Supporting Information

Palladium-catalysed aminocarbonylation of aryl thianthrenium salts to aryl carboxamides using $Mo(CO)_6$ as a CO source

Zhuo Chen, Hui-Qiang Li, Shun-Bin Xu, Ling-Rui Zhou, Tong-Ling Qin, Wen-Xia Hu, Tao Li, Ruo-Yi Hou, Zi-Yu Lin and E Tang*

Key Laboratory of Medicinal Chemistry for Natural Resources, Ministry of Education, Yunnan Key Laboratory of Research and Development for Natural Products, Yunnan Characteristic Plant Extraction Laboratory, School of Pharmacy, Yunnan Key Laboratory of Cross-Border Infectious Disease Control and Prevention and Novel Drug Development, Yunnan University, Kunming, 650500, P. R. China.

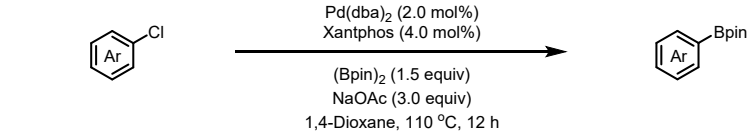
Table of Content

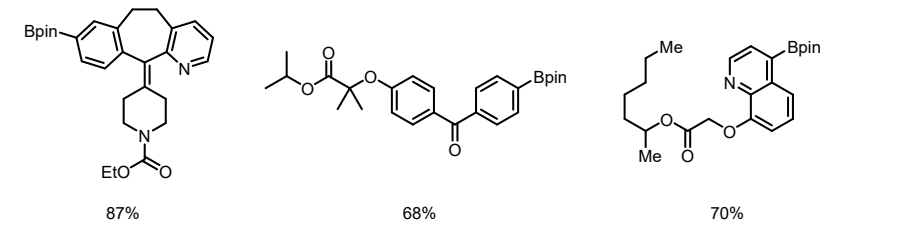
1. General Information

Unless otherwise noted, all other reagents and starting materials were purchased from commercial sources and used without further purification. Thin layer chromatography (TLC) was carried out on GF 254 plates (0.25 mm layer thickness). Flash chromatography was performed with 200-300 mesh silica gels. Reactions were monitored by TLC and visualized by a dual short-wave/long-wave UV lamp. ^{1}H NMR, ^{13}C NMR, and ^{19}F NMR spectra were recorded on Bruker Avance 400, 500, or 600 MHz spectrophotometers. Chemical shifts in ^{1}H NMR spectra were reported in parts per million (ppm) on the δ scale. Data for ^{1}H NMR were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, br. s = broad singlet), coupling constant in herts (Hz) and integration. Data for ^{13}C NMR spectra were reported in terms of chemical shift in ppm. ESI-MS was taken on a Thermo Fisher Scientific LTQ-Orbitrap XL spectrometer.

2. Preparation of Substrates

2.1 Preparation of Bioactive Molecule-Derived Arylborons

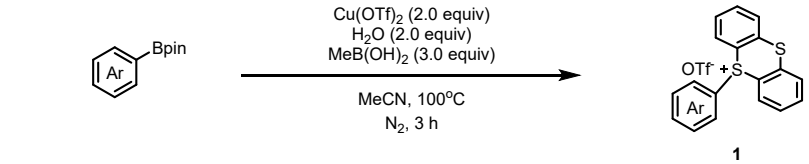


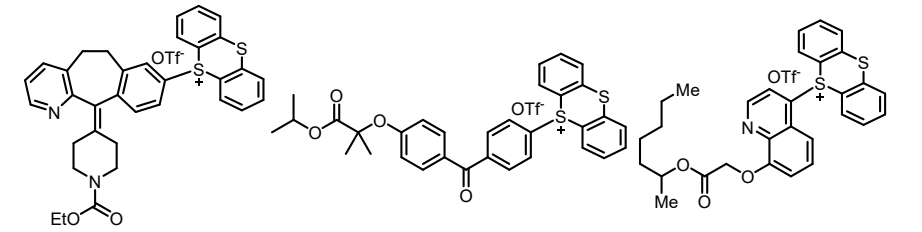


To a mixture of aryl chloride (5.0 mmol, 1.0 equiv.), $Pd_2(dba)_3$ (95.0 mg, 0.1 mmol, 2.0 mol%), Xantphos (97.5 mg, 0.2 mmol, 4.0 mol%), $(BPin)_2$ (1.9 g, 7.5 mmol, 1.5 equiv.), and NaOAc (1.23 g, 18.0 mmol, 3.0 equiv.) in a Schlenk tube under N_2 was added 1.4-dioxane (10 mL). The tube was sealed, and the reaction mixture was stirred at 110 °C for 12 h and then cooled to room temperature. Water was added, and the mixture was extracted with ethyl acetate (EA, 2×150 mL). The combined organic layers were washed with brine (100 mL), dried over Na_2SO_4, filtered, and concentrated under

reduced pressure. The residue was purified by flash column chromatography to give the corresponding aryl boronic acid pinacol ester.

