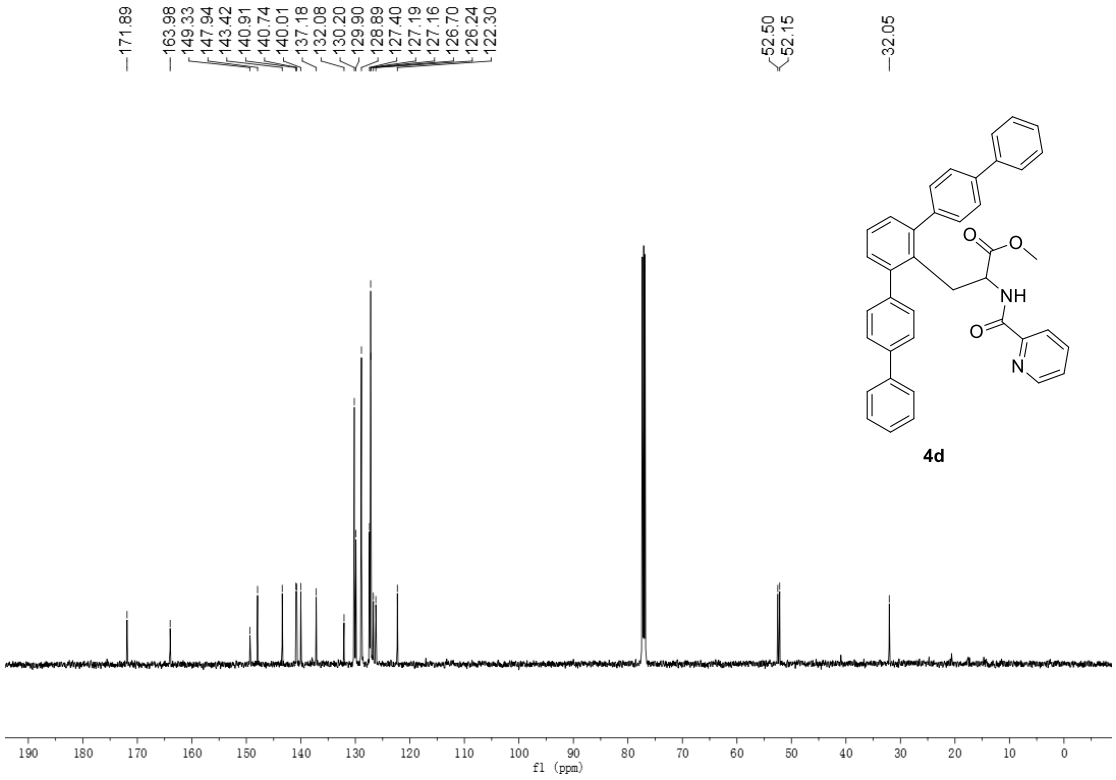

171.97
163.96
158.78
149.34
147.88
143.34
137.17
134.17
132.22
130.77
129.81
126.49
126.23
122.27
113.85
55.36
52.27
52.11
31.85
f1 (ppm)

4c


8.53 8.53 8.53 8.52 8.52 8.02 8.00 7.86 7.84 7.78 7.78 7.77 7.76 7.75 7.75 7.70 7.70 7.69 7.68 7.68 7.67 7.50 7.50 7.49 7.47 7.47 7.40 7.40 7.39 7.39 7.39 7.38 7.38 7.37 7.37 7.37 7.33 7.31 7.31 7.30 7.25 7.24 7.23 4.57 4.56 4.56 4.56 4.55 4.54 3.61 3.60 3.58 3.57 3.41 3.29 3.27 3.26 3.24
1.00 1.00 1.02 1.10 7.95 8.07 3.10 1.11 2.05 1.03 1.03 3.05 1.06
f1 (ppm)

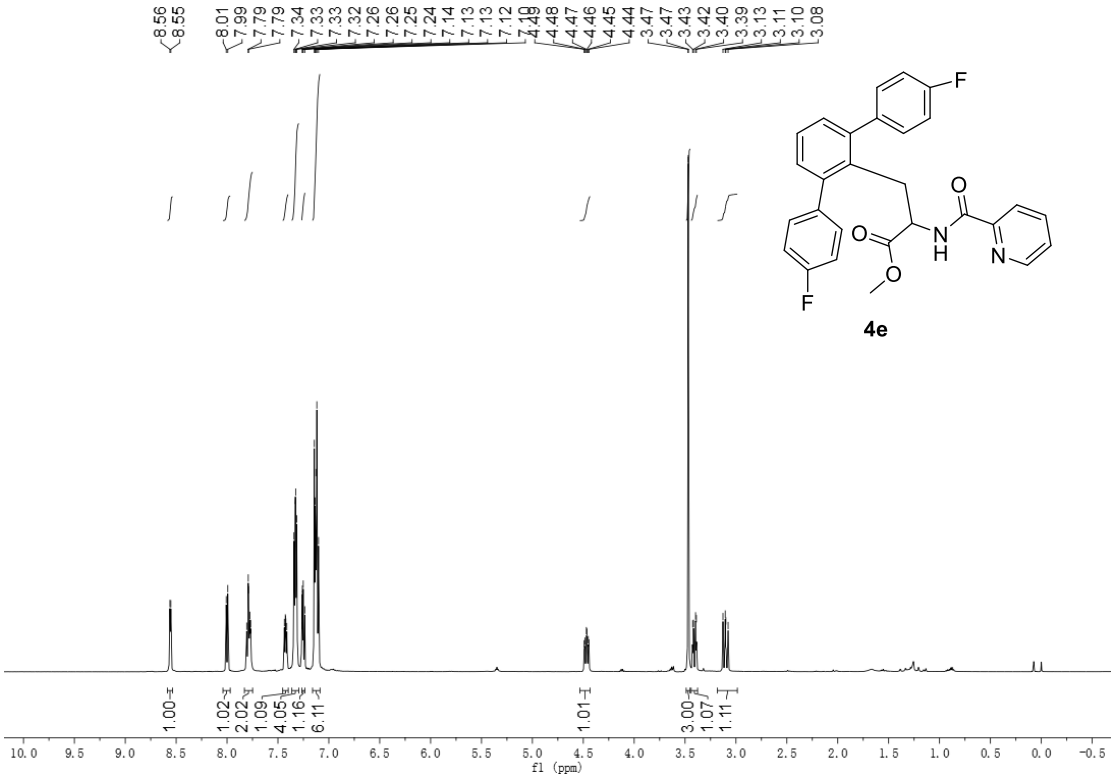
O
O
NH
O
N

4d


171.89
163.98 149.33 147.94 143.42 140.91 140.74 140.01 137.18 132.08 130.20 129.90 128.89 127.40 127.19 127.16 126.70 126.24 122.30
52.50 52.15
32.05
f1 (ppm)

O
O
NH
O
N

4d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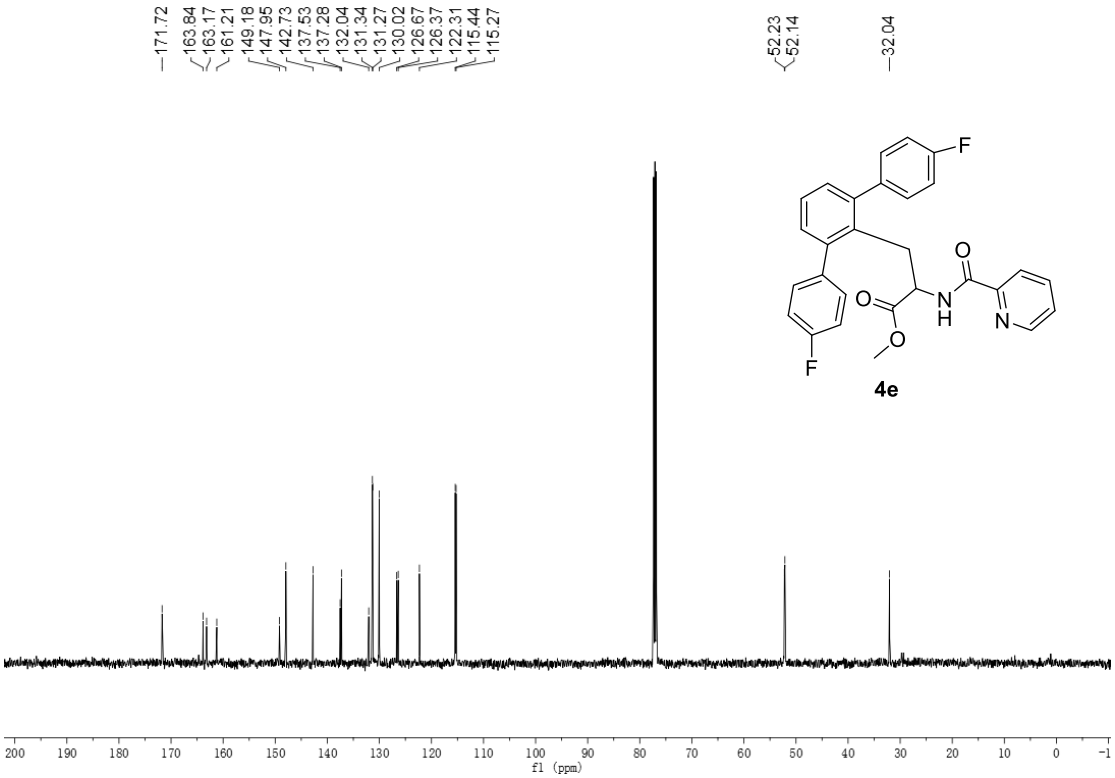
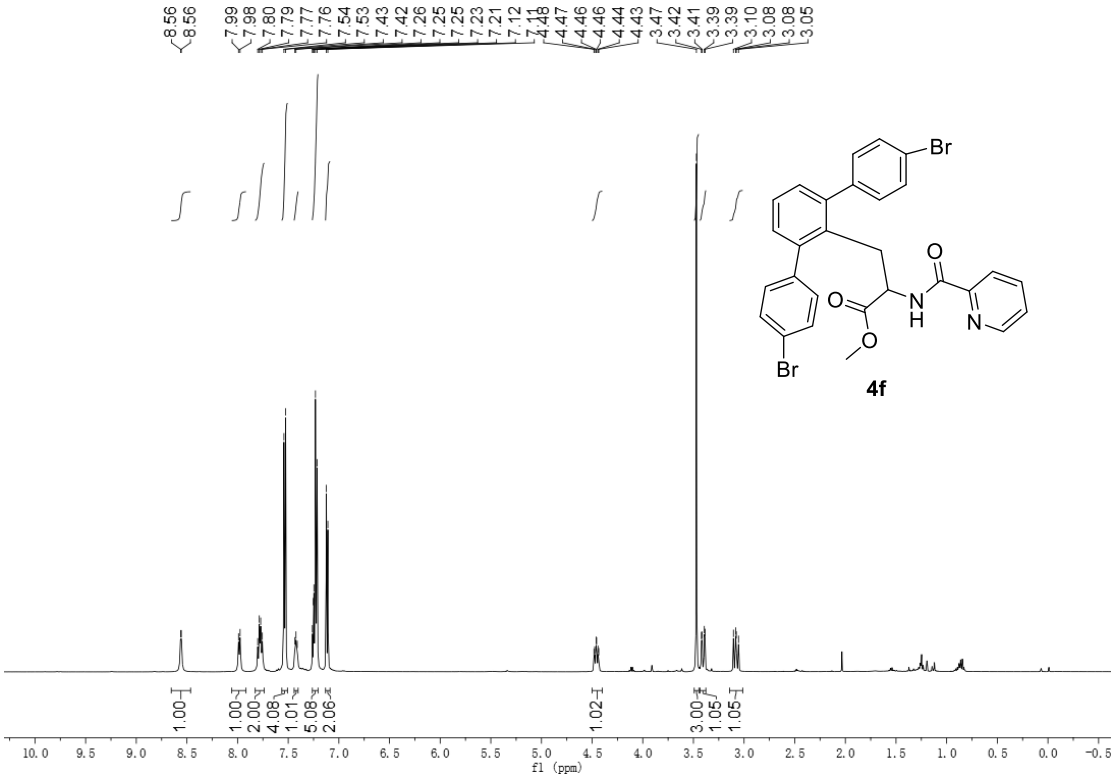
8.55
8.01
7.99
7.79
7.79
7.34
7.33
7.33
7.32
7.26
7.26
7.25
7.24
7.14
7.13
7.13
7.12
4.48
4.47
4.46
4.45
4.44
3.47
3.47
3.43
3.42
3.40
3.39
3.13
3.11
3.10
3.08
1.00
1.02
2.02
1.09
4.05
1.16
6.11
1.01
3.00
1.07
1.11
F
O
O
N
H
N
O
F
4e
f1 (ppm)


171.72
163.84
163.17
161.21
149.18
147.95
142.73
137.53
137.28
132.04
131.34
131.27
130.02
126.67
126.37
122.31
115.44
115.27
52.23
52.14
32.04
F
O
O
N
H
N
O
F
4e
f1 (ppm)


8.56
8.56
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7.98
7.80
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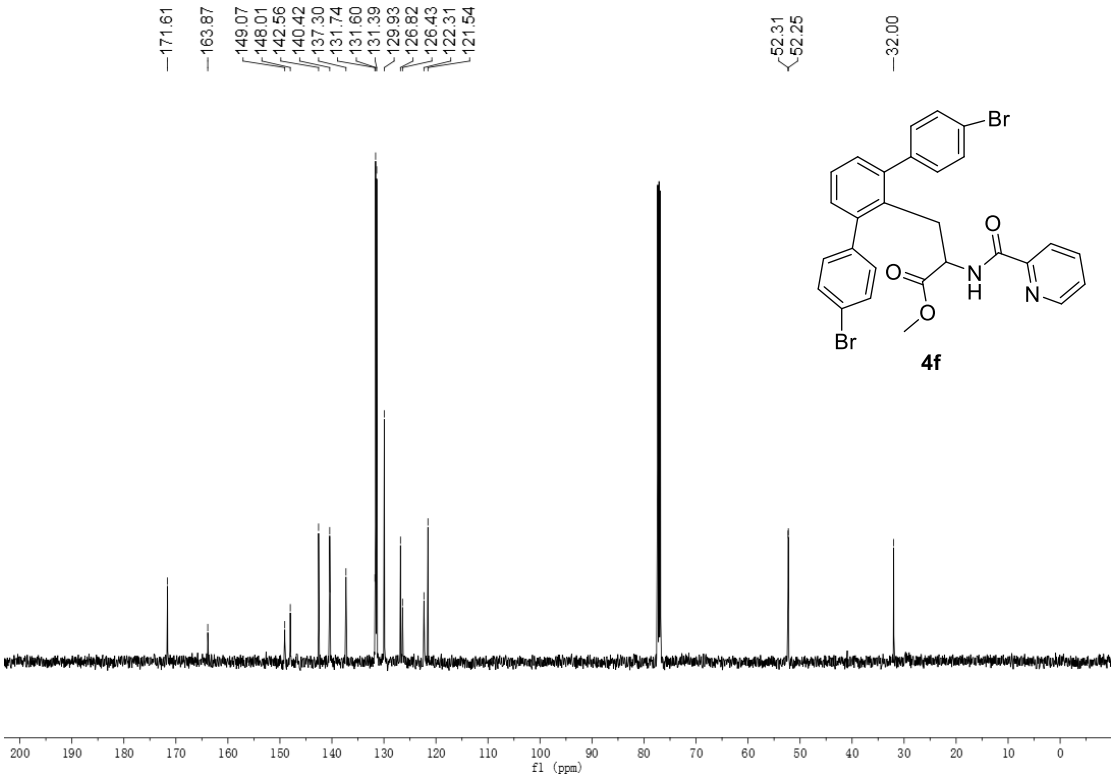
7.53
7.43
7.42
7.26
7.25
7.25
7.23
7.21
7.12
7.11
4.48
4.47
4.46
4.46
4.44
4.43
3.47
3.42
3.41
3.39
3.39
3.10
3.08
3.08
3.05
Br
O
O
N
H
N
O
Br
4f
1.00
1.00
2.00
4.08
1.01
5.08
2.06
1.02
3.00
1.05
1.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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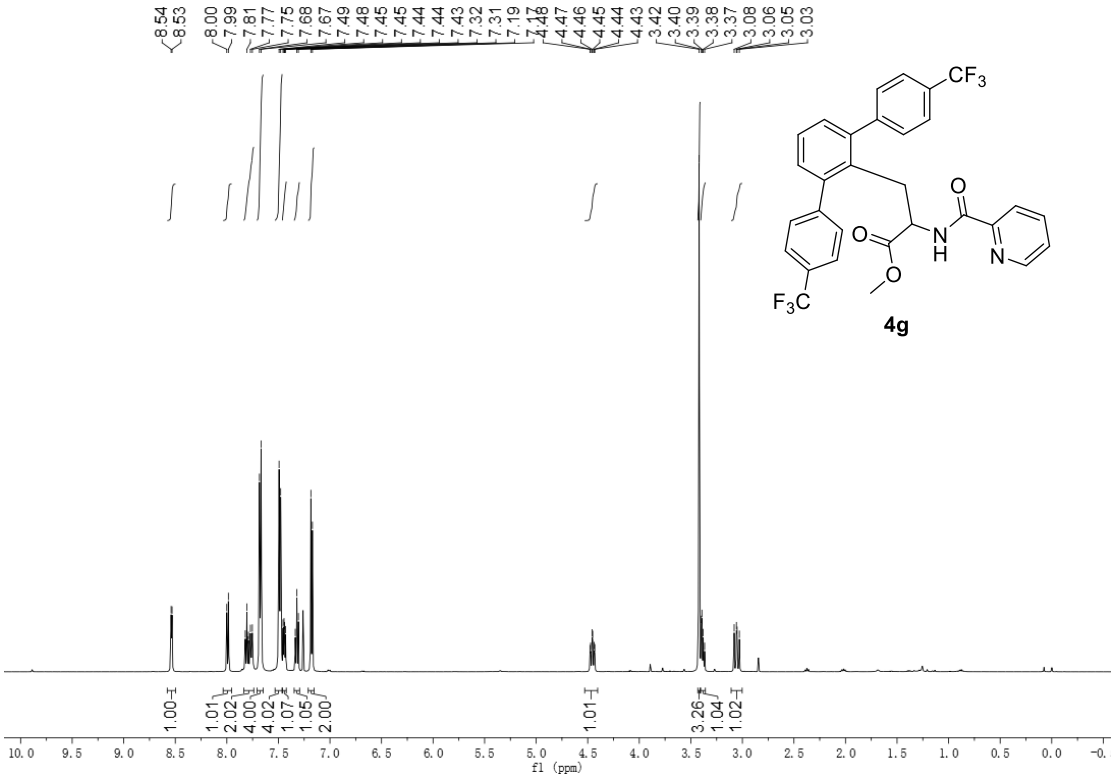
163.87
149.07
148.01
142.56
140.42
137.30
131.74
131.60
131.39
129.93
126.82
126.43
122.31
121.54
52.31
52.25
32.00
Br
O
O
N
H
N
O
Br
4f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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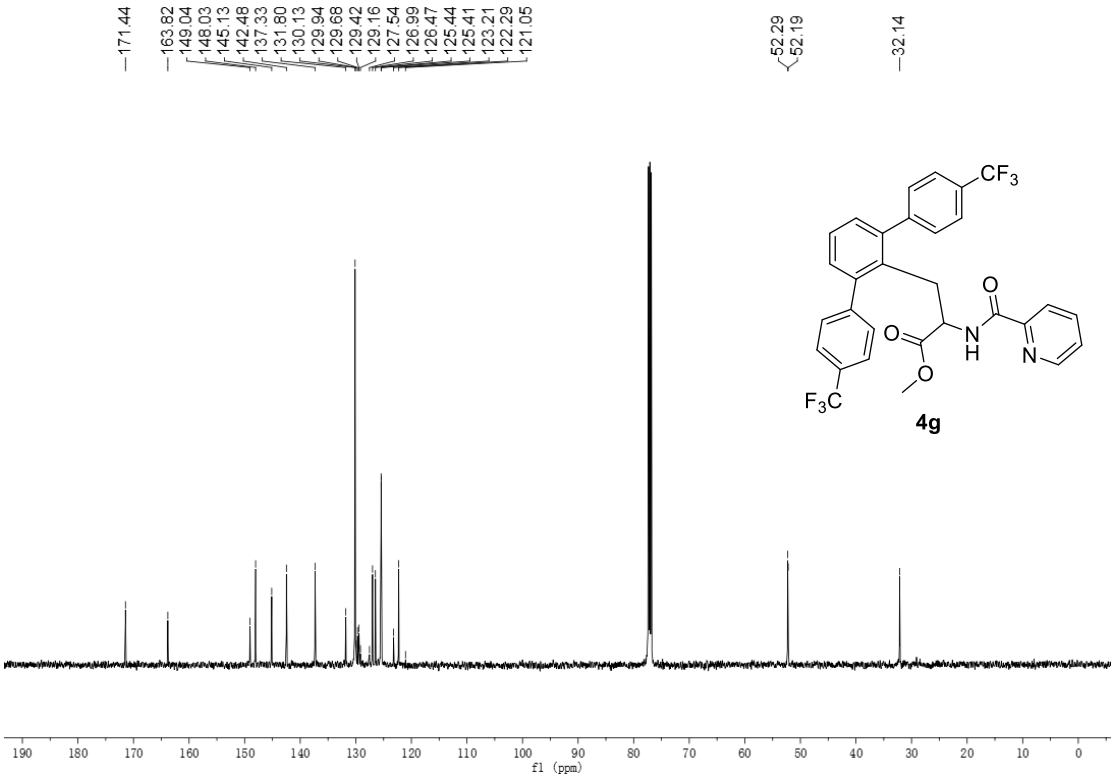
8.53
8.00
7.99
7.81
7.77
7.75
7.68
7.67
7.49
7.48
7.45
7.45
7.44
7.44
7.43
7.32
7.31
7.19
7.18
4.47
4.46
4.45
4.44
4.43
3.42
3.40
3.39
3.38
3.37
3.08
3.06
3.05
3.03
1.00
1.01
2.02
4.00
4.02
1.07
1.05
2.00
1.01
3.26
1.04
1.02
f1 (ppm)
CF3
O
N
H
O
O
N
F3C
4g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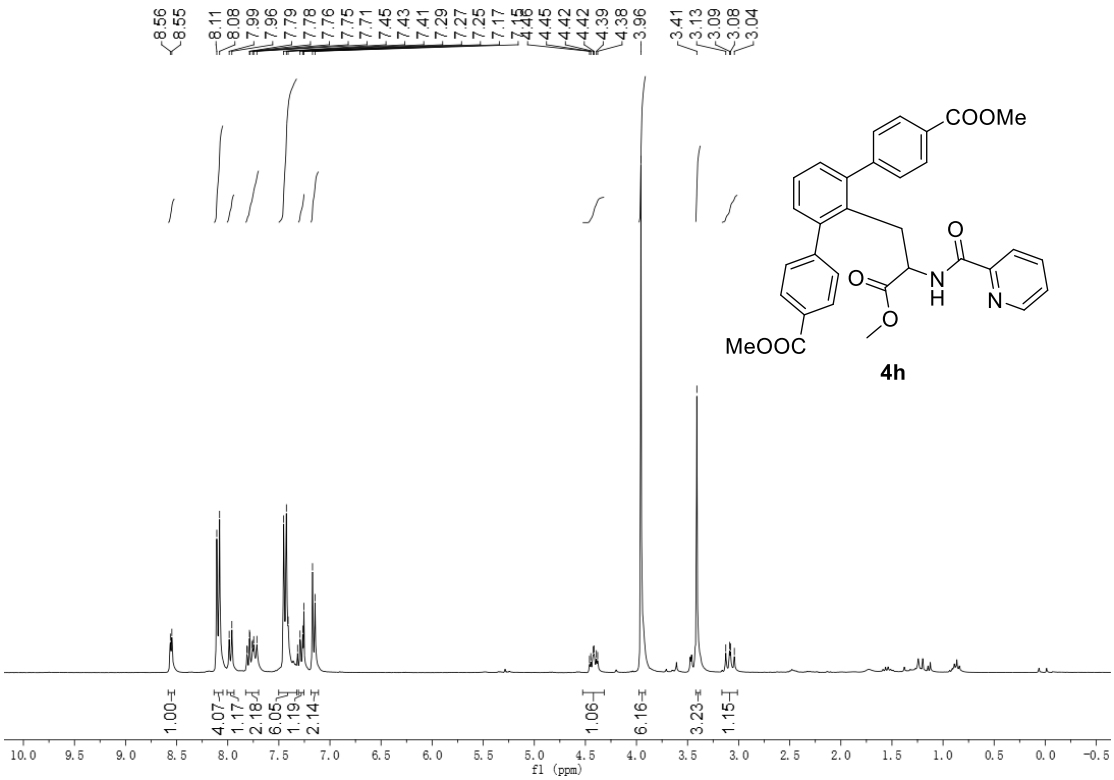
163.82
149.04
148.03
145.13
142.48
137.33
131.80
130.13
129.94
129.68
129.42
129.16
127.54
126.99
126.47
125.44
125.41
123.21
122.29
121.05
52.29
52.19
32.14
f1 (ppm)
CF3
O
N
H
O
O
N
F3C
4g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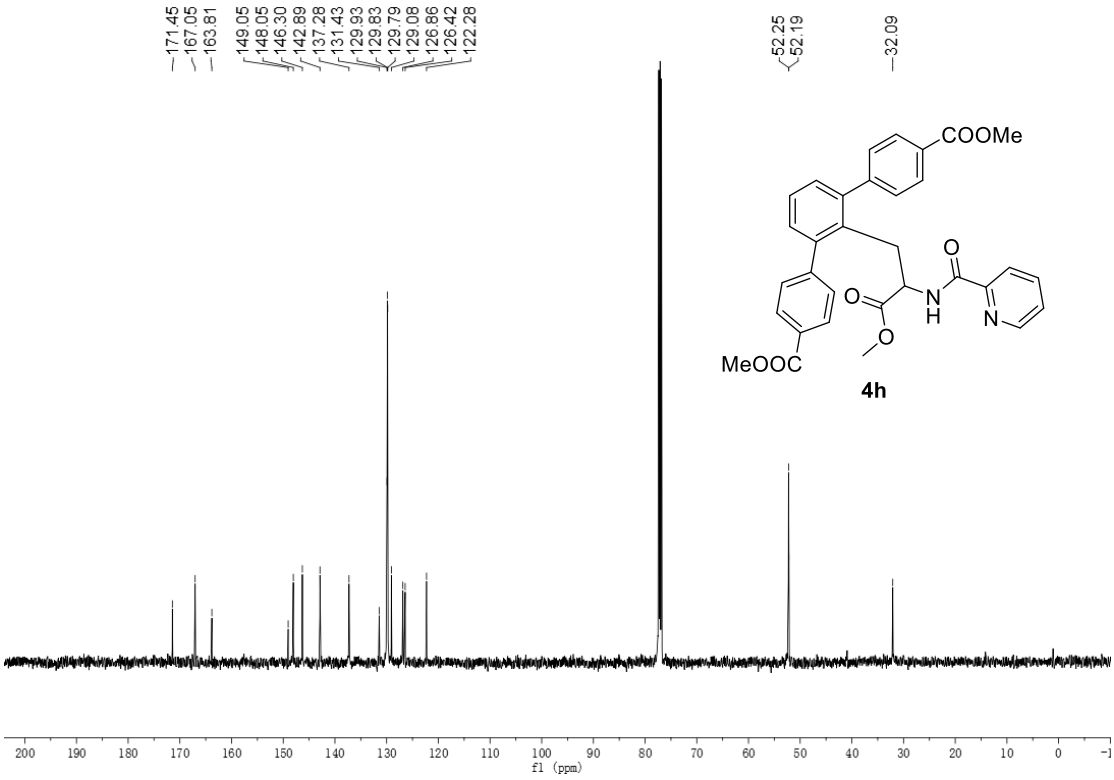
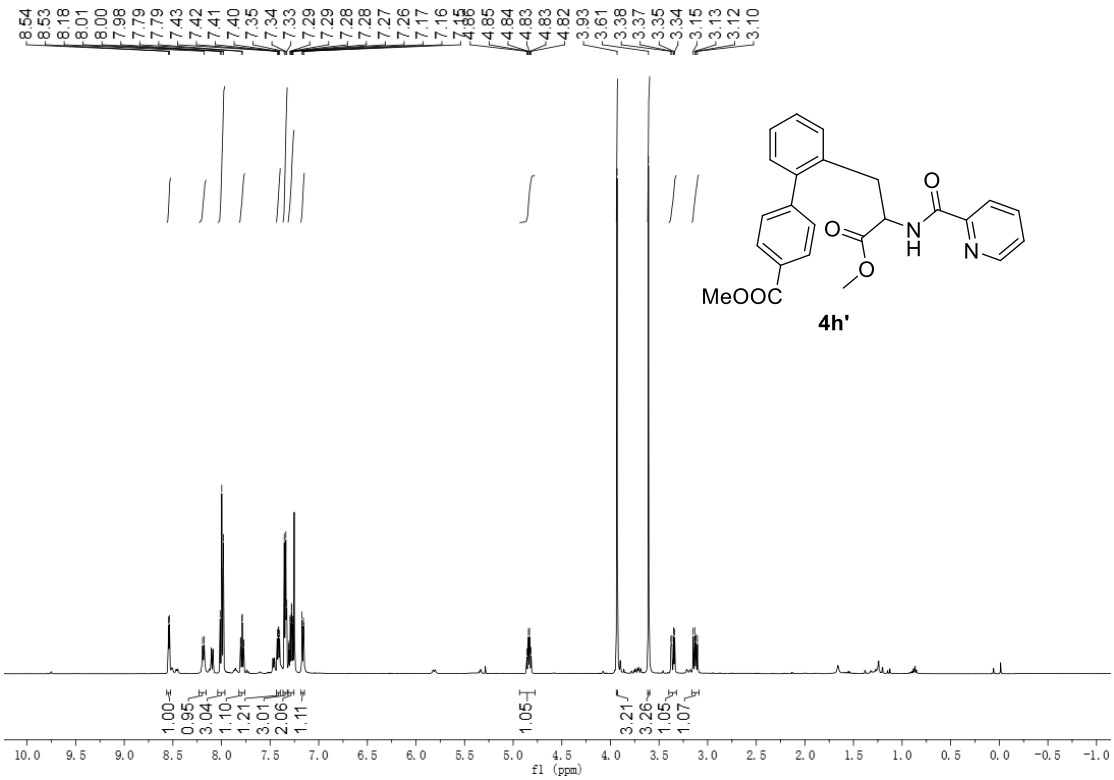
8.55
8.11
8.08
7.99
7.96
7.79
7.78
7.76
7.75
7.71
7.45
7.43
7.41
7.29
7.27
7.25
7.17
4.46
4.45
4.42
4.42
4.39
4.38
3.96
3.41
3.13
3.09
3.08
3.04
1.00
4.07
1.17
2.18
6.05
1.19
2.14
1.06
6.16
3.23
1.15
f1 (ppm)
COOMe
MeOOC
4h


171.45
167.05
163.81
149.05
148.05
146.30
142.89
137.28
131.43
129.93
129.83
129.79
129.08
126.86
126.42
122.28
52.25
52.19
32.09
f1 (ppm)
COOMe
MeOOC
4h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8.53
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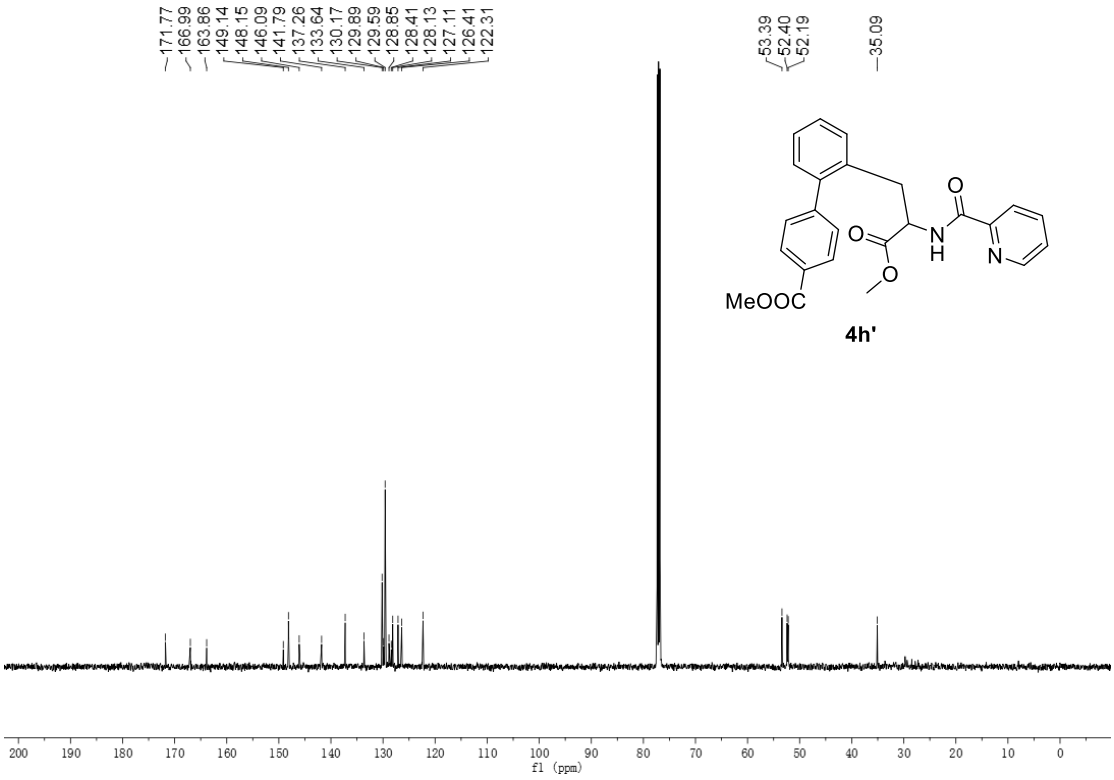
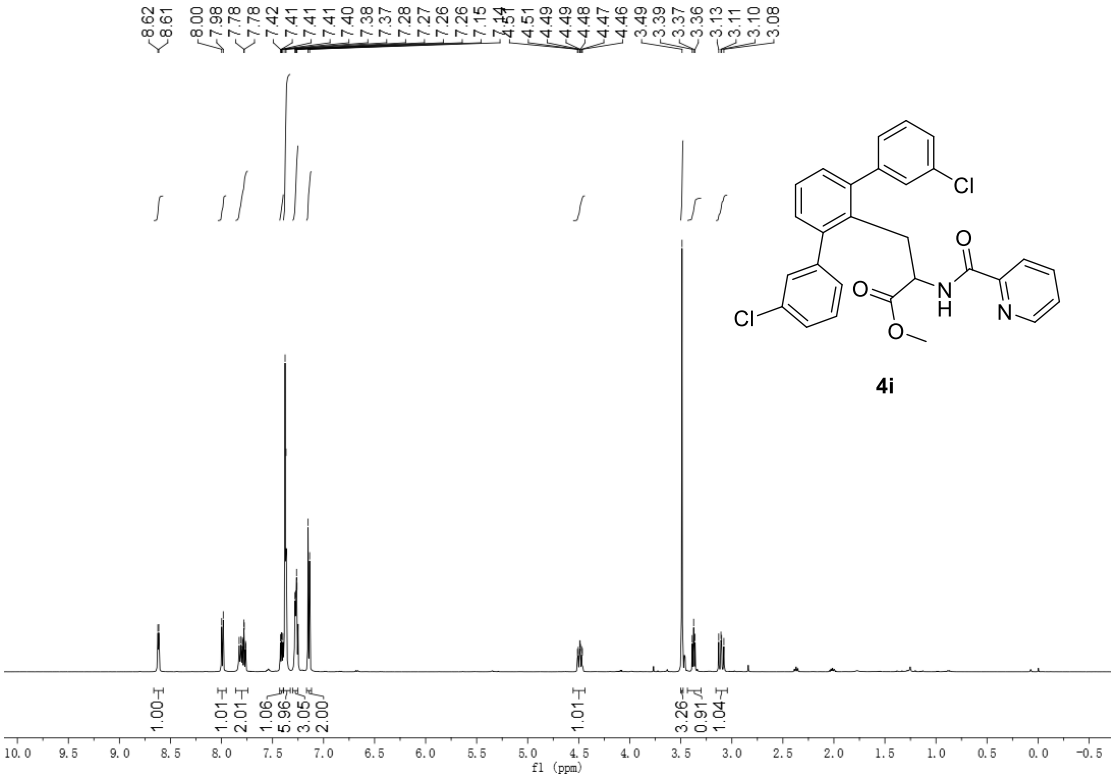
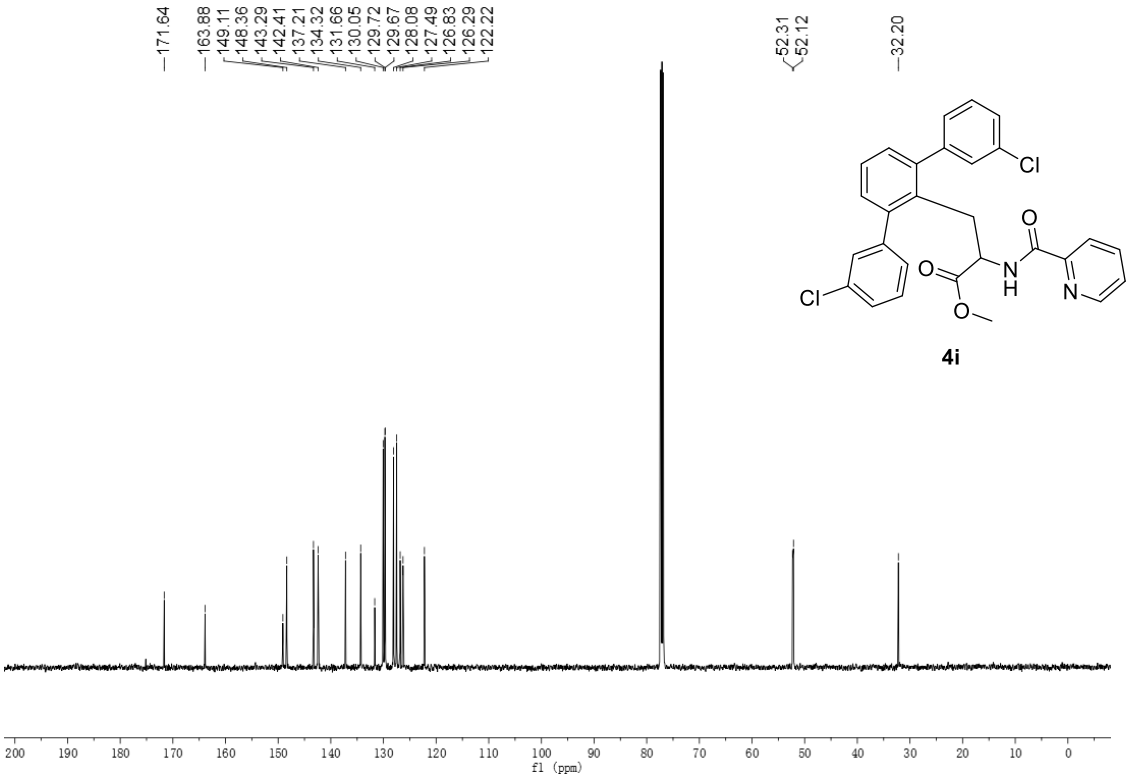
8.00
7.98
7.79
7.79
7.43
7.42
7.41
7.40
7.35
7.34
7.33
7.29
7.29
7.28
7.28
7.27
7.26
7.17
7.16
7.15
4.86
4.85
4.84
4.83
4.83
4.82
3.93
3.61
3.38
3.37
3.35
3.34
3.15
3.13
3.12
3.10
1.00
0.95
3.04
1.10
1.21
3.01
2.06
1.11
1.05
3.21
3.26
1.05
1.07
f1 (ppm)
MeOOC
4h'