2.2 Thianthrenation of Bioactive Molecule-Derived Arylborons

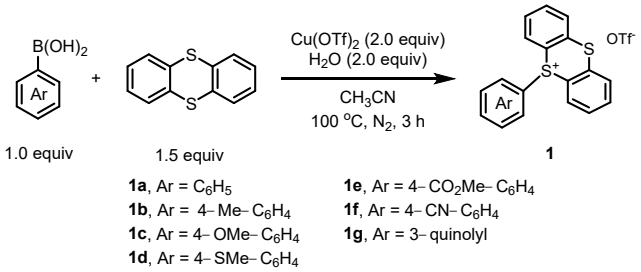




A 10 mL Schlenk tube was charged with aryl boronic acid pinacol ester (2.0 mmol), thianthrene (648 mg, 6.0 mmol, 1.5 equiv.), and $MeB(OH)_2$ (360 mg, 12 mmol, 3.0 equiv.), followed by the addition of $Cu(OTf)_2$ (1.45 g, 4.0 mmol, 2.0 equiv.). Then, the reaction tube was backfilled with N_2 three times. H_2O (18 μL, 2.0 mmol, 2.0 equiv.) and MeCN (2 mL) were added under a nitrogen atmosphere. The reaction mixture was stirred at 100 °C for 3 h. After cooling to room temperature, the reaction mixture was added into ammonia solution (50 mL, 25%–28% aqueous solution), and the water phase was extracted with DCM (2×60 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under vacuum. The residue was dissolved in DCM, and precipitated by adding the solution into the stirring Et_2O (200 mL). The solid was collected by filtration to afford the arylthianthrenium salts **1** without further purification.

2.3 Preparation of Aryl Thianthrenium Salts (1)

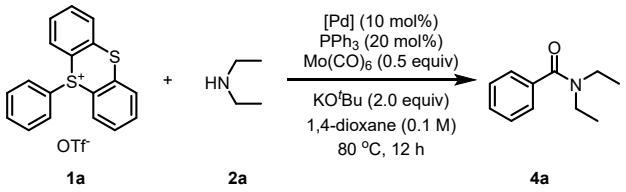
Compounds **1** were prepared according to literature procedures[1]. And the spectra are matching with the literature reports.



A 10 mL Schlenk tube was charged with aryl boronic acid or heteroaryl boronic acid (2.0 mmol, 1.0 equiv.), thianthrene (649 mg, 3.0 mmol, 1.5 equiv.), followed by the addition of $Cu(OTf)_2$ (1.45 g, 4.0 mmol, 2.0 equiv.). Then, the reaction tube was backfilled with N_2 three times, and H_2O (180 μL, 4.0 mmol, 2.0 equiv.) and MeCN (2.0 mL) were added under a nitrogen atmosphere. The reaction mixture was stirred at 100 °C for 3 h. After cooling to room temperature, the reaction mixture was added into ammonia solution (200 mL, 25%–28% aqueous solution), and the water phase was extracted with DCM (2×60 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under vacuum. The residue was dissolved in DCM, and precipitated by adding the solution into the stirring Et_2O (200 mL). The solid was collected by filtration to afford the arylthianthrenium salts **1** without further purification.

3. Optimization of Reaction Conditions of the Aminocarbonylation of 1a with Aliphatic Amines

3.1 Evaluation of Pd Catalysts[a]

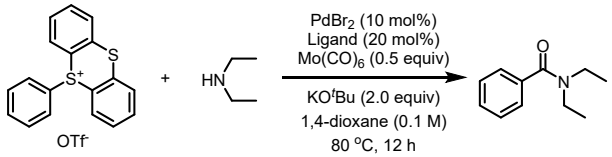


Entry	[Pd]	Yield of **4a**[b] (%)
1	$Pd(OAc)_2$	23
2	$PdCl_2$	20
3	$Pd(dppf)_2Cl_2$	28
4	$Pd(dba)_2$	31
5	$Pd(PPh_3)_4$	18
6	$Pd(PPh_3)_2Cl_2$	31

7	$Pd(acac)_2$	32
8	$PdBr_2$	33

[a]The reaction was performed using **1a** (0.2 mmol), **2a** (0.2 mmol). [b]Determined by crude ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as internal standard.

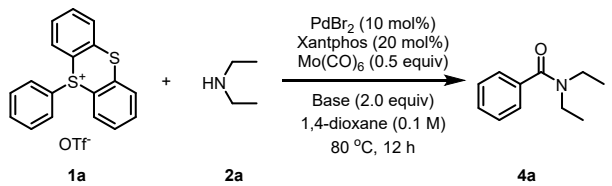
3.2 Evaluation of Ligands[a]



Entry	Ligand	Yield of **4a**[b] (%)
1	PPh_3	33
2	PCy_3	32
3	$(o\text{-}CH_3OC_6H_4)_3P$	28
4	$(p\text{-}CH_3OC_6H_4)_3P$	33
5	$(p\text{-}CF_3OC_6H_4)_3P$	36
6	$(o\text{-}CH_3C_6H_4)_3P$	37
7	$(p\text{-}CH_3C_6H_4)_3P$	30
8	BINAP	46
9	$BuPAd_2$	31
10	Xantphos	60

[a]The reaction was performed using **1a** (0.2 mmol), **2a** (0.2 mmol). [b]Determined by crude ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as internal standard.

3.3 Evaluation of Bases[a]

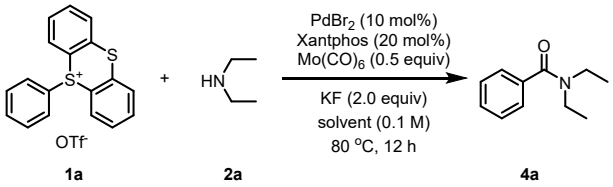


Entry	Base	Yield of **4a**[b] (%)

1	KO^{t}Bu	60
2	Cs_2CO_3	79
3	CsOAc	66
4	CsOPiv	63
5	K_2CO_3	80
6	NaO^{t}Bu	19
7	CsF	85
8	KF	89

[a]The reaction was performed using **1a** (0.2 mmol), **2a** (0.2 mmol). [b]Determined by crude ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as internal standard.

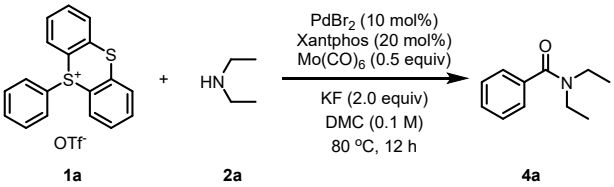
3.4 Evaluation of Solvents[a]



Entry	Solvent	Yield of **4a**[b] (%)
1	1,4-dioxane	89
2	Toluene	72
3	CH_3CN	73
4	DCE	67
5	DMC	90

[a]The reaction was performed using **1a** (0.2 mmol), **2a** (0.2 mmol). [b]Determined by crude ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as internal standard.

3.5 Evaluation of Loading of 1a[a]

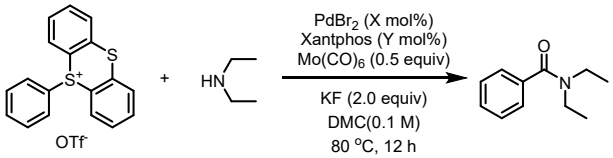


Entry	**1a** (equiv.)	Yield of **4a**[b] (%)

1	1.0	90
2	1.2	92
3	1.5	97 (94[c])
4	2.0	84

[a]The reaction was performed using **2a** (0.2 mmol). [b]Determined by crude ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as internal standard. [c]Isolated yield.

3.6. Evaluation of Loading of $PdBr_2$ and Xantphos[a]

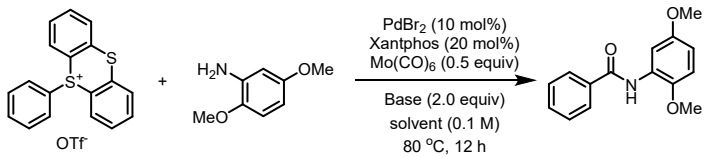


1a **2a** **4a**

Entry	$PdBr_2$ (mol%)	Xantphos (mol%)	Yield of **4a**[b] (%)
1	5.0	10	79
2	2.0	4.0	51
3[c]	2.0	4.0	80

[a]The reaction was performed using **1a** (0.3 mmol) and **2a** (0.2 mmol). [b]Determined by crude ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as internal standard. [c]The reaction was performed for 24 h.

4. Optimization of the Reaction Conditions of the Aminocarbonylation of 1a with Aromatic Amines[a]



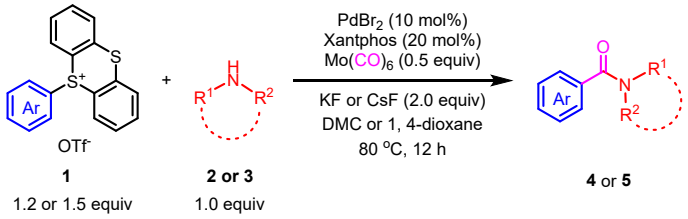
1a **3a** **5a**

Entry	Base	Solvent	Yield of **5a**[b] (%)
1	KO^{t}Bu	1,4-dioxane	53
2	NaO^{t}Bu	1,4-dioxane	15
3	K_2CO_3	1,4-dioxane	75
4	Cs_2CO_3	1,4-dioxane	80
5	CsOAc	1,4-dioxane	96

6	CsOPiv	1,4-dioxane	21
7	KF	1,4-dioxane	86
8	CsF	1,4-dioxane	98
10[c]	CsF	1,4-dioxane	99 (98[f])
11[d]	CsF	1,4-dioxane	82
12[e]	CsF	1,4-dioxane	74

[a]The reaction was performed using **1a** (0.2 mmol), **3a** (0.2 mmol). [b]Determined by crude ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as internal standard. [c]1.2 equiv. of **1a**. [d]1.5 equiv. of **1a**. [e]2.0 equiv. of **1a**. [f]Isolated yield.