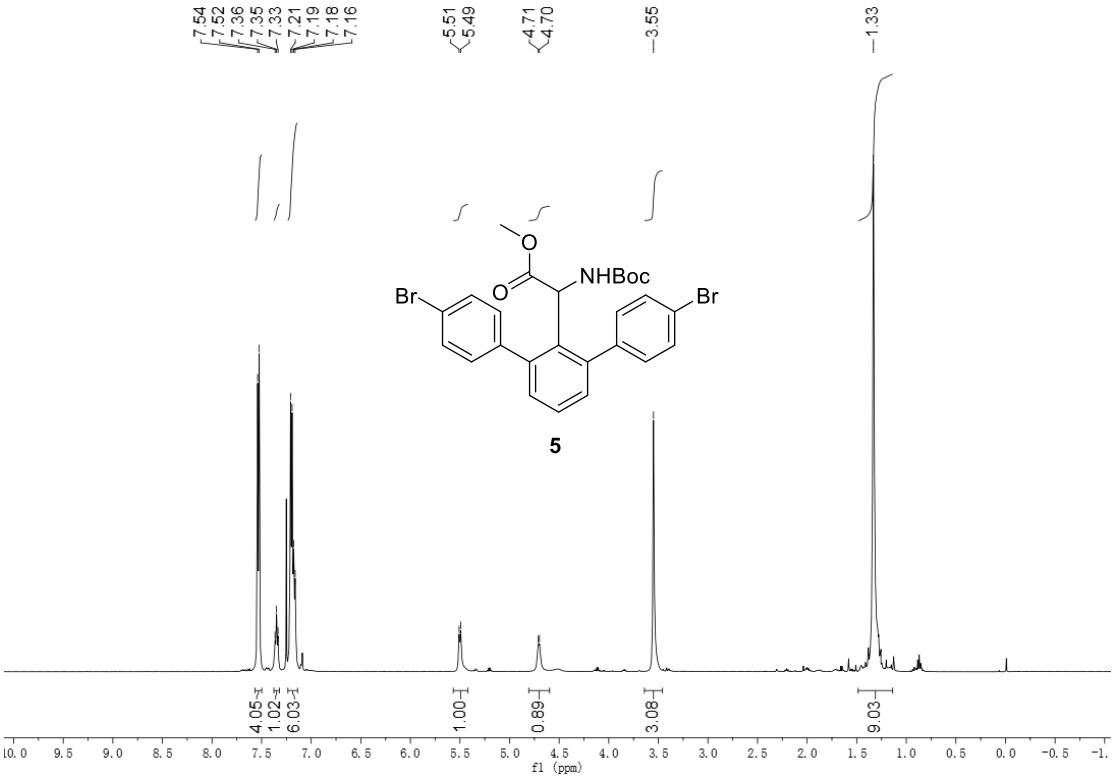

171.77
166.99
163.86
149.14
148.15
146.09
141.79
137.26
133.64
130.17
129.89
129.59
128.85
128.41
128.13
127.11
126.41
122.31
53.39
52.40
52.19
35.09
f1 (ppm)
MeOOC
4h'


8.62
8.61
8.00
7.98
7.78
7.78
7.42
7.41
7.41
7.41
7.40
7.38
7.37
7.28
7.27
7.26
7.26
7.15
4.51
4.51
4.49
4.49
4.48
4.47
4.46
3.49
3.39
3.37
3.36
3.13
3.11
3.10
3.08
Cl
Cl
O
O
N
H
N
O
1.00
1.01
2.01
1.06
5.96
3.05
2.00
1.01
3.26
0.91
1.04
f1 (ppm)

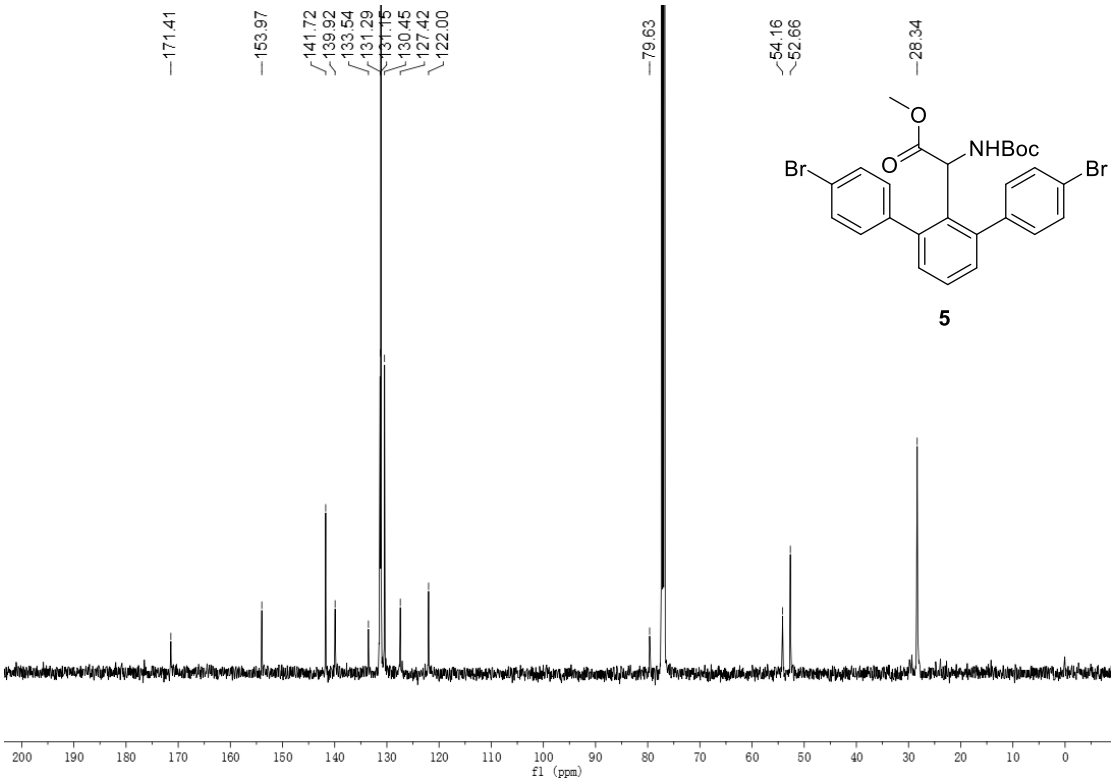
4i


171.64
163.88
149.11
148.36
143.29
142.41
137.21
134.32
131.66
130.05
129.72
129.67
128.08
127.49
126.83
126.29
122.22
52.31
52.12
32.20
Cl
Cl
O
O
N
H
N
O
f1 (ppm)

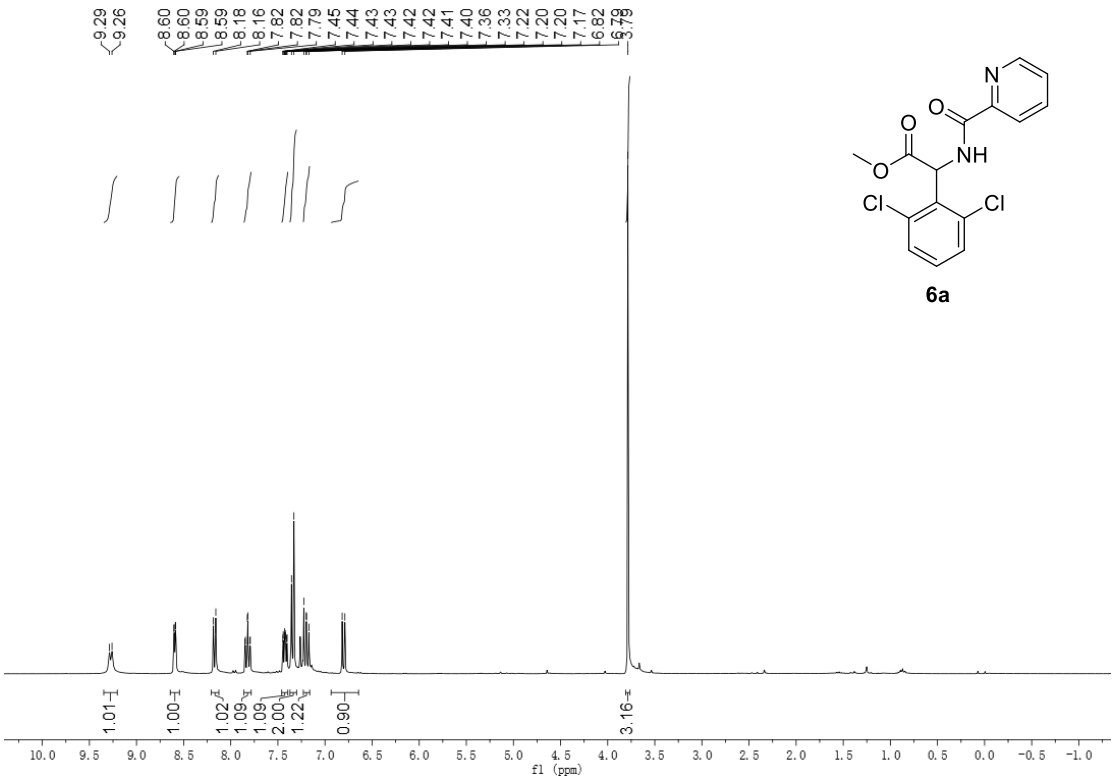
4i


7.54
7.52
7.36
7.35
7.33
7.21
7.19
7.18
7.16
5.51
5.49
4.71
4.70
3.55
1.33
O
O
NHBoc
Br
Br
4.05
1.02
6.03
1.00
0.89
3.08
9.03
f1 (ppm)

5

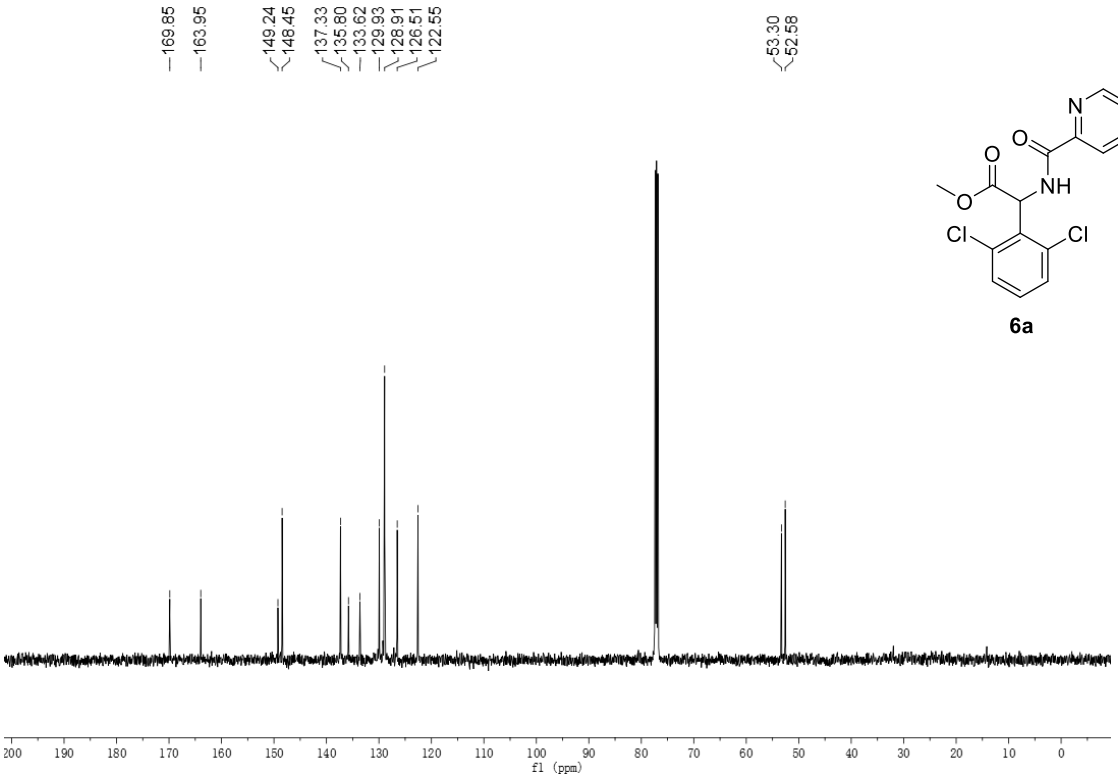

171.41
153.97
141.72
139.92
133.54
131.29
131.15
130.45
127.42
122.00
79.63
54.16
52.66
28.34
O
O
NHBoc
Br
Br
f1 (ppm)

5


9.29
9.26
8.60
8.60
8.59
8.59
8.18
8.16
7.82
7.82
7.79
7.45
7.44
7.43
7.43
7.42
7.42
7.41
7.40
7.36
7.33
7.22
7.20
7.20
7.17
6.82
6.79
6.78
1.01
1.00
1.02
1.09
1.09
2.00
1.22
0.90
3.16
f1 (ppm)

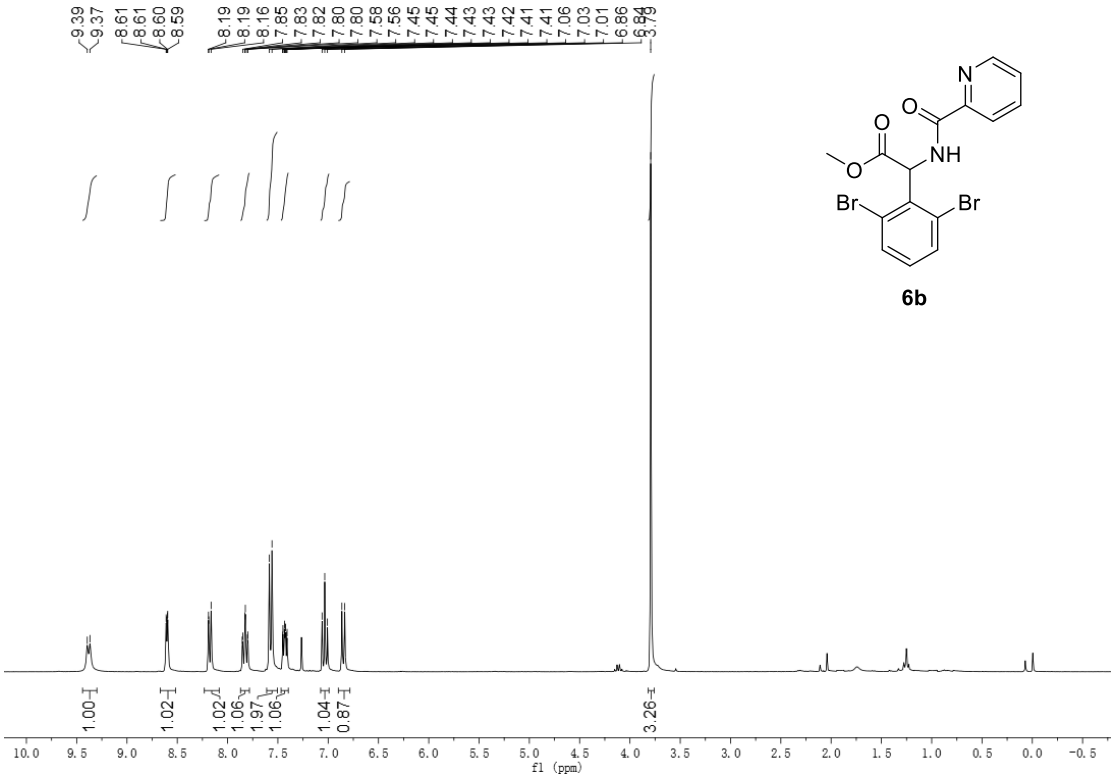
N
O
O
NH
O
Cl
Cl

6a


169.85
163.95
149.24
148.45
137.33
135.80
133.62
129.93
128.91
126.51
122.55
53.30
52.58
f1 (ppm)

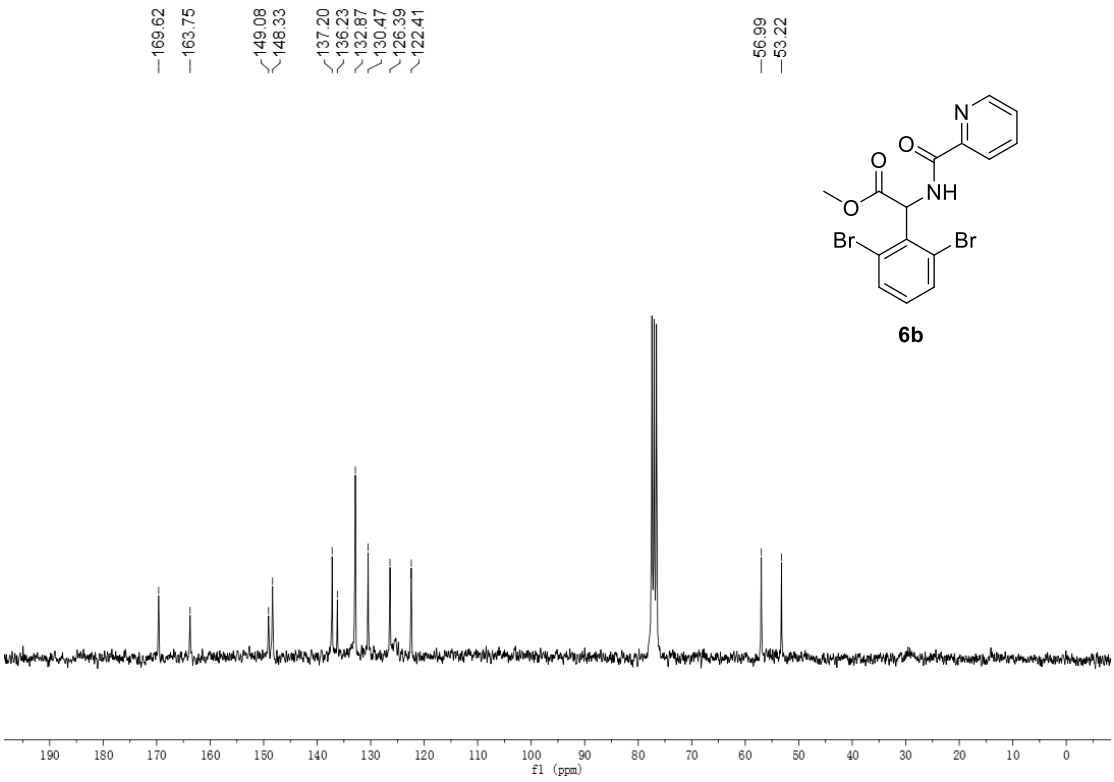
N
O
O
NH
O
Cl
Cl

6a


9.39
9.37
8.61
8.61
8.60
8.59
8.19
8.19
8.16
7.85
7.83
7.82
7.80
7.80
7.58
7.56
7.45
7.45
7.44
7.43
7.43
7.42
7.41
7.41
7.06
7.03
7.01
6.86
6.84
3.79
1.00
1.02
1.02
1.06
1.97
1.06
1.04
0.87
3.26
f1 (ppm)