5. General Procedure for the Synthesis of Substituted Aryl Aminocarboxamide Compounds by Aminocarbonylation of Aryl Thianthrenium Salts



General procedure 1:

To a mixture of corresponding aryl thianthrenium salts **1** (0.3 mmol, 1.5 equiv.), $PdBr_2$ (10 mol%), $Mo(CO)_6$ (0.5 equiv.), Xantphos (20 mol%), KF (2.0 equiv.), and corresponding aliphatic amines **2** (0.2 mmol, 1.0 equiv.) in a sealed tube equipped with a magnetic stir bar was added DMC (dimethyl carbonate 2.0 mL). The reaction mixture was then stirred at the rate of 1000 rpm at 80 °C for 12 h. The reaction was monitored by TLC until it was over. After cooling to room temperature, the reaction mixture was passed through a pad of Celite with EA as the eluent to remove any insoluble precipitate. The resulting solution was concentrated, and the residual mixture was purified by silica gel chromatography (petroleum ether/EA=150:1 to 5:1) to afford the corresponding products **4**.

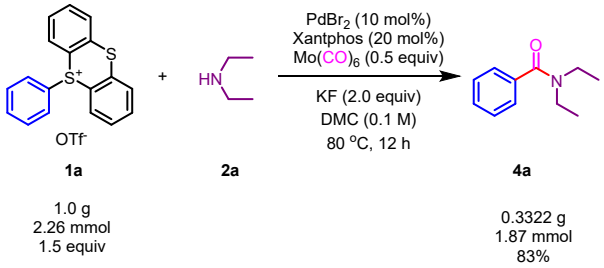
General procedure 2:

To a mixture of corresponding aryl thianthrenium salts **1** (0.24 mmol, 1.2 equiv.), $PdBr_2$ (10 mol%), $Mo(CO)_6$ (0.5 equiv.), Xantphos (20 mol%), CsF (2.0 equiv.), and corresponding aromatic amines **3** (0.2 mmol, 1.0 equiv.) in a sealed tube equipped with

a magnetic stir bar was added 1,4-dioxane (2.0 mL). The reaction mixture was then stirred at the rate of 1000 rpm at 80 °C for 12 h. The reaction was monitored by TLC until it was over. After cooling to room temperature, the reaction mixture was passed through a pad of Celite with EA as the eluent to remove any insoluble precipitate. The resulting solution was concentrated, and the residual mixture was purified by silica gel chromatography (petroleum ether/EA=150:1 to 1:1) to afford the corresponding products **5**.

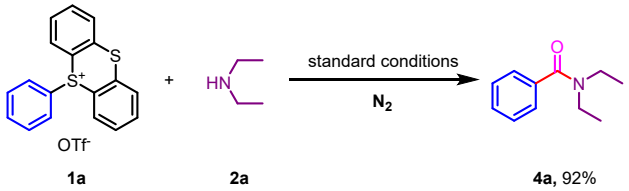
6. Gram-Scale and Control Experiments

6.1 Gram-Scale Experiment



To a mixture of phenyl thianthrenium salt **1a** (2.26 mmol, 1.0 g, 1.5 equiv.), $PdBr_2$ (39.9 mg, 10 mol%), $Mo(CO)_6$ (198.0 mg, 0.5 equiv.), Xantphos (173.6 mg, 20 mol%), KF (174.3 mg, 2.0 equiv.), and diethylamine (1.5 mmol, 109.7 mg, 1.0 equiv.) in a sealed tube equipped with a magnetic stir bar was added DMC (dimethyl carbonate, 15 mL). The reaction mixture was then stirred at the rate of 1000 rpm at 80 °C for 12 h. The reaction was monitored by TLC until it was over. After cooling to room temperature, the reaction mixture was passed through a pad of Celite with EA as the eluent to remove any insoluble precipitate. The resulting solution was concentrated, and the residual mixture was purified by silica gel chromatography (petroleum ether/EA=150:1 to 5:1) to afford the corresponding product *N*,*N*-diethylbenzamide **4a** (332.2 mg, 83% yield).

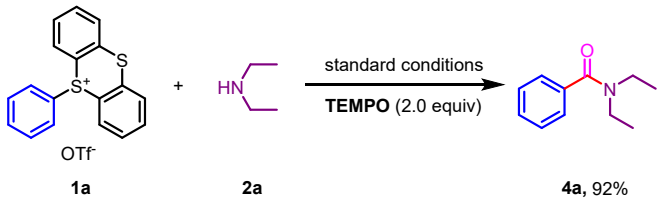
6.2 The Reaction under a N_2 Atmosphere

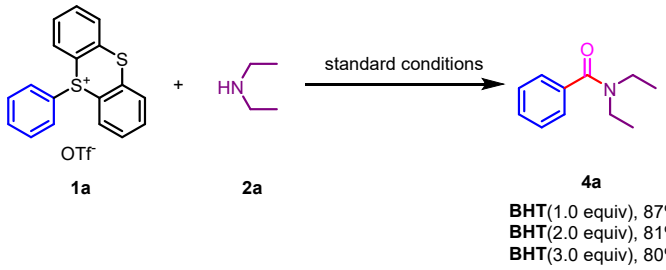


To a mixture of phenyl thianthrenium salt **1a** (0.3 mmol, 1.5 equiv.), $PdBr_2$ (10

mol%), $Mo(CO)_6$ (0.5 equiv.), Xantphos (20 mol%), and KF (2.0 equiv.) in a sealed tube equipped with a magnetic stir bar. The reaction tube was backfilled with N_2 three times. DMC (dimethyl carbonate, 2.0 mL) and diethylamine (0.2 mmol, 1.0 equiv.) were added under a nitrogen atmosphere. The reaction mixture was then stirred at the rate of 1000 rpm at 80 °C for 12 h. The reaction was monitored by TLC until it was over. After cooling to room temperature, the reaction mixture was passed through a pad of Celite with EA as the eluent to remove any insoluble precipitate. The resulting solution was concentrated, and the residual mixture was purified by silica gel chromatography (petroleum ether/EA=150:1 to 5:1) to afford the corresponding product *N*,*N*-diethylbenzamide **4a** in 92% yield.

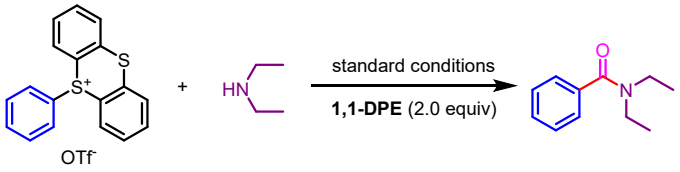
6.3 Radical Inhibition Experiments





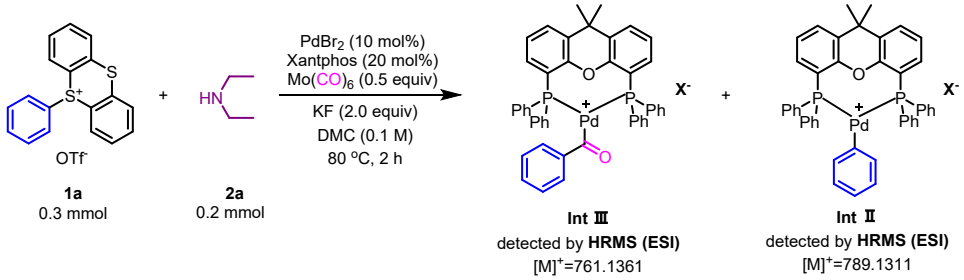
To a mixture of phenyl thianthrenium salt **1a** (0.3 mmol, 1.5 equiv.), $PdBr_2$ (10 mol%), $Mo(CO)_6$ (0.5 equiv.), Xantphos (20 mol%), KF (2.0 equiv.), diethylamine (0.2 mmol, 1.0 equiv.) and TEMPO (2.0 equiv.) or BHT (butylated hydroxytoluene, 1.0 to 3.0 equiv.) in a sealed tube equipped with a magnetic stir bar was added DMC (dimethyl carbonate, 2.0 mL). The reaction mixture was then stirred at the rate of 1000 rpm at 80 °C for 12 h. The reaction was monitored by TLC until it was over. After cooling to room temperature, the reaction mixture was passed through a pad of Celite with EA as the eluent to remove any insoluble precipitate. The resulting solution was concentrated, and the residual mixture was purified by silica gel chromatography (petroleum ether/EA=150:1 to 5:1) to afford the corresponding product *N*,*N*-diethylbenzamide **4a** in 80% to 92% yields.

6.4 Radical Capture Experiment



To a mixture of phenyl thianthrenium salt **1a** (0.3 mmol, 1.5 equiv.), $PdBr_2$ (10 mol%), $Mo(CO)_6$ (0.5 equiv.), Xantphos (20 mol%), KF (2.0 equiv.), diethylamine (0.2 mmol, 1.0 equiv.) and 1,1-DPE (1,1-diphenylethylene, 2.0 equiv.) in a sealed tube equipped with a magnetic stir bar was added DMC (dimethyl carbonate, 2.0 mL). The reaction mixture was then stirred at the rate of 1000 rpm at 80 °C for 12 h. Until the reaction was over (monitored by TLC), after being allowed to cool to room temperature, the reaction mixture was passed through a pad of Celite with EA as the eluent to remove any insoluble precipitate. The resulting solution was concentrated, and the residual mixture was purified by silica gel chromatography (petroleum ether/EA=150:1 to 5:1) to afford the corresponding product *N*,*N*-diethylbenzamide **4a** in 90% yield.

6.5 Identification of Pd complex by ESI-HRMS.



To a mixture of phenyl thianthrenium salt **1a** (0.3 mmol, 1.5 equiv.), $PdBr_2$ (10 mol%), $Mo(CO)_6$ (0.5 equiv.), Xantphos (20 mol%), KF (2.0 equiv.), and diethylamine **2a** (0.2 mmol, 1.0 equiv.) in a sealed tube equipped with a magnetic stir bar was added DMC (2.0 mL). After the reaction mixture was stirred at the rate of 1000 rpm at 80 °C for 1 h, 2 h, and 12 h, respectively, 1 drop of the reaction solution was sampled using a dropper, diluted with methanol, filtered through a PTFE syringe filter (0.45 μm), and analyzed by ESI-HRMS. Ion peaks corresponding to **Int II**, **Int III**, Xantphos oxide, and Xantphos dioxide were detected in the mass spectra. In contrast, no signal for $Mo(CO)_5DMC$ was detected in the three samples, indicating that this species was not formed under these conditions.