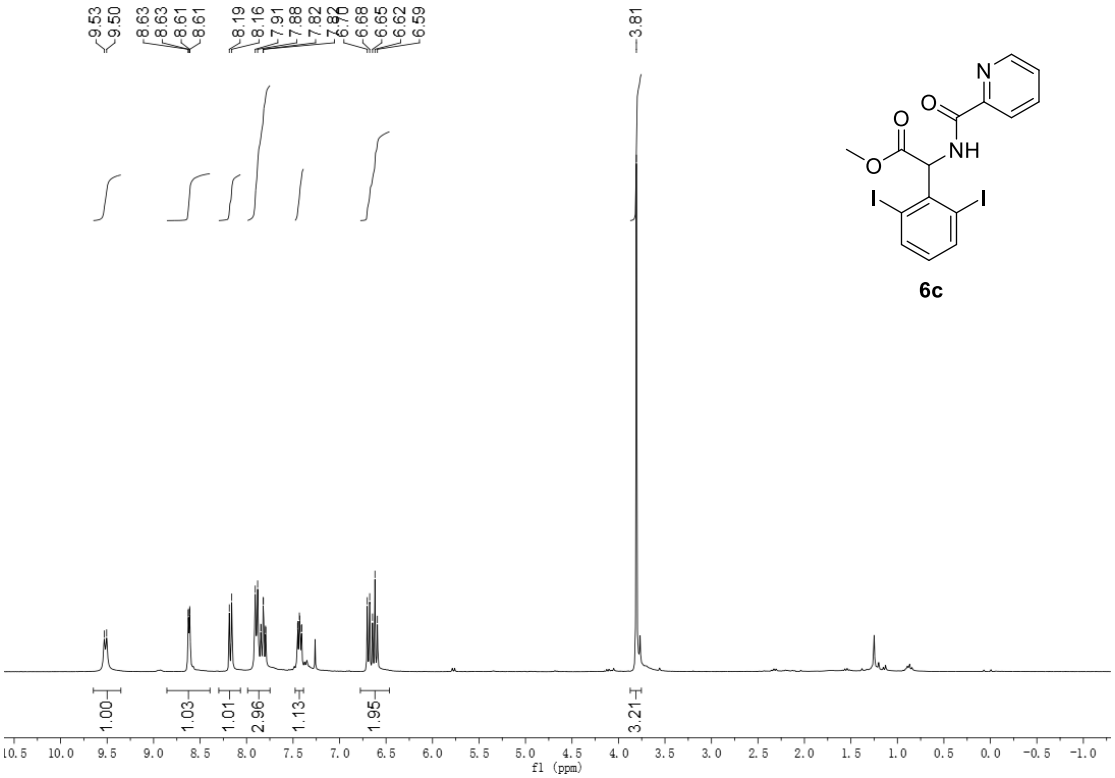
N
O
O
NH
O
Br
Br

6b

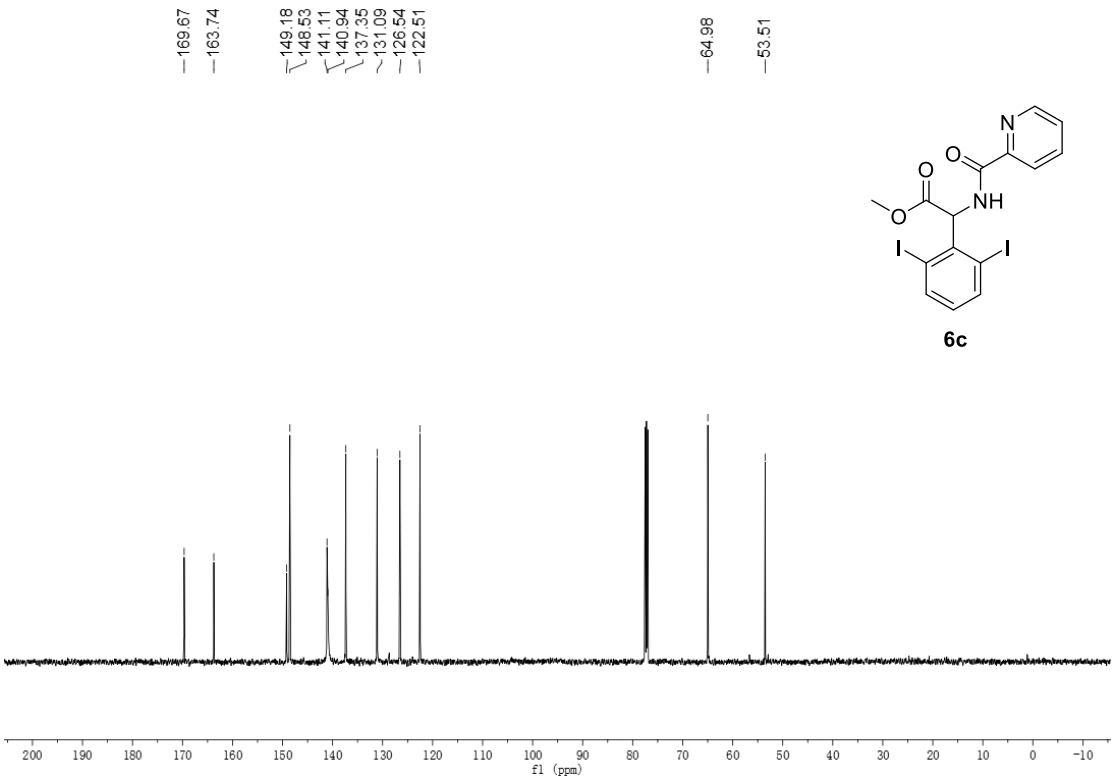

169.62
163.75
149.08
148.33
137.20
136.23
132.87
130.47
126.39
122.41
56.99
53.22
f1 (ppm)

N
O
O
NH
O
Br
Br

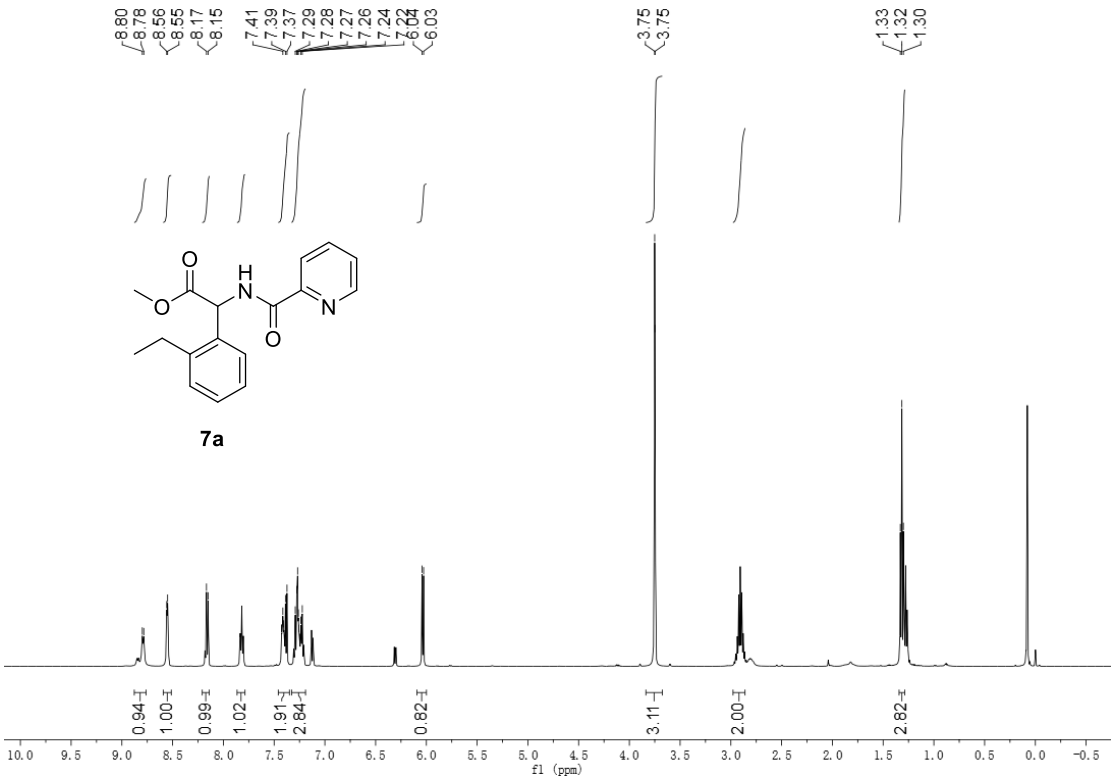
6b


9.53
9.50
8.63
8.63
8.61
8.61
8.19
8.16
7.91
7.88
7.82
7.82
6.70
6.68
6.65
6.62
6.59
3.81
1.00
1.03
1.01
2.96
1.13
1.95
3.21
f1 (ppm)

6c


169.67
163.74
149.18
148.53
141.11
140.94
137.35
131.09
126.54
122.51
64.98
53.51
f1 (ppm)

6c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8.78
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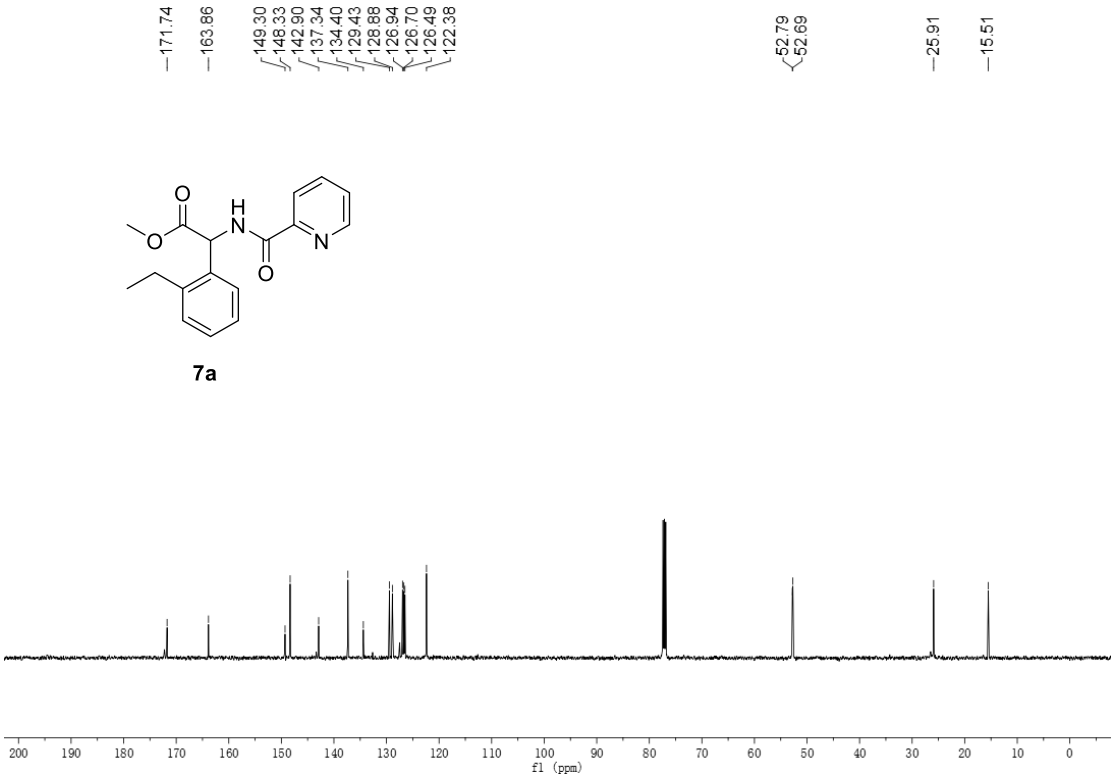
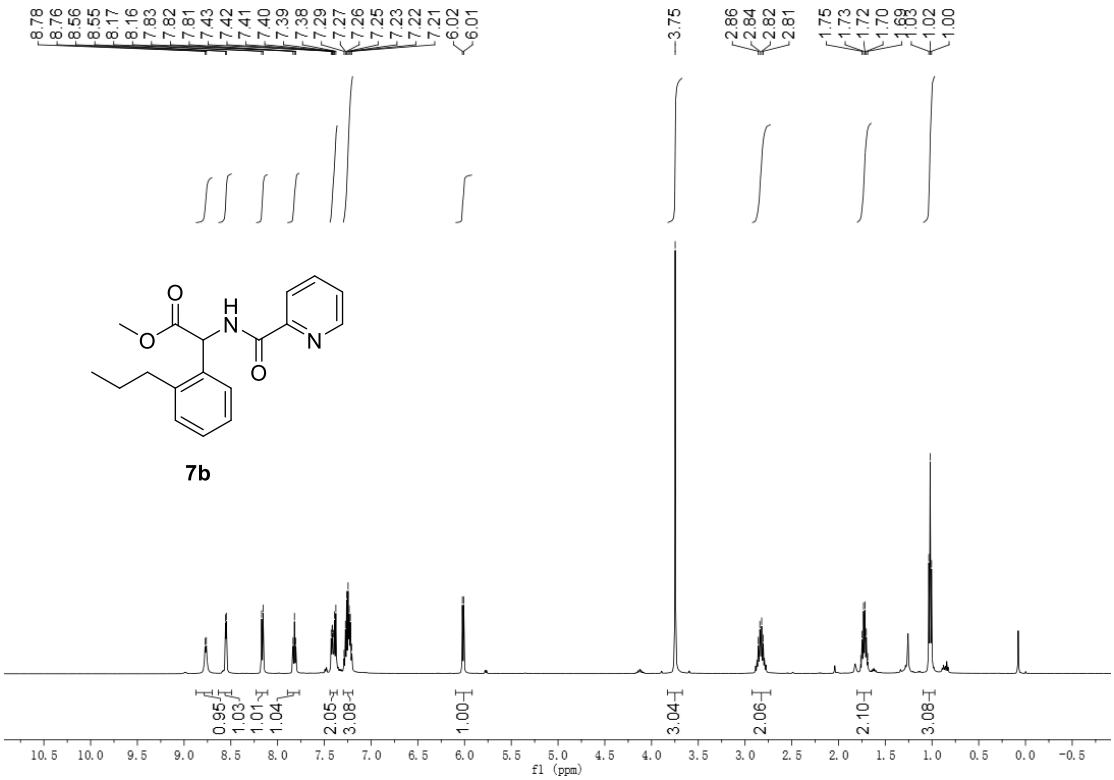
8.55
8.17
8.15
7.41
7.39
7.37
7.29
7.28
7.27
7.26
7.24
7.22
6.04
6.03
3.75
3.75
1.33
1.32
1.30
O
H
N
O
O
N
0.94
1.00
0.99
1.02
1.91
2.84
0.82
3.11
2.00
2.82
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

7a


171.74
163.86
149.30
148.33
142.90
137.34
134.40
129.43
128.88
126.94
126.70
126.49
122.38
52.79
52.69
25.91
15.51
O
H
N
O
O
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

7a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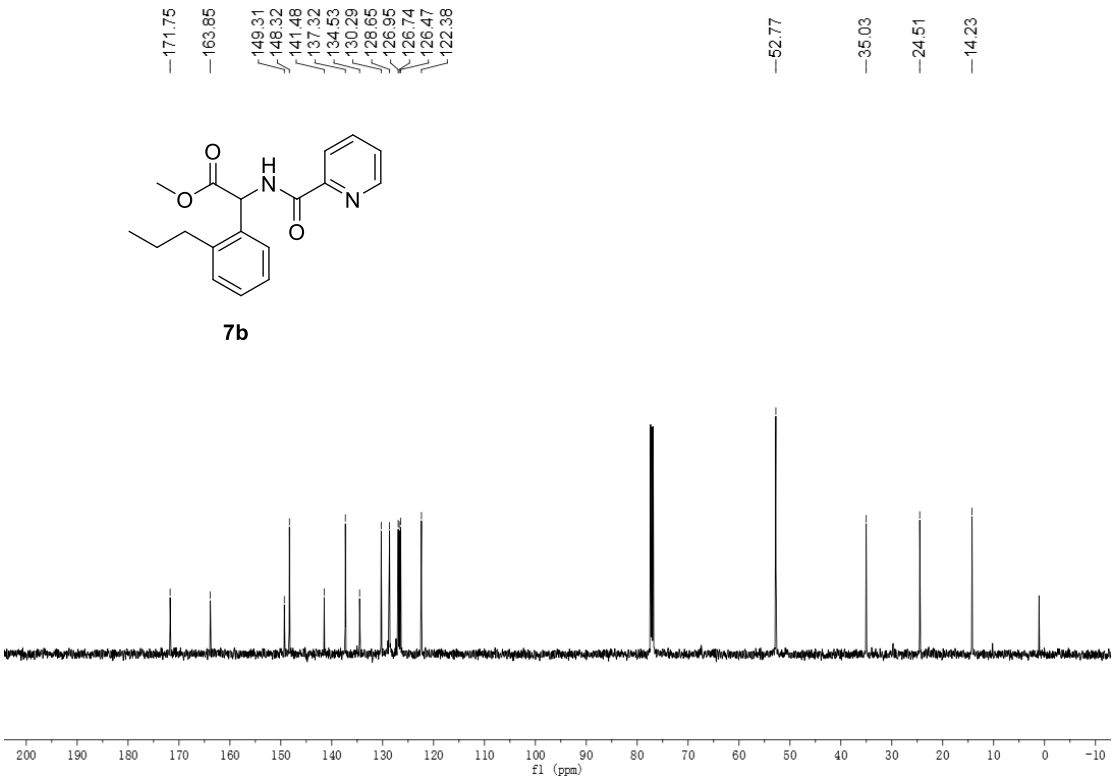
8.76
8.56
8.55
8.17
8.16
7.83
7.82
7.81
7.43
7.42
7.41
7.40
7.39
7.38
7.29
7.27
7.26
7.25
7.23
7.22
7.21
6.02
6.01
3.75
2.86
2.84
2.82
2.81
1.75
1.73
1.72
1.70
1.03
1.02
1.00
O
H
N
O
O
N
0.95
1.03
1.01
1.04
2.05
3.08
1.00
3.04
2.06
2.10
3.08
f1 (ppm)

7b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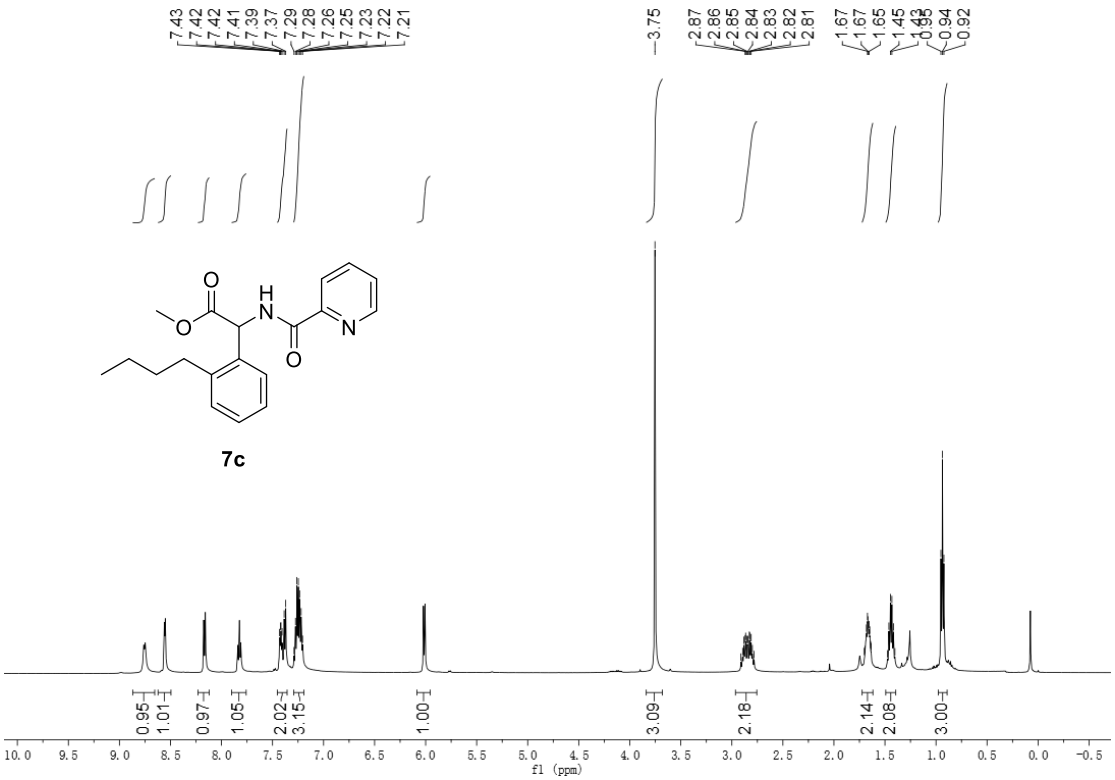
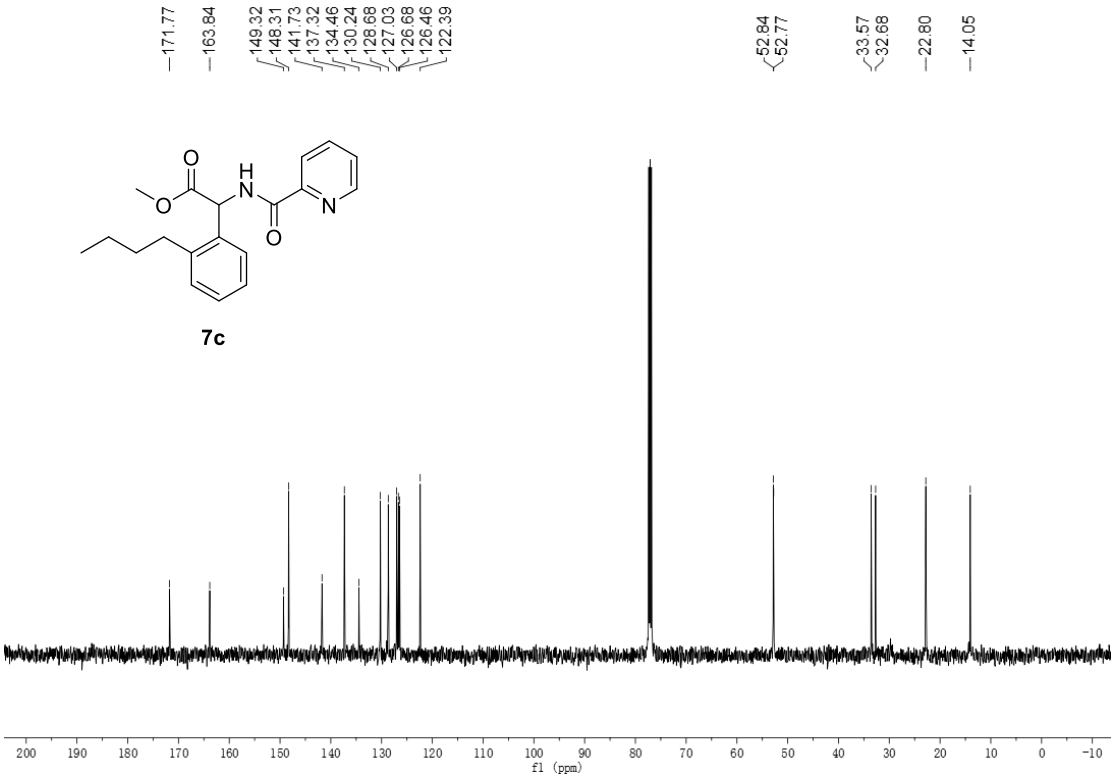
163.85
149.31
148.32
141.48
137.32
134.53
130.29
128.65
126.95
126.74
126.47
122.38
52.77
35.03
24.51
14.23
O
H
N
O
O
N
f1 (ppm)

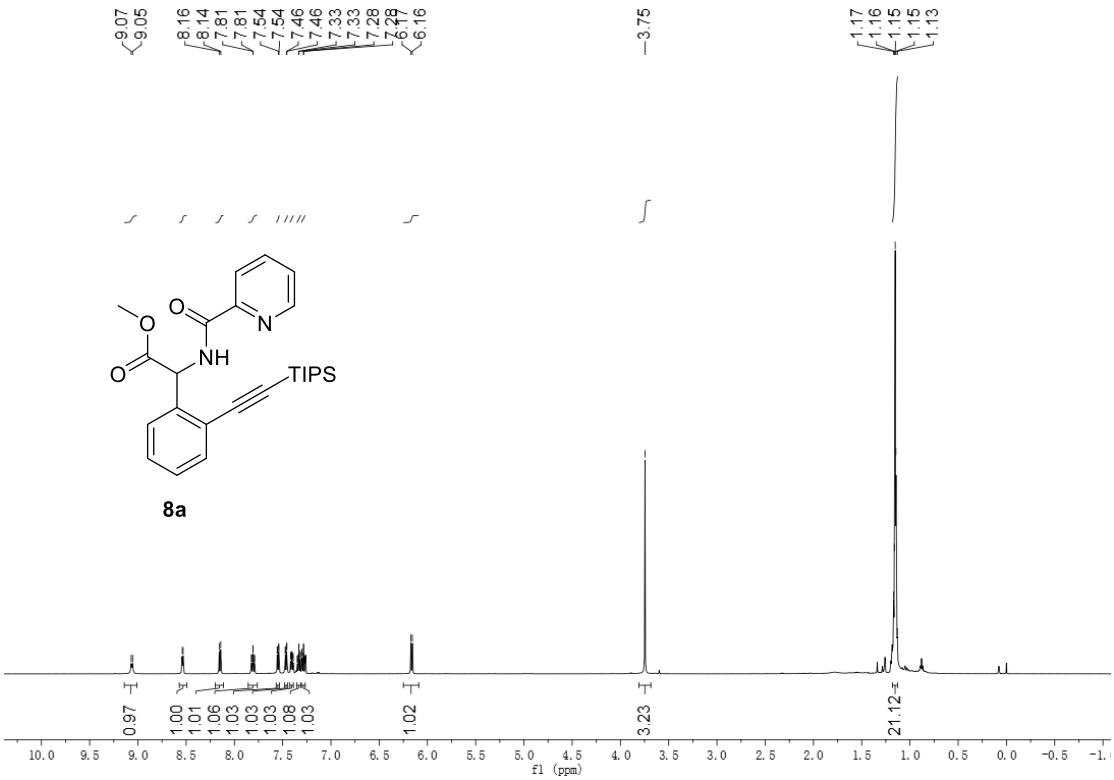
7b


7.43
7.42
7.42
7.41
7.39
7.37
7.29
7.28
7.26
7.25
7.23
7.22
7.21
3.75
2.87
2.86
2.85
2.84
2.83
2.82
2.81
1.67
1.67
1.65
1.45
0.95
0.94
0.92
0.95
1.01
0.97
1.05
2.02
3.15
1.00
3.09
2.18
2.14
2.08
3.00
f1 (ppm)

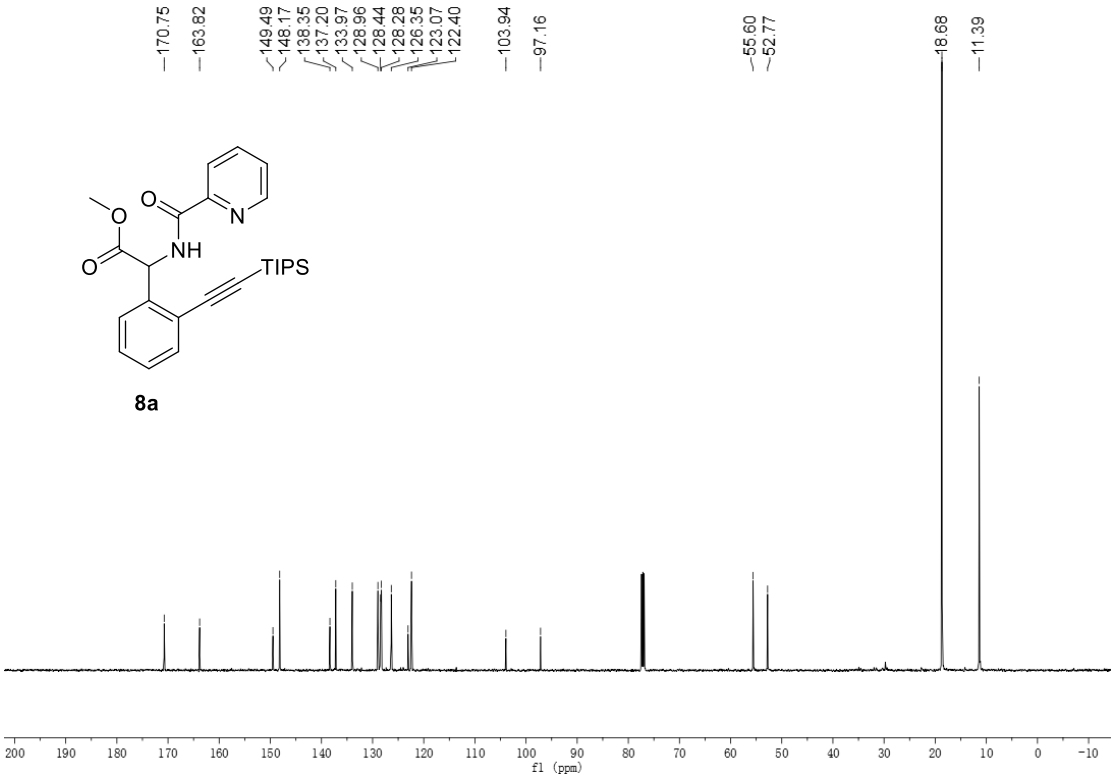
7c


171.77
163.84
149.32
148.31
141.73
137.32
134.46
130.24
128.68
127.03
126.68
126.46
122.39
52.84
52.77
33.57
32.68
22.80
14.05
f1 (ppm)

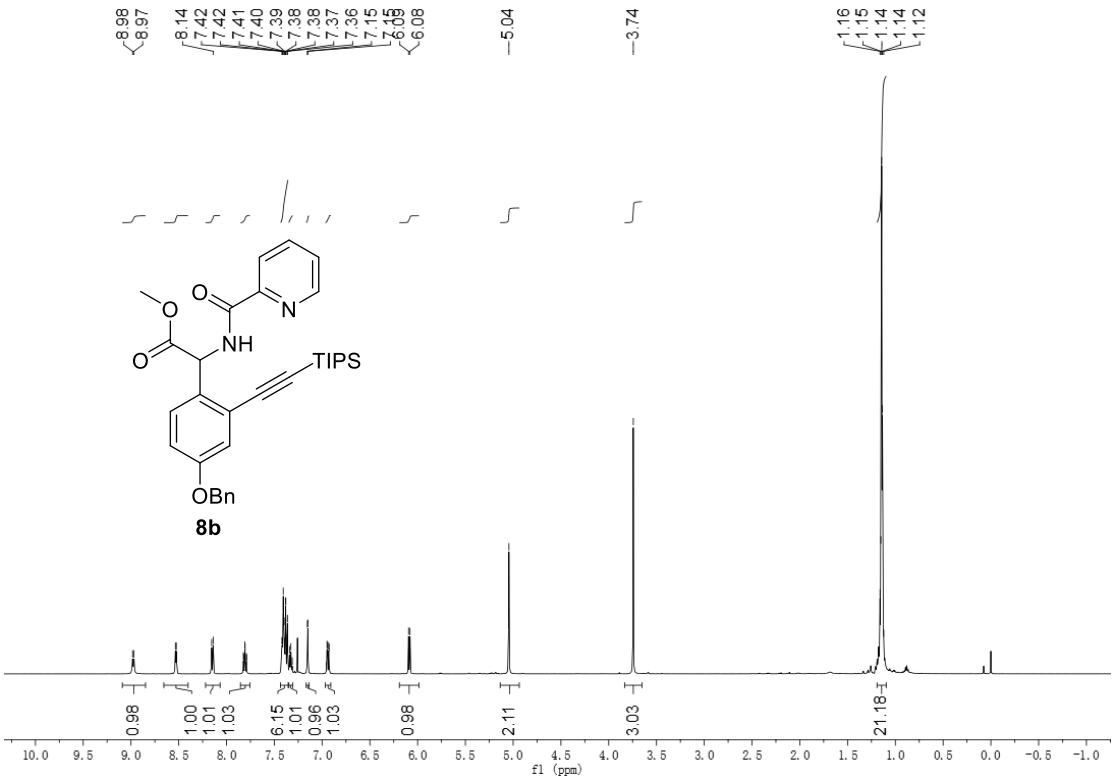
7c

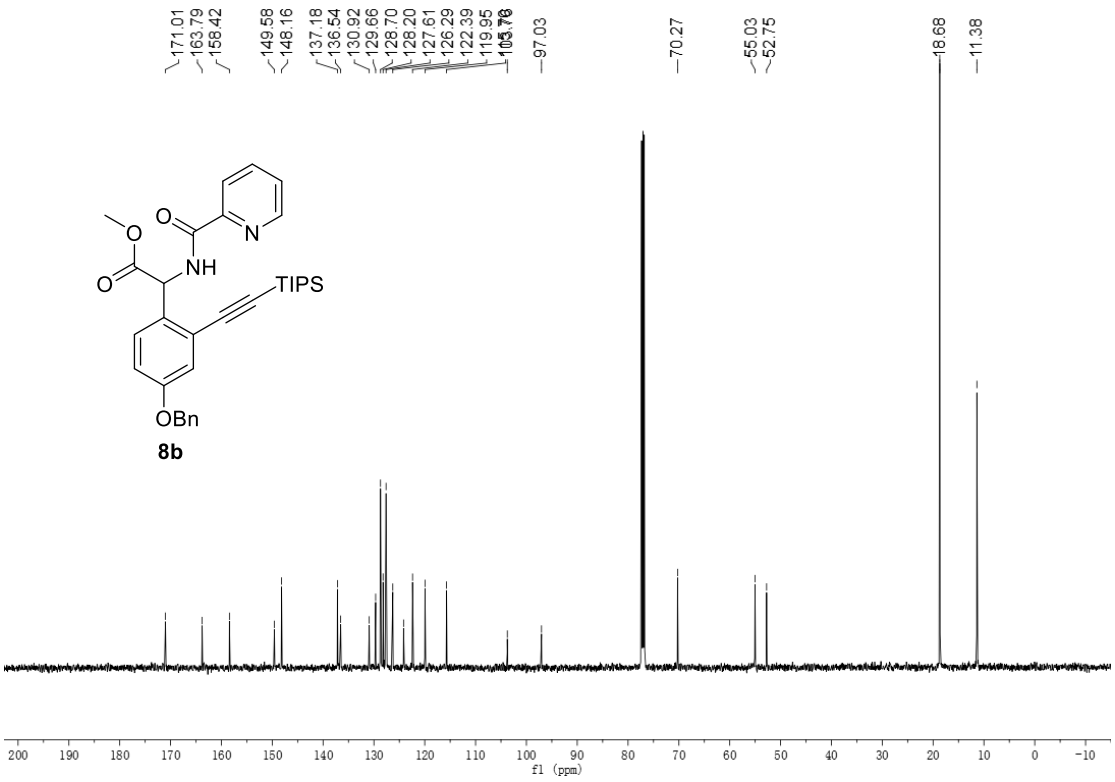

9.07
9.05
8.16
8.14
7.81
7.81
7.54
7.54
7.46
7.46
7.33
7.33
7.28
7.28
6.17
6.16
3.75
1.17
1.16
1.15
1.15
1.13
O
O
N
NH
O
TIPS
0.97
1.00
1.01
1.06
1.03
1.03
1.03
1.08
1.03
1.02
3.23
21.12
f1 (ppm)

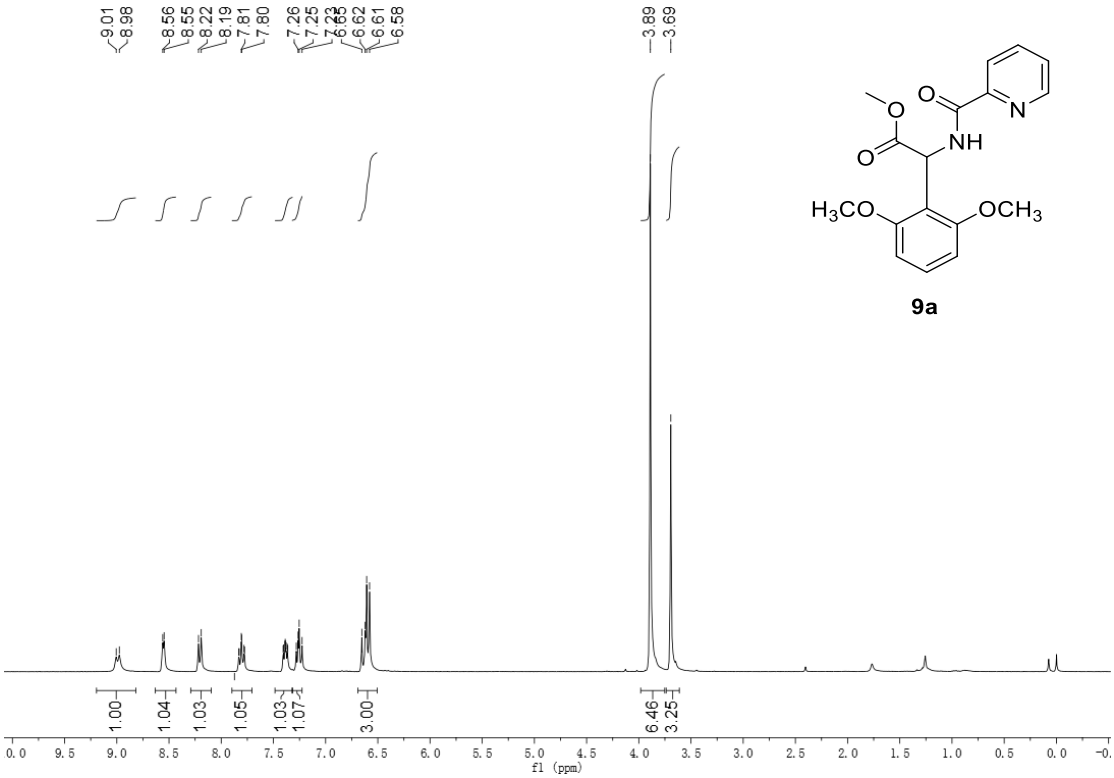
8a


170.75
163.82
149.49
148.17
138.35
137.20
133.97
128.96
128.44
128.28
126.35
123.07
122.40
103.94
97.16
55.60
52.77
18.68
11.39
O
O
N
NH
O
TIPS
f1 (ppm)

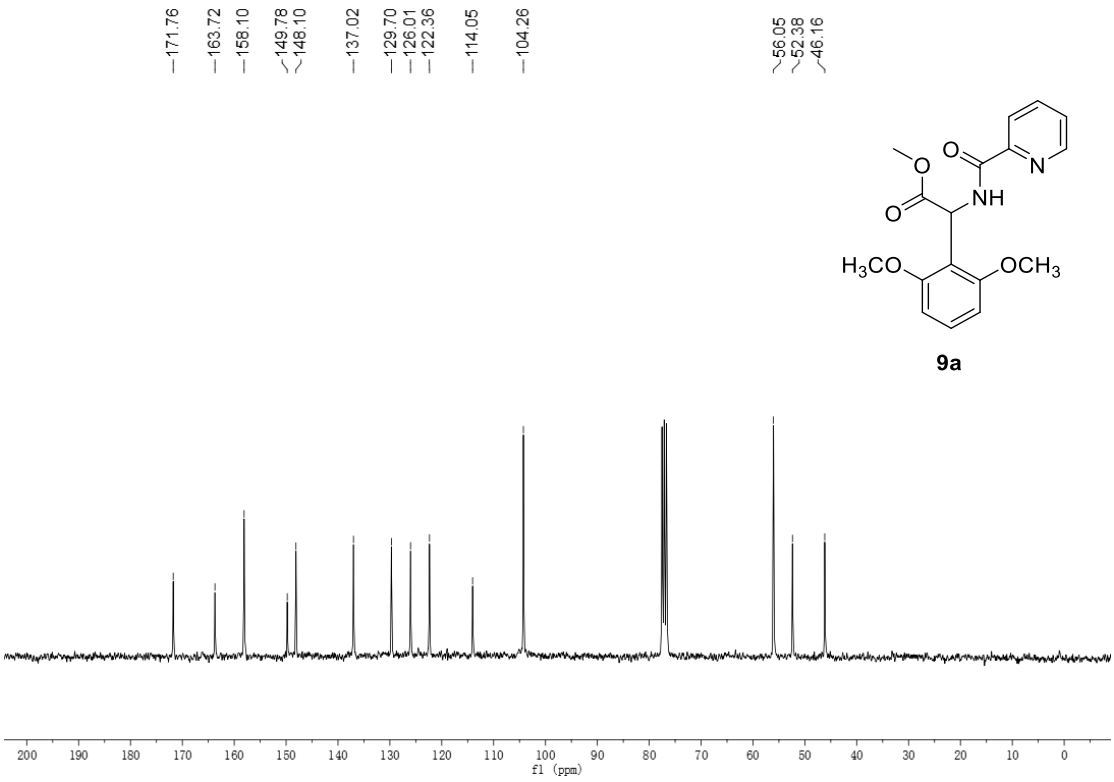
8a


8.98
8.97
8.14
7.42
7.42
7.41
7.40
7.39
7.38
7.38
7.37
7.36
7.15
6.09
6.08
5.04
3.74
1.16
1.15
1.14
1.14
1.12
O
O
N
NH
O
TIPS
OBn
8b
0.98
1.00
1.01
1.03
6.15
1.01
0.96
1.03
0.98
2.11
3.03
21.18
f1 (ppm)


171.01
163.79
158.42
149.58
148.16
137.18
136.54
130.92
129.66
128.70
128.20
127.61
126.29
122.39
119.95
97.03
70.27
55.03
52.75
18.68
11.38
O
O
N
NH
O
TIPS
OBn
8b
f1 (ppm)


9.01
8.98
8.56
8.55
8.22
8.19
7.81
7.80
7.26
7.25
7.23
6.65
6.62
6.61
6.58
3.89
3.69
1.00
1.04
1.03
1.05
1.03
1.07
3.00
6.46
3.25
f1 (ppm)