Int II: ESI-HRMS (*m/z*): calcd. for $C_{45}H_{37}OP_2Pd^+$ $[M]^+$ 761.1349, found 761.1361.

Int III: ESI-HRMS (*m/z*): calcd. for $C_{46}H_{37}O_2P_2Pd^+$ $[M]^+$ 789.1298, found 789.1311.

Xantphos oxide: ESI-HRMS (*m/z*): calcd. for $C_{39}H_{33}O_2P_2^+$ $[M+H]^+$ 595.1950, found 595.1964.

Xantphos dioxide: ESI-HRMS (*m/z*): calcd. for $C_{39}H_{33}O_3P_2^+$ $[M+H]^+$ 611.1899, found 611.1893.

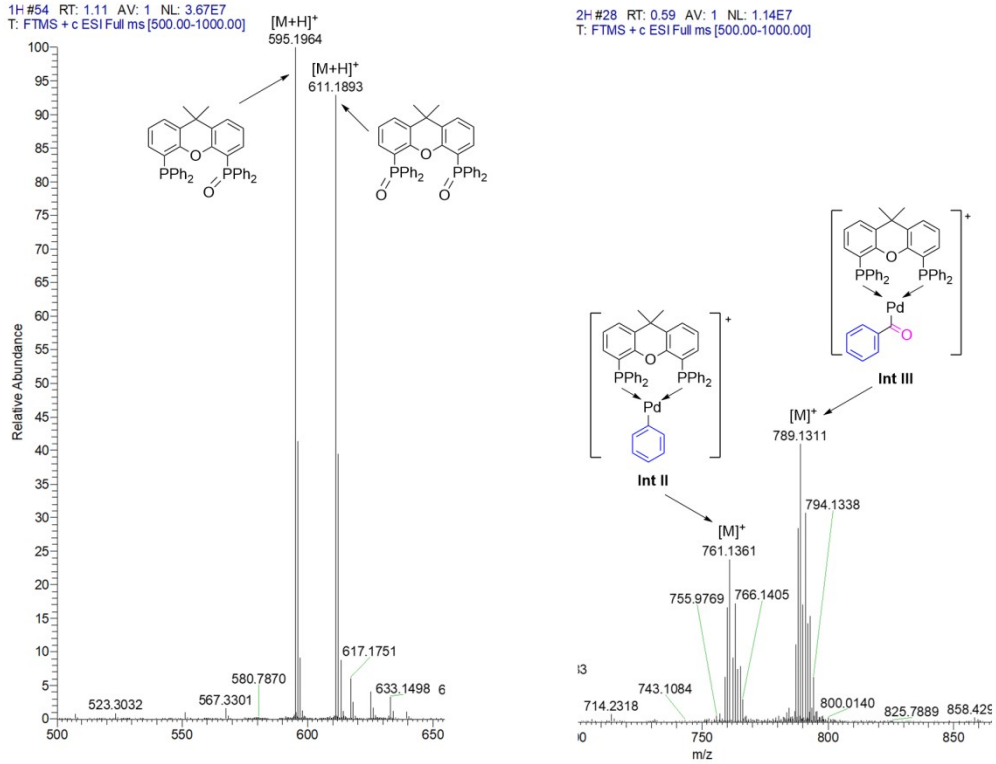
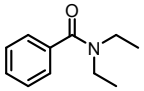


Fig. 1 The mass spectra of the reaction mixtures for 1 h and 2 h.

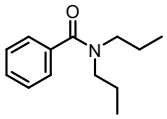
7. Characterization of Products

N,N-Diethylbenzamide (4a)



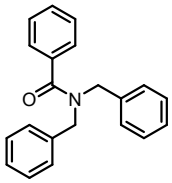
Colourless oil. 94% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45 – 7.33 (m, 5H), 3.56 (q, *J* = 6.0 Hz, 2H), 3.25 (q, *J* = 5.9 Hz, 2H), 1.25 (t, *J* = 3.4 Hz, 3H), 1.10 (t, *J* = 7.8 Hz, 3H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.3, 137.3, 129.1, 128.4, 126.3, 43.3, 39.2, 14.2, 12.9. **ESI-MS** (m/z): calcd. for $C_{11}H_{16}NO$ $[M+H]^+$ 178.1226, found 178.1228.

***N,N*-Dipropylbenzamide (4b)**



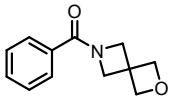
Colourless oil. 85% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 – 7.32 (m, 5H), 3.46 (t, *J* = 7.7 Hz, 2H), 3.16 (t, *J* = 7.5 Hz, 2H), 1.76-1.62 (m, 2H), 1.45-1.58 (m, 2H), 0.98 (t, *J* = 7.4 Hz, 3H), 0.74 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.8, 137.4, 129.0, 128.3, 126.4, 50.7, 46.3, 21.9, 20.7, 11.5, 11.0. **ESI-MS** (m/z): calcd. for $C_{13}H_{20}NO$ $[M+H]^+$ 206.1539, found 206.1543.