9a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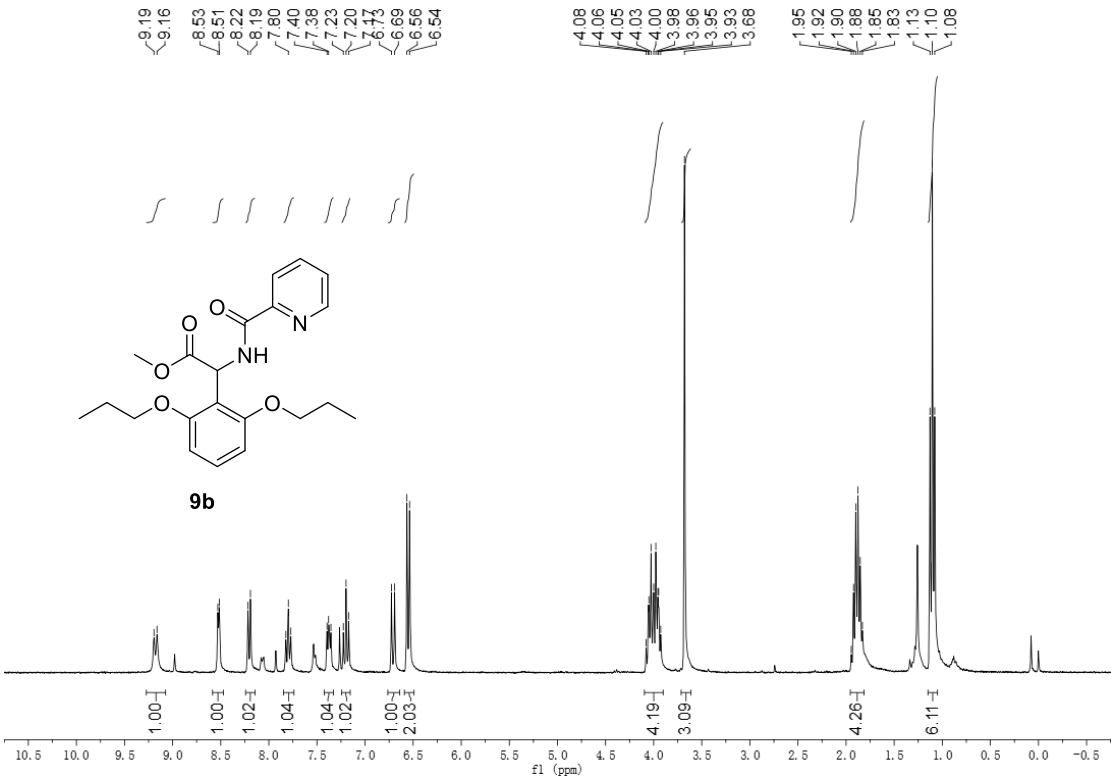
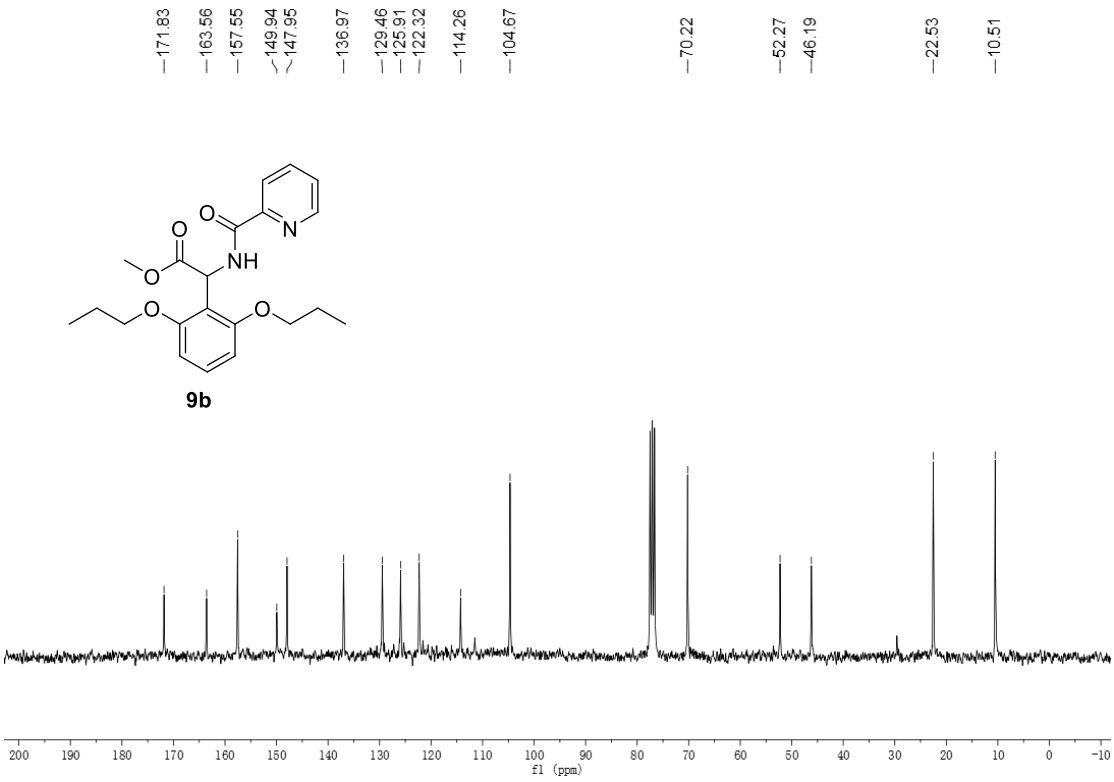
163.72
158.10
149.78
148.10
137.02
129.70
126.01
122.36
114.05
104.26
56.05
52.38
46.16
f1 (ppm)

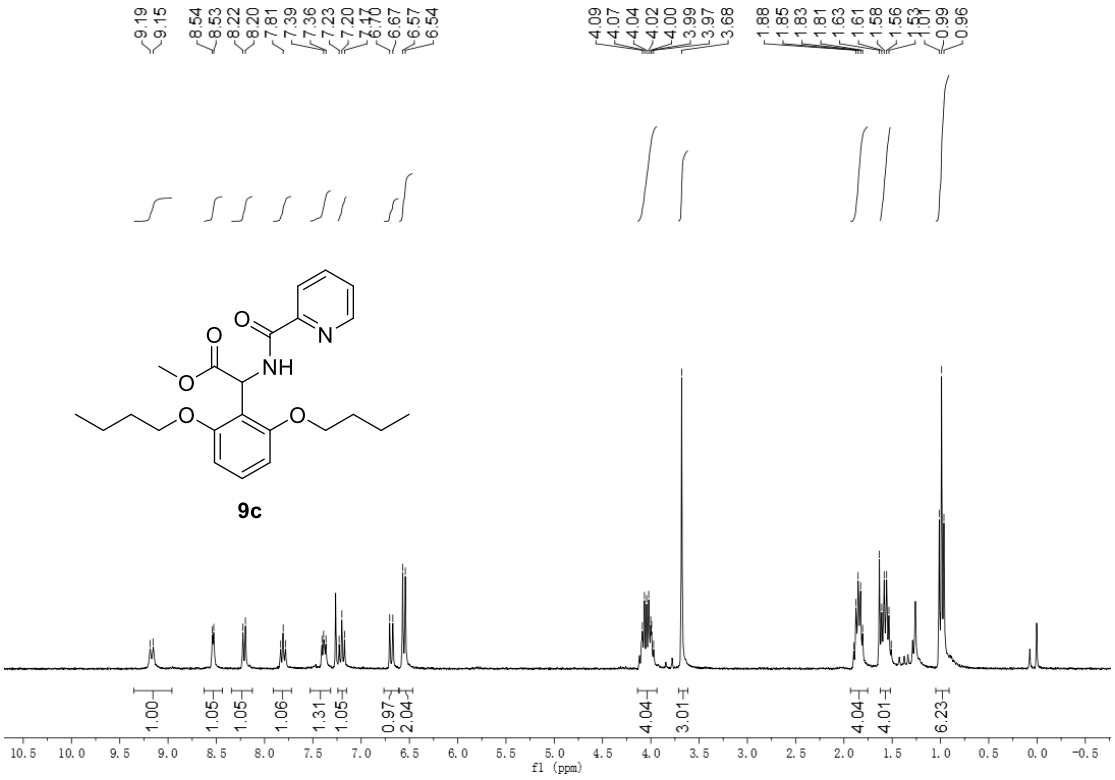
9a


9.19
9.16
8.53
8.51
8.22
8.19
7.80
7.40
7.38
7.23
7.20
6.69
6.56
6.54
4.08
4.06
4.05
4.03
4.00
3.98
3.96
3.95
3.93
3.68
1.95
1.92
1.90
1.88
1.85
1.83
1.13
1.10
1.08
O
N
O
NH
O
O
O
1.00
1.00
1.02
1.04
1.04
1.02
1.00
2.03
4.19
3.09
4.26
6.11
f1 (ppm)

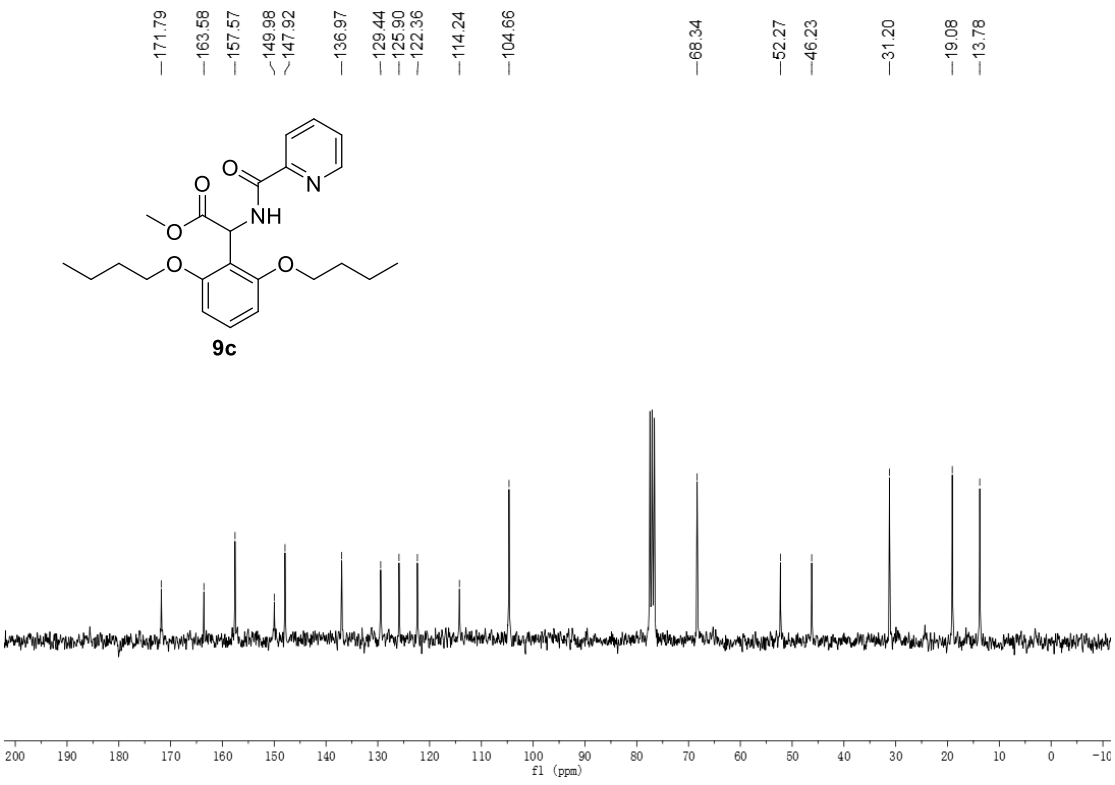
9b


171.83
163.56
157.55
149.94
147.95
136.97
129.46
125.91
122.32
114.26
104.67
70.22
52.27
46.19
22.53
10.51
O
N
O
NH
O
O
O
f1 (ppm)

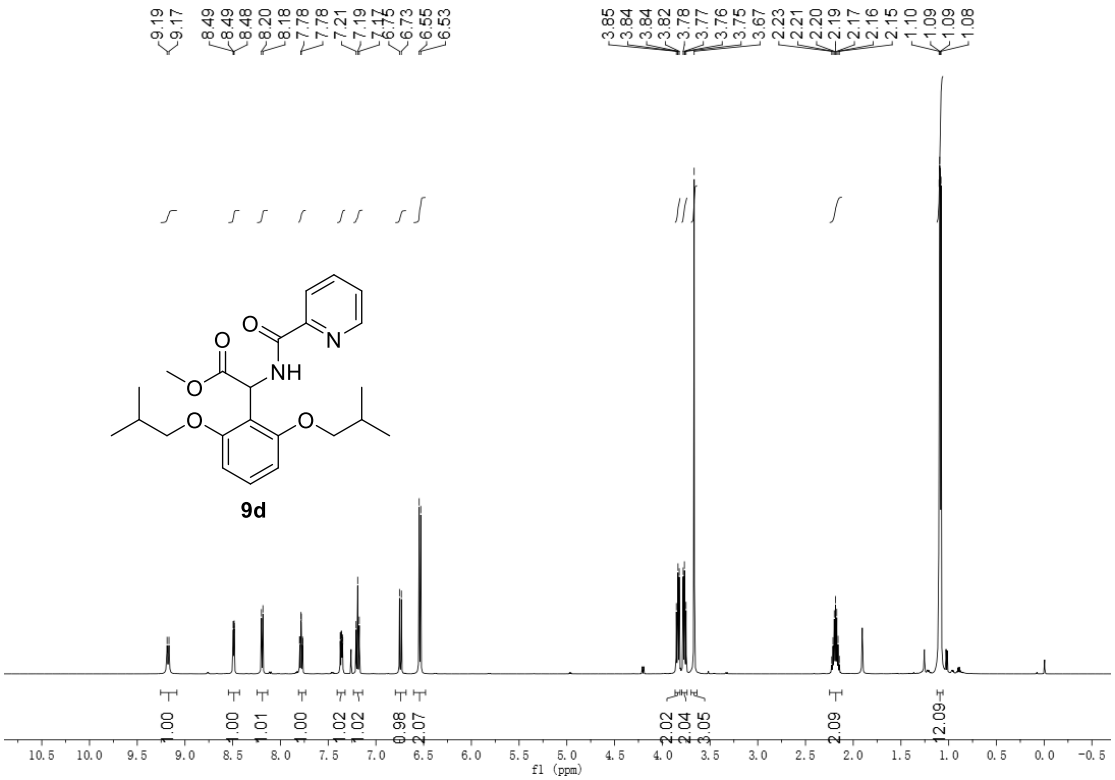
9b


9.19
9.15
8.54
8.53
8.22
8.20
7.81
7.39
7.36
7.23
7.20
7.17
7.10
6.67
6.57
6.54
4.09
4.07
4.04
4.02
4.00
3.99
3.97
3.68
1.88
1.85
1.83
1.81
1.63
1.61
1.58
1.56
1.53
1.51
0.99
0.96
O
N
O
NH
O
O
O
1.00
1.05
1.05
1.06
1.31
1.05
0.97
2.04
4.04
3.01
4.04
4.01
6.23
f1 (ppm)

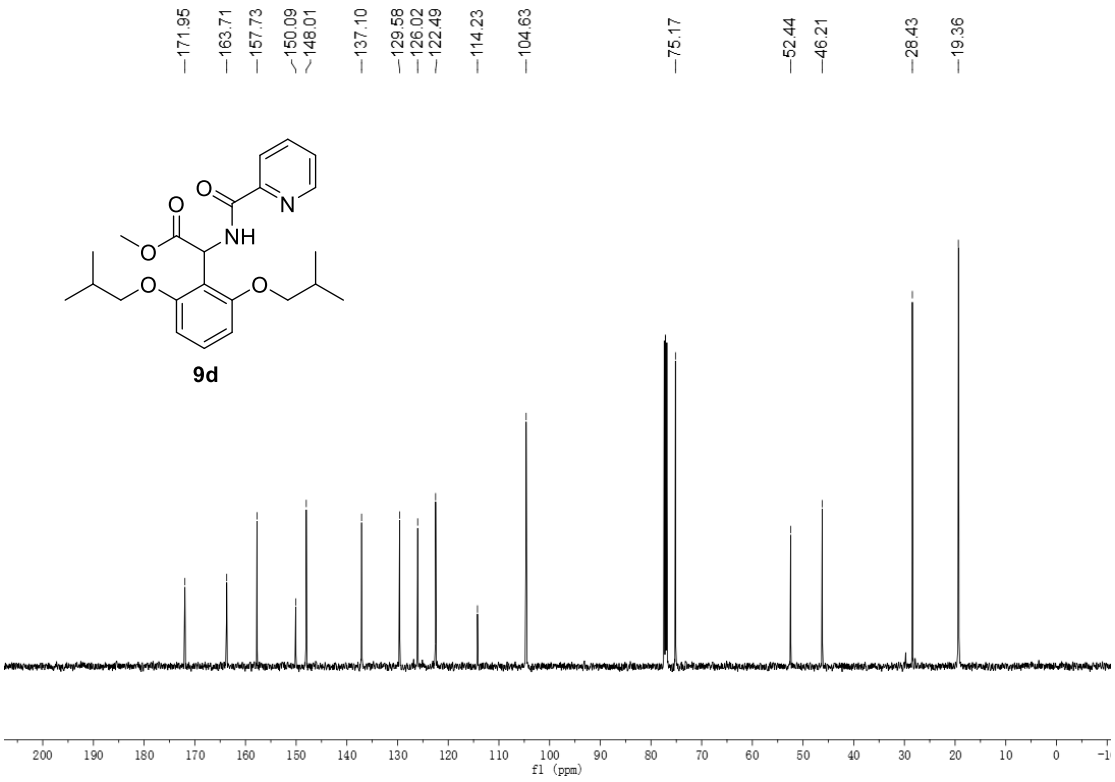
9c


171.79
163.58
157.57
149.98
147.92
136.97
129.44
125.90
122.36
114.24
104.66
68.34
52.27
46.23
31.20
19.08
13.78
O
N
O
NH
O
O
O
f1 (ppm)