***N,N*-Dibenzylbenzamide (4c)**



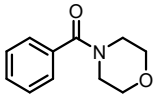
Colourless oil. 84% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.52 – 7.47 (m, 2H), 7.40 – 7.33 (m, 7H), 7.30 (t, *J* = 7.3 Hz, 4H), 7.14 (d, *J* = 7.4 Hz, 2H), 4.70 (s, 2H), 4.40 (s, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 172.3, 137.0, 136.4, 136.2, 129.7, 128.9, 128.8, 128.6, 128.5, 127.7, 127.6, 127.1, 126.8, 51.6, 46.8. **ESI-MS** (m/z): calcd. for $C_{21}H_{20}NO$ $[M+H]^+$ 302.1539, found 302.1540.

Phenyl(2-oxa-6-azaspiro[3.3]heptan-6-yl)methanone (4d)



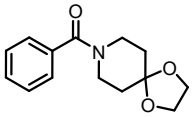
Colourless oil. 76% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.63 – 7.59 (m, 2H), 7.51 – 7.45 (m, 1H), 7.42 (dd, *J* = 8.4, 6.9 Hz, 2H), 4.84 (d, *J* = 6.9 Hz, 2H), 4.78 (d, *J* = 7.0 Hz, 2H), 4.44 (s, 2H), 4.35 (s, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 170.5, 132.8, 131.3, 128.6, 128.5, 128.0, 127.9, 80.9, 62.9, 58.2, 38.4. **ESI-MS** (m/z): calcd. for $C_{12}H_{14}NO_2$ $[M+H]^+$ 204.1019, found 204.1022.

Morpholino(phenyl)methanone (4e)



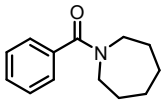
White solid. 82% yield. m.p.= 62–64 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.42 (dt, *J* = 7.8, 3.8 Hz, 5H), 3.84 – 3.44 (m, 8H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 170.5, 135.3, 129.9, 128.6, 127.1, 66.9, 48.2, 29.7. **ESI-MS** (m/z): calcd. for $C_{11}H_{14}NO_2$ $[M+H]^+$ 192.1019, found 192.1023.

Phenyl(1,4-dioxa-8-azaspiro[4.5]decan-8-yl)methanone (4f)



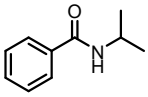
Pale-yellow oil. 85% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 (m, 5H), 3.99 (d, *J* = 3.8 Hz, 4H), 3.86 (s, 2H), 3.49 (s, 2H), 1.81 (s, 2H), 1.65 (s, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.4, 136.0, 129.6, 128.5, 126.8, 107.0, 64.5, 45.7, 40.3, 35.7, 34.8. **ESI-MS** (m/z): calcd. for $C_{14}H_{18}NO_3$ $[M+H]^+$ 248.1281, found 248.1280.

Azepan-1-yl(phenyl)methanone (4g)



Pale-yellow oil. 92% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 5H), 3.70 – 3.62 (t, *J* = 5.6 Hz, 2H), 3.35 (t, *J* = 5.6 Hz, 2H), 1.8 8-1.80 (m, 2H), 1.68-1.54 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.7, 137.3, 129.2, 128.5, 126.5, 49.9, 46.4, 29.6, 27.9, 27.3, 26.5. **ESI-MS** (m/z): calcd. for $C_{13}H_{18}NO$ $[M+H]^+$ 204.1383, found 204.1385.

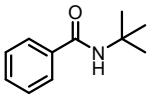
***N*-Isopropylbenzamide (4h)**



Pale-yellow solid. 87% yield. m.p=83-85°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 – 7.71 (m, 2H), 7.49 – 7.43 (m, 1H), 7.42 – 7.36 (m, 2H), 6.11 (s, 1H), 4.27 (m, 6.5 Hz, 1H), 1.24 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.8, 135.0, 131.3, 128.6,

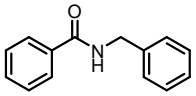
126.9, 42.0, 22.9. **ESI-MS** (m/z): calcd. for $C_{10}H_{14}NO$ $[M+H]^+$ 164.1070, found 164.1071.

***N*-(*tert*-Butyl)benzamide (4i)**



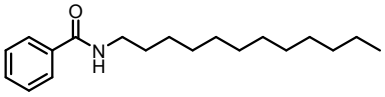
White solid. 92% yield. m.p. = 170–171°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.69 (m, 2H), 7.48 – 7.43 (m, 1H), 7.42 – 7.36 (m, 2H), 5.99 (s, 1H), 1.46 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.7, 135.7, 130.8, 128.2, 126.5, 51.4, 28.6. **ESI-MS** (m/z): calcd. for $C_{11}H_{16}NO$ $[M+H]^+$ 178.1226, found 178.1229.