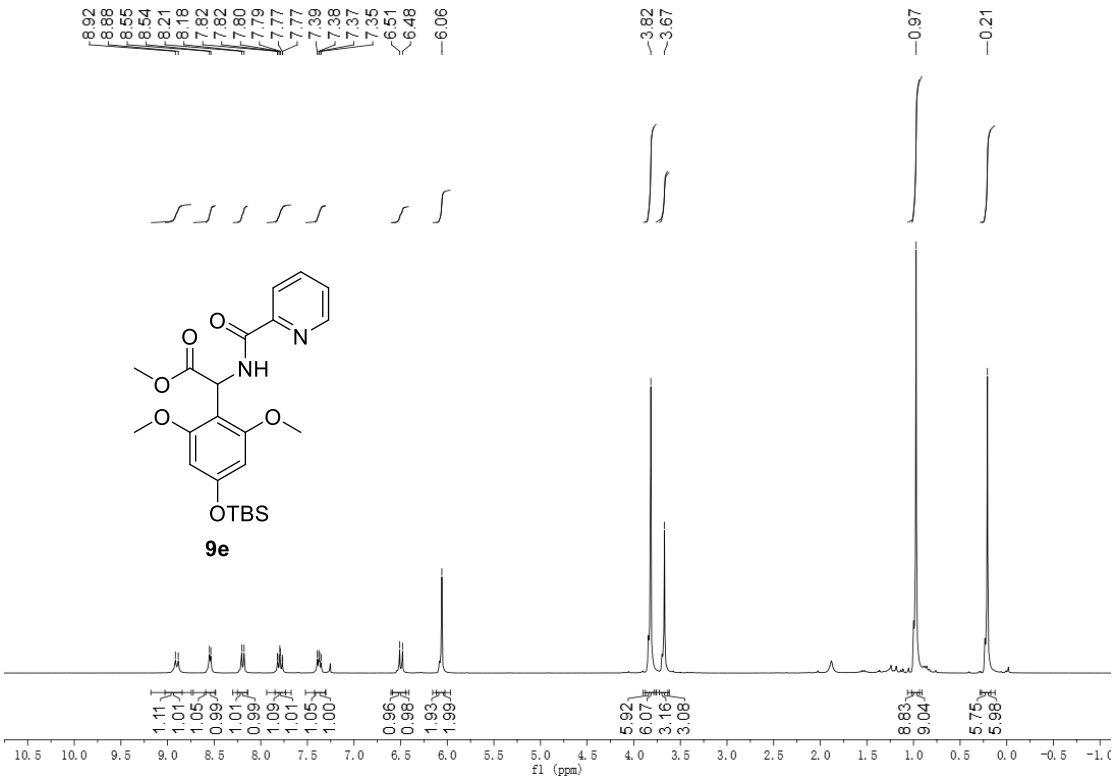
9c

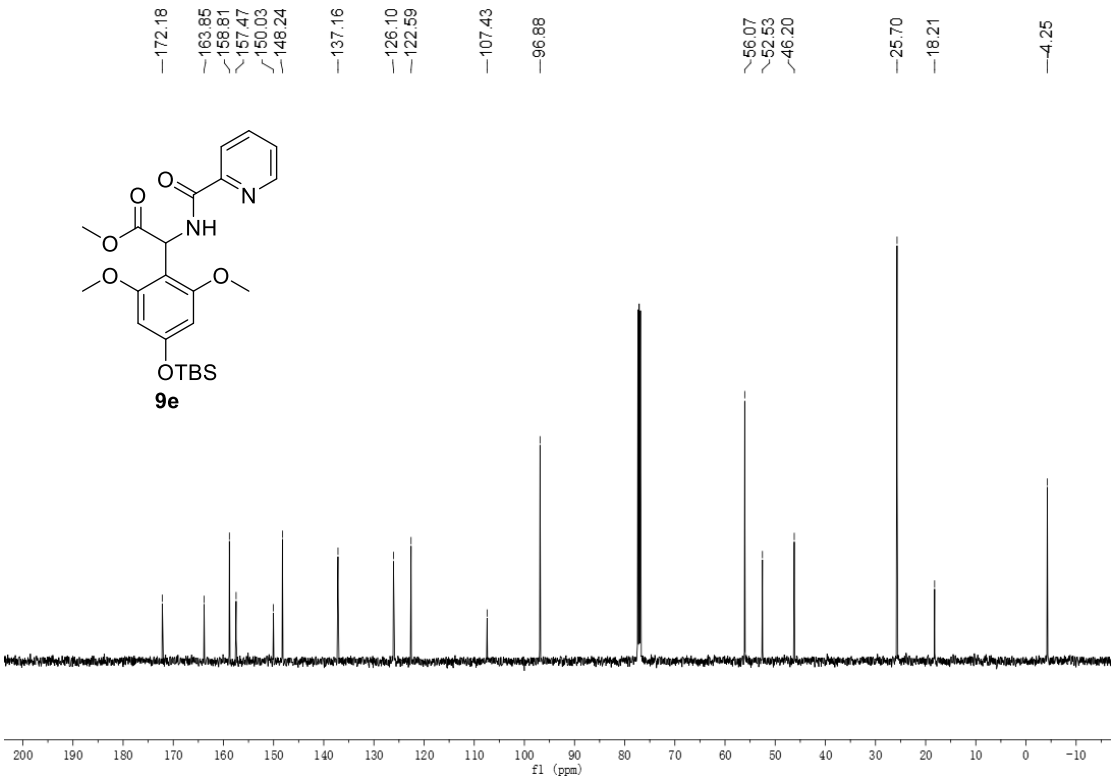

9.19
9.17
8.49
8.49
8.48
8.20
8.18
7.78
7.78
7.21
7.19
7.17
6.75
6.73
6.55
6.53
3.85
3.84
3.84
3.82
3.78
3.77
3.76
3.75
3.67
2.23
2.21
2.20
2.19
2.17
2.16
2.15
1.10
1.09
1.09
1.08
O
N
O
NH
O
O
O
1.00
1.00
1.01
1.00
1.02
1.02
0.98
2.07
2.02
2.04
3.05
2.09
12.09
f1 (ppm)

9d


171.95
163.71
157.73
150.09
148.01
137.10
129.58
126.02
122.49
114.23
104.63
75.17
52.44
46.21
28.43
19.36
O
N
O
NH
O
O
O
f1 (ppm)

9d


8.92
8.88
8.55
8.54
8.21
8.18
7.82
7.82
7.80
7.79
7.77
7.77
7.39
7.38
7.37
7.35
6.51
6.48
6.06
3.82
3.67
0.97
0.21
O
N
NH
O
O
O
O
OTBS
9e
1.11
1.01
1.05
0.99
1.01
0.99
1.09
1.01
1.05
1.00
0.96
0.98
1.93
1.99
5.92
6.07
3.16
3.08
8.83
9.04
5.75
5.98
f1 (ppm)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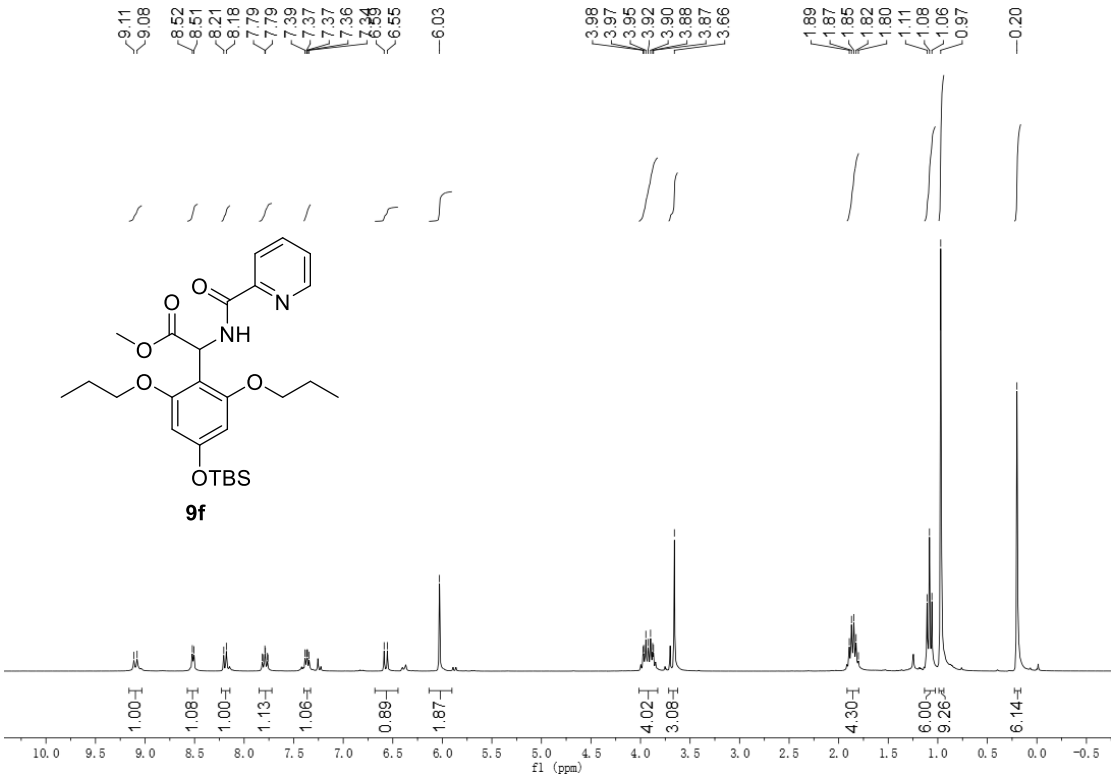
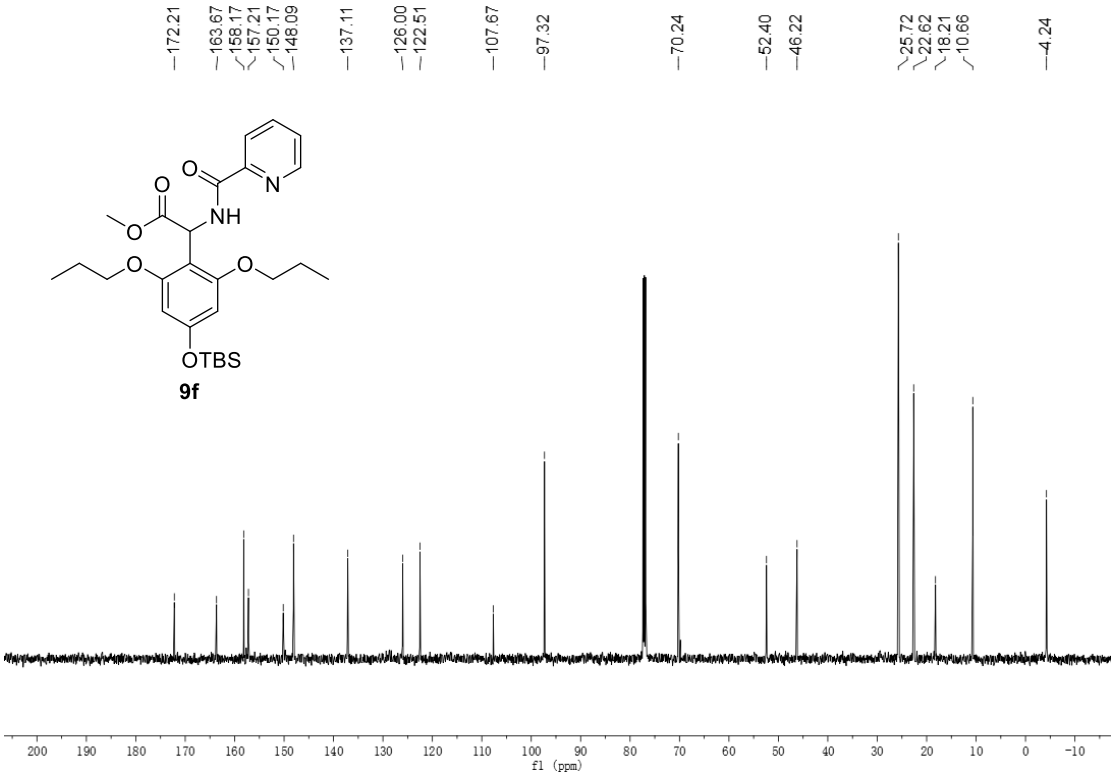
163.85
158.81
157.47
150.03
148.24
137.16
126.10
122.59
107.43
96.88
56.07
52.53
46.20
25.70
18.21
4.25
O
N
NH
O
O
O
O
OTBS
9e
f1 (ppm)


9.11
9.08
8.52
8.51
8.21
8.18
7.79
7.79
7.39
7.37
7.37
7.36
6.56
6.55
6.03
3.98
3.97
3.95
3.92
3.90
3.88
3.87
3.66
1.89
1.87
1.85
1.82
1.80
1.11
1.08
1.06
0.97
0.20
O
N
O
NH
O
O
O
OTBS
9f
1.00
1.08
1.00
1.13
1.06
0.89
1.87
4.02
3.08
4.30
6.00
9.26
6.14
f1 (ppm)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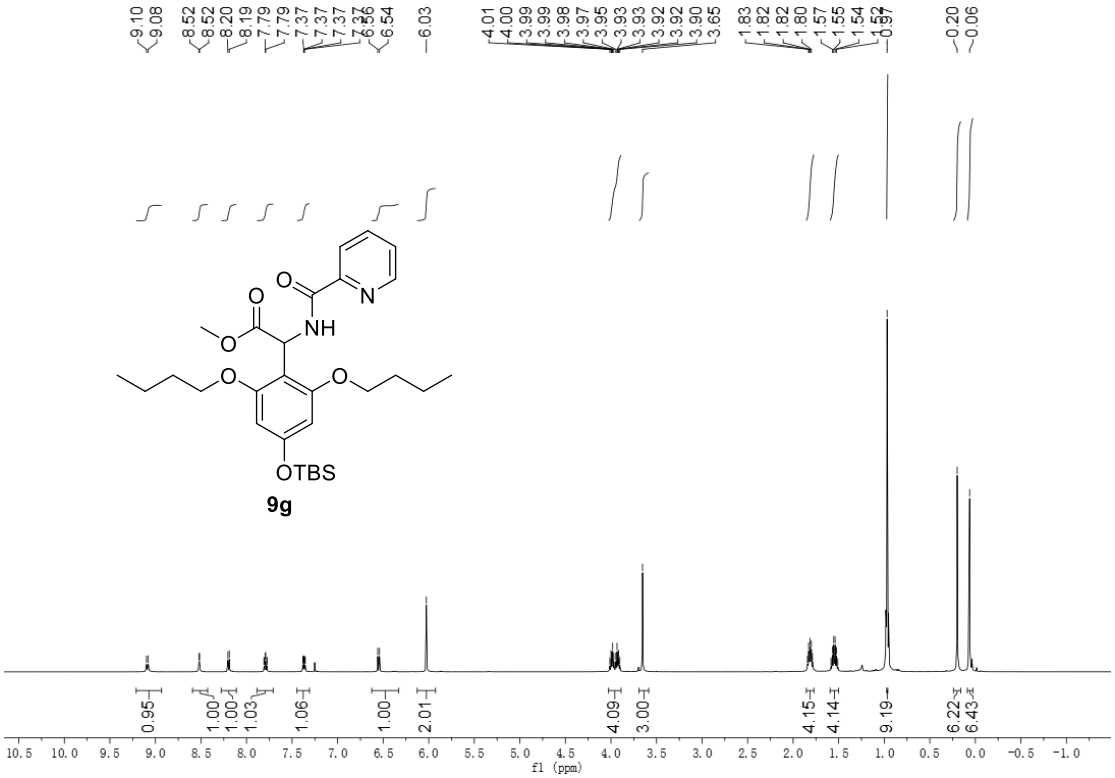
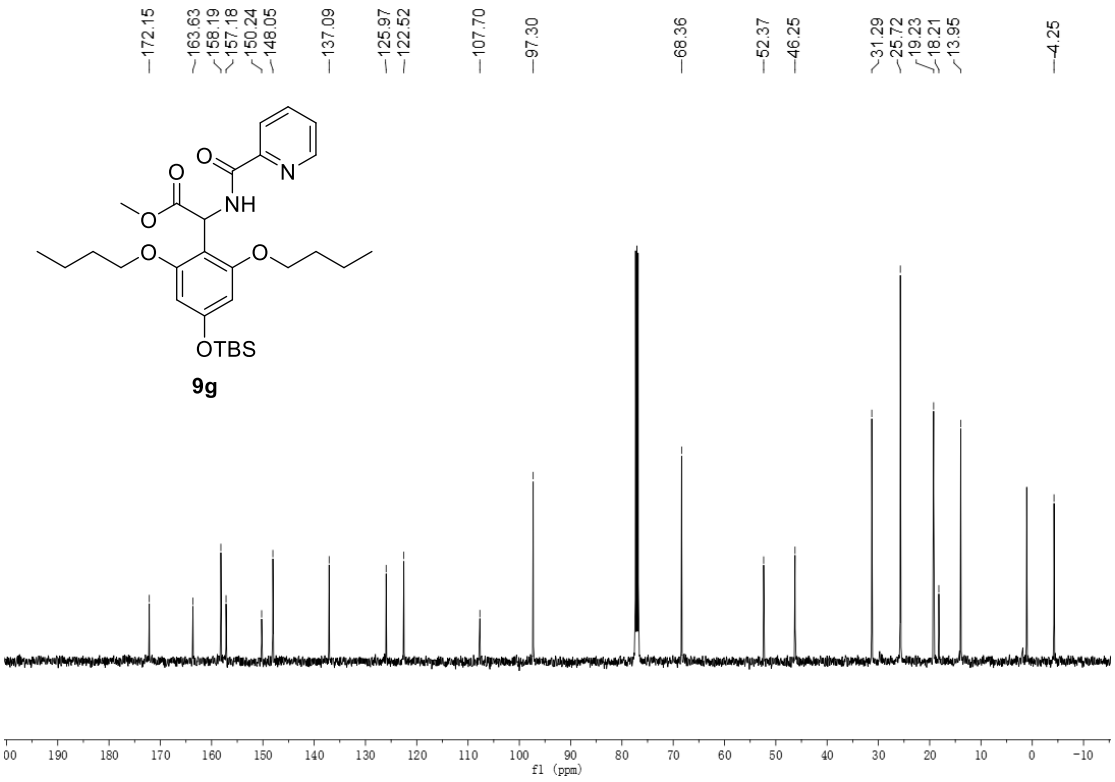
158.17
157.21
150.17
148.09
137.11
126.00
122.51
107.67
97.32
70.24
52.40
46.22
25.72
22.62
18.21
10.66
4.24
O
N
O
NH
O
O
O
OTBS
9f
f1 (ppm)


9.10
9.08
8.52
8.52
8.20
8.19
7.79
7.79
7.37
7.37
7.37
6.56
6.54
6.03
4.01
4.00
3.99
3.99
3.98
3.97
3.95
3.93
3.93
3.92
3.92
3.90
3.65
1.83
1.82
1.82
1.80
1.57
1.55
1.54
0.97
0.20
0.06
O
N
O
NH
O
O
O
OTBS
9g
0.95
1.00
1.00
1.03
1.06
1.00
2.01
4.09
3.00
4.15
4.14
9.19
6.22
6.43
f1 (ppm)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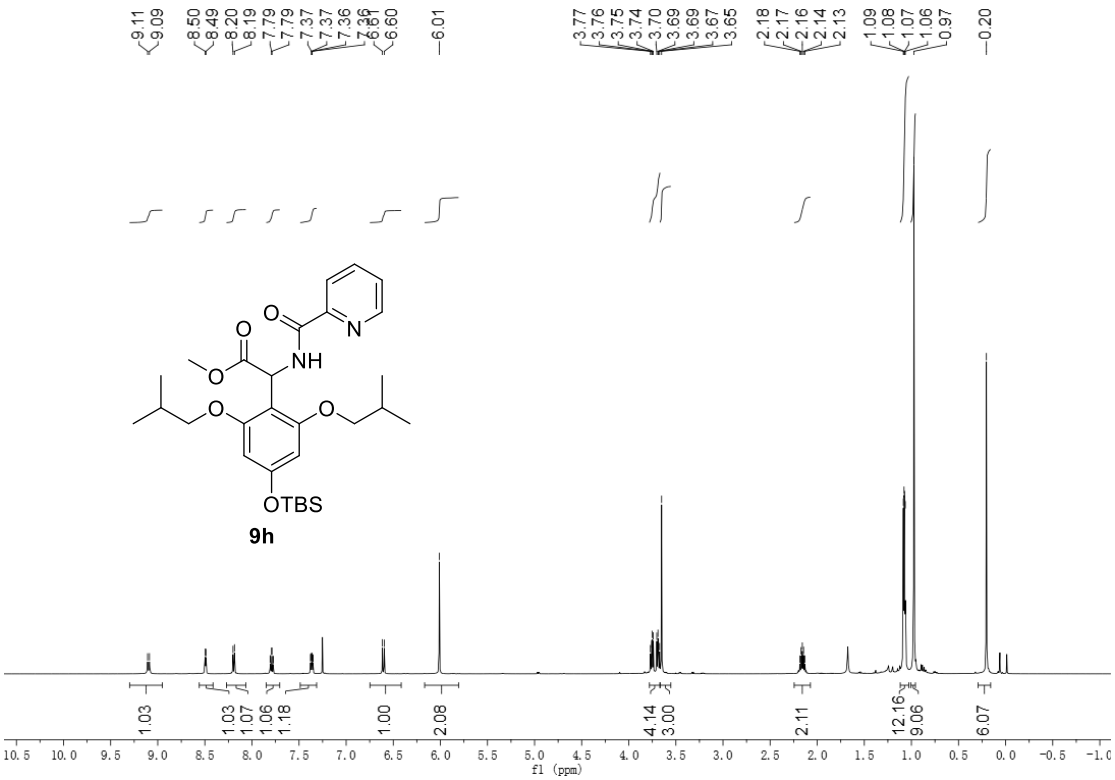
163.63
158.19
157.18
150.24
148.05
137.09
125.97
122.52
107.70
97.30
68.36
52.37
46.25
31.29
25.72
19.23
18.21
13.95
4.25
O
N
O
NH
O
O
O
OTBS
9g
f1 (ppm)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9.09
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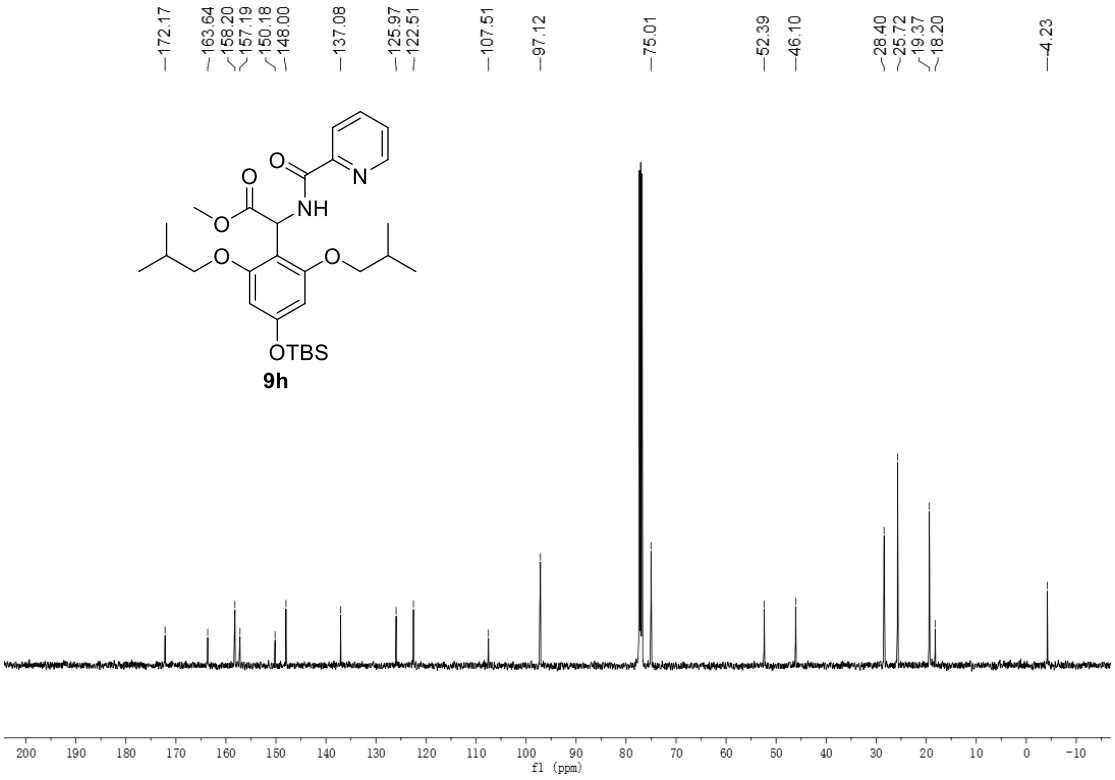
8.49
8.20
8.19
7.79
7.79
7.37
7.37
7.36
7.36
6.61
6.60
6.01
3.77
3.76
3.75
3.74
3.70
3.69
3.69
3.67
3.65
2.18
2.17
2.16
2.14
2.13
1.09
1.08
1.07
1.06
0.97
0.20
O
N
O
NH
O
O
O
OTBS
9h
1.03
1.03
1.07
1.06
1.18
1.00
2.08
4.14
3.00
2.11
12.16
9.06
6.07
f1 (ppm)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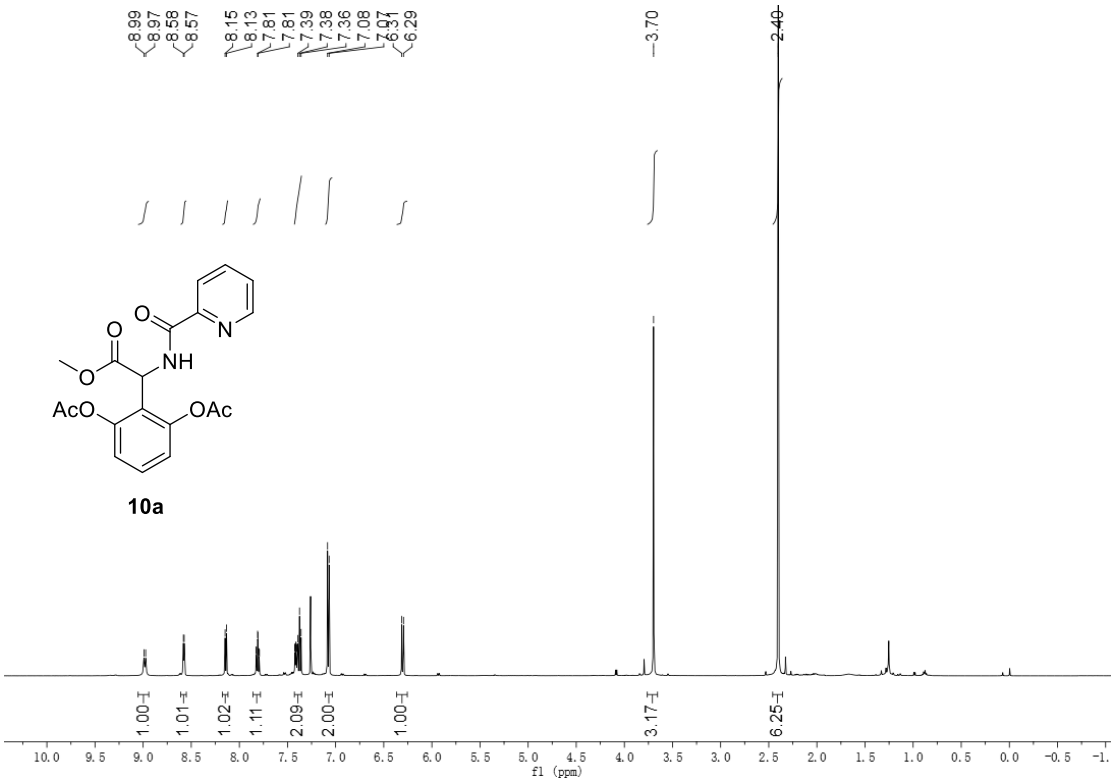
158.20
157.19
150.18
148.00
137.08
125.97
122.51
107.51
97.12
75.01
52.39
46.10
28.40
25.72
19.37
18.20
4.23
O
N
O
NH
O
O
O
OTBS
9h
f1 (ppm)


8.99
8.97
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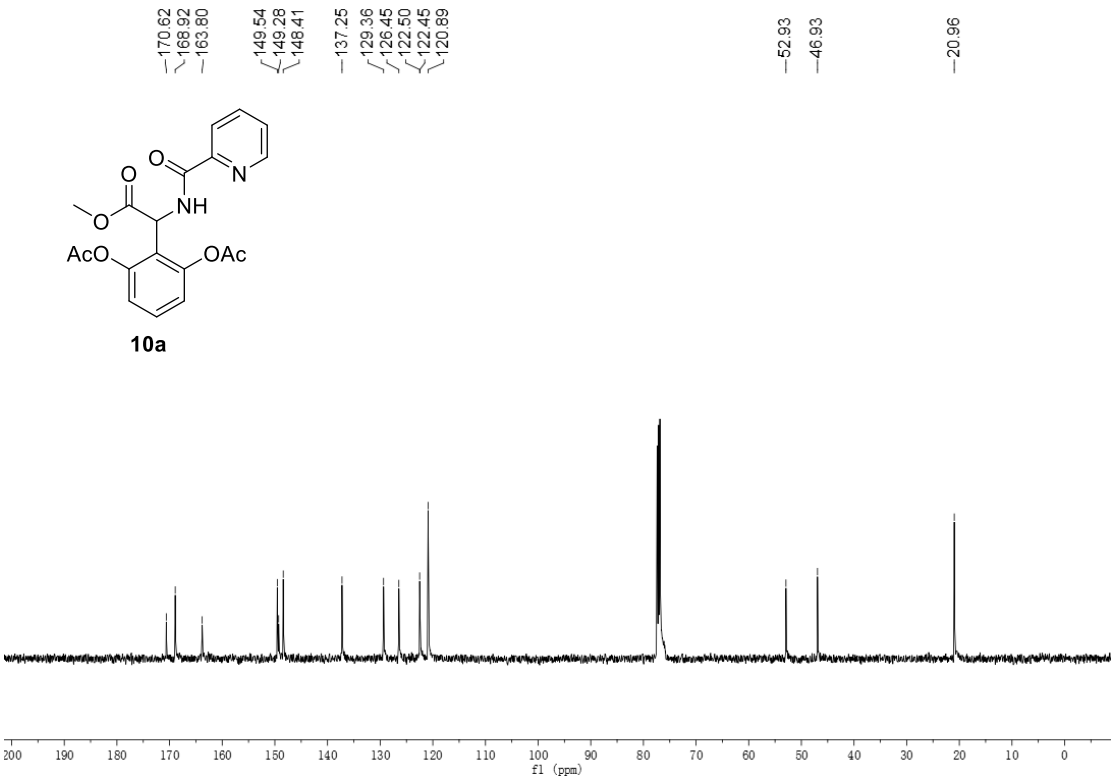
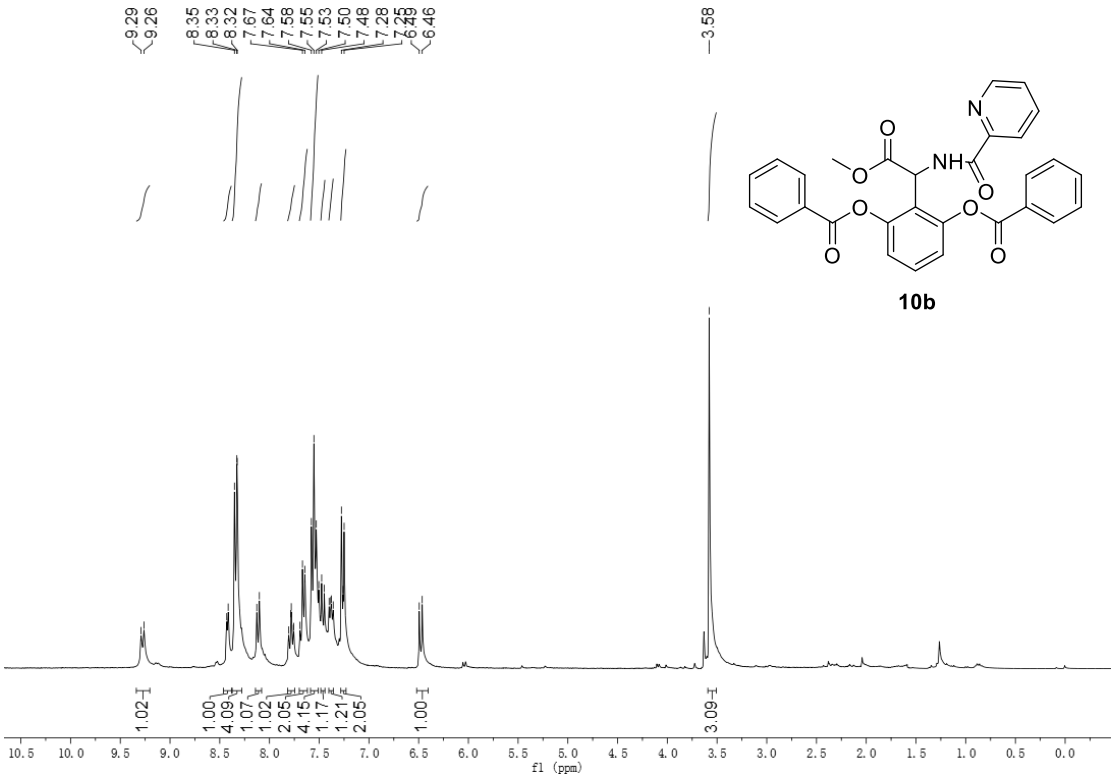
8.57
8.15
8.13
7.81
7.81
7.39
7.38
7.36
7.08
6.31
6.29
3.70
2.40
O
N
O
NH
O
AcO
OAc
1.00
1.01
1.02
1.11
2.09
2.00
1.00
3.17
6.25
f1 (ppm)

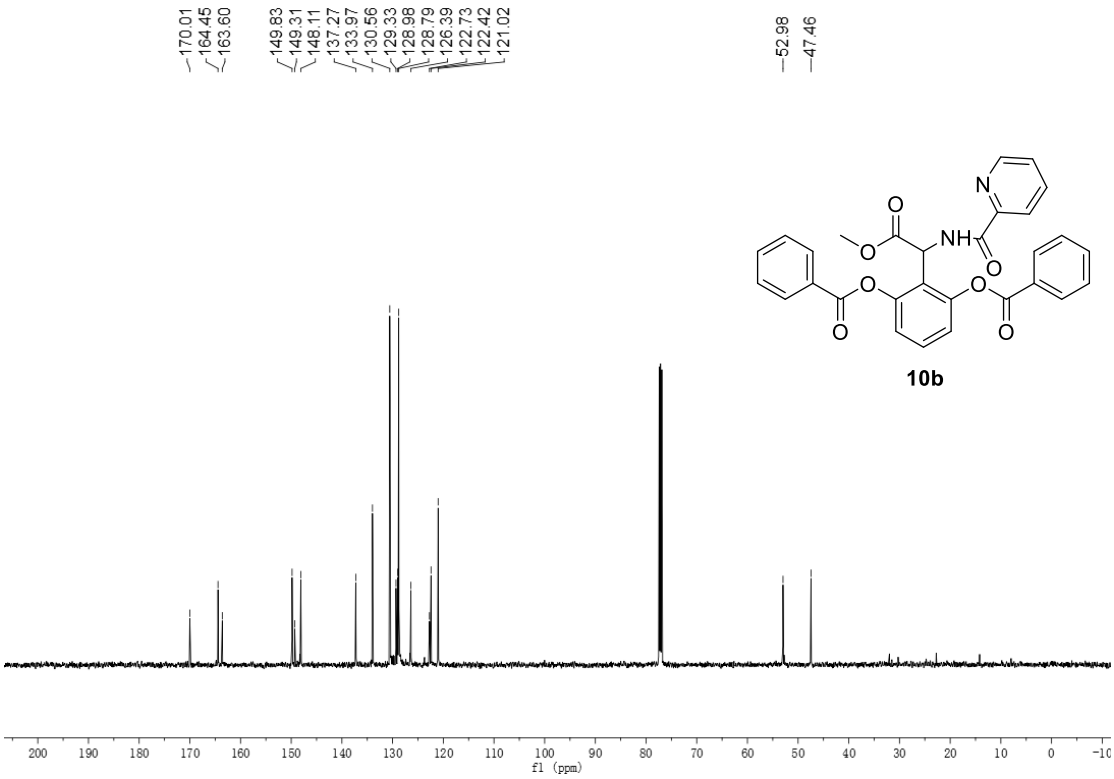
10a


170.62
168.92
163.80
149.54
149.28
148.41
137.25
129.36
126.45
122.50
122.45
120.89
52.93
46.93
20.96
O
N
O
NH
O
AcO
OAc
f1 (ppm)

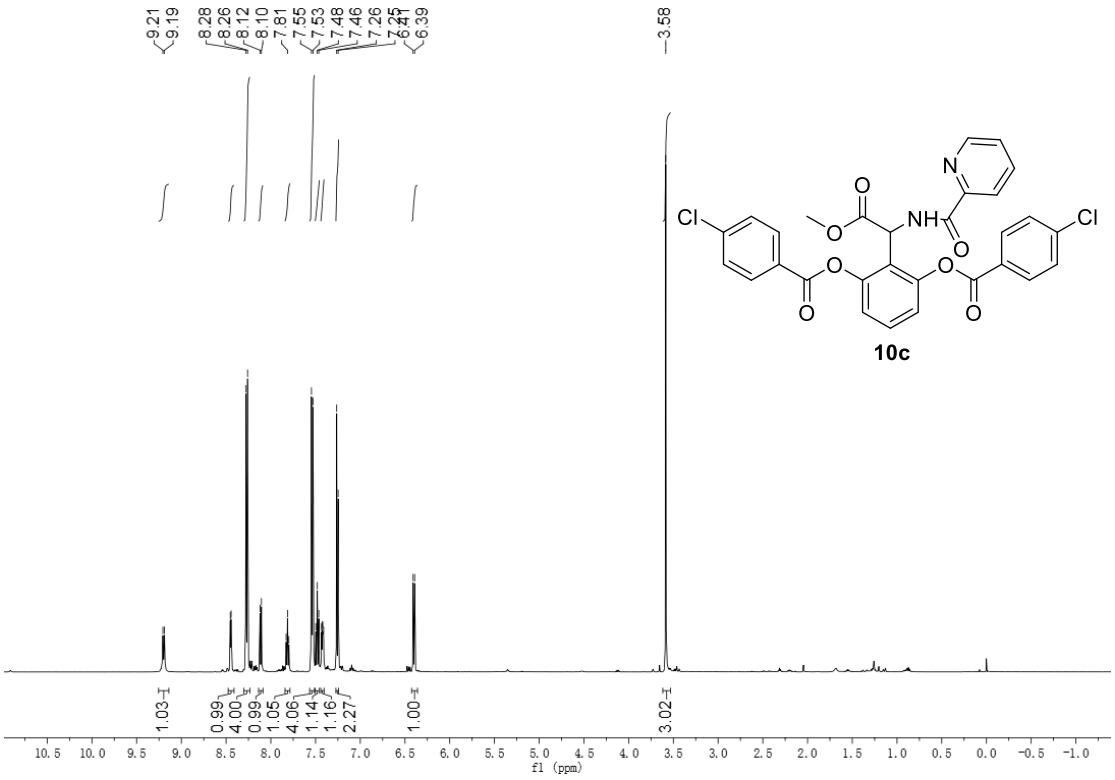
10a


9.29
9.26
8.35
8.33
8.32
7.67
7.64
7.58
7.55
7.53
7.50
7.48
7.28
6.49
6.46
3.58
1.02
1.00
4.09
1.07
1.02
2.05
4.15
1.17
1.21
2.05
1.00
3.09
f1 (ppm)

10b


170.01
164.45
163.60
149.83
149.31
148.11
137.27
133.97
130.56
129.33
128.98
128.79
126.39
122.73
122.42
121.02
52.98
47.46
f1 (ppm)

10b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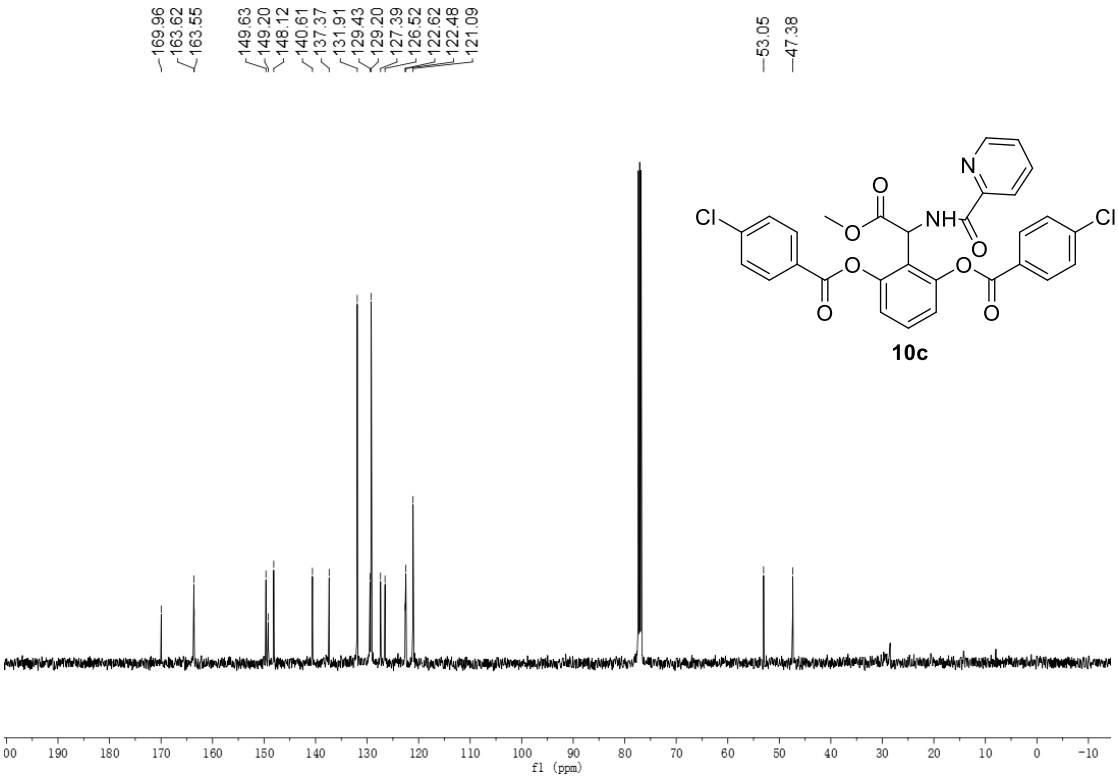
9.19
8.28
8.26
8.12
8.10
7.81
7.55
7.53
7.48
7.46
7.26
6.41
6.39
3.58
1.03
0.99
4.00
0.99
1.05
4.06
1.14
1.16
2.27
1.00
3.02
f1 (ppm)

N
O
NH
O
Cl
Cl
O
O
O
O

10c


169.96
163.62
163.55
149.63
149.20
148.12
140.61
137.37
131.91
129.43
129.20
127.39
126.52
122.62
122.48
121.09
53.05
47.38
f1 (ppm)

N
O
NH
O
Cl
Cl
O
O
O
O

10c