***N*-Benzylbenzamide (4j)**



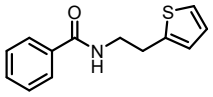
White solid. 84% yield. m.p. = 101−104°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 – 7.77 (m, 2H), 7.53 – 7.47 (m, 1H), 7.42 (ddt, *J* = 8.3, 6.5, 1.4 Hz, 2H), 7.37 – 7.27 (m, 5H), 6.51 (s, 1H), 4.64 (d, *J* = 5.7 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.5, 138.3, 134.5, 131.7, 128.9, 128.7, 128.0, 127.8, 127.1, 44.2. **ESI-MS** (m/z): calcd. for $C_{14}H_{14}NO$ $[M+H]^+$ 212.1070, found 212.1068.

***N*-Dodecylbenzamide (4k)**



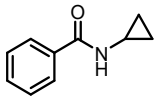
White solid. 62% yield. m.p. = 61–62°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 – 7.73 (m, 2H), 7.49 – 7.43 (m, 1H), 7.39 (dd, *J* = 8.2, 6.6 Hz, 2H), 6.41 (d, *J* = 5.8 Hz, 1H), 3.41 (q, *J* = 7.3, 5.8 Hz, 2H), 1.64-1.55 (m, 2H), 1.38 – 1.19 (m, 18H), 0.87 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 167.7, 135.0, 131.3, 128.5, 127.0, 40.2, 32.0, 29.8, 29.7, 29.7, 29.7, 29.6, 29.4, 27.1, 22.8, 14.2. **ESI-MS** (m/z): calcd. for $C_{19}H_{32}NO$ $[M+H]^+$ 290.2478, found 290.2475.

***N*-(2-(Thiophen-2-yl)ethyl)benzamide (4l)**



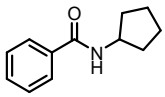
Pale-yellow oil. 94% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 – 7.70 (m, 2H), 7.51 – 7.45 (dt, 1H), 7.39 (dd, *J* = 8.2, 6.7 Hz, 2H), 7.16 (dd, *J* = 5.2, 1.2 Hz, 1H), 6.95 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.85 (dd, *J* = 3.4, 1.2 Hz, 1H), 6.69 (s, 1H), 3.70 (q, *J* = 6.4 Hz, 2H), 3.14 (t, *J* = 6.7 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.8, 141.3, 134.4, 131.6, 128.6, 127.2, 127.0, 125.5, 124.1, 41.5, 29.9. **ESI-MS** (m/z): calcd. for $C_{13}H_{14}NOS$ $[M+H]^+$ 232.0791, found 232.0795.

***N*-Cyclopropylbenzamide (4m)**



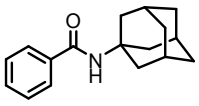
Colourless oil. 86% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 – 7.70 (m, 2H), 7.49 – 7.43 (m, 1H), 7.41 – 7.35 (m, 2H), 6.52 (s, 1H), 2.92-2.85 (m, 1H), 0.83 (t, *J* = 6.1 Hz, 2H), 0.64 – 0.58 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.1, 134.5, 131.6, 128.6, 127.0, 23.2, 6.8. **ESI-MS** (m/z): calcd. for $C_{10}H_{12}NO$ $[M+H]^+$ 162.0913, found 162.0915.

***N*-Cyclopentylbenzamide (4n)**



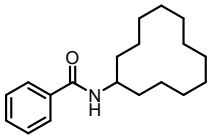
Colorless oil. 94% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.77 – 7.70 (m, 2H), 7.51 – 7.46 (m, 1H), 7.42 (dd, *J* = 8.4, 6.9 Hz, 2H), 6.07 (s, 1H), 4.46-4.35 (m, 1H), 2.15-2.05 (m, 2H), 1.77-1.70 (m, 2H), 1.68 – 1.62 (m, 2H), 1.54 – 1.45 (m, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 167.3, 135.0, 131.4, 128.7, 126.9, 51.8, 33.4, 23.9. **ESI-MS** (m/z): calcd. for $C_{12}H_{16}NO$ $[M+H]^+$ 190.1226, found 190.1230.

***N*-((3s,5s,7s)-Adamantan-1-yl)benzamide (4o)**



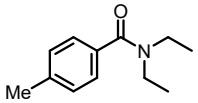
White solid. 88% yield. m.p.=138–139℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.69 (m, 2H), 7.48 – 7.43 (m, 1H), 7.43 – 7.37 (m, 2H), 5.82 (s, 1H), 2.12 (s, 9H), 1.72 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.8, 136.1, 131.2, 128.6, 126.8, 52.4, 41.8, 36.5, 36.3, 29.7, 29.6, 29.4. **ESI-MS** (m/z): calcd. for $C_{17}H_{22}NO$ $[M+H]^+$ 256.1696, found 256.1693.

***N*-Cyclododecylbenzamide (4p)**



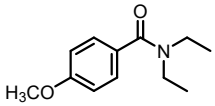
White solid. 86% yield. m.p. = 181–184℃. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.77 – 7.73 (m, 2H), 7.50 – 7.47 (m, 1H), 7.42 (dd, *J* = 8.3, 6.9 Hz, 2H), 5.91 (d, *J* = 8.6 Hz, 1H), 4.32-4.25 (m, 1H), 1.75-1.68 (m, 2H), 1.56 – 1.30 (m, 20H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 166.9, 135.1, 131.4, 128.7, 126.9, 46.7, 30.4, 24.1, 23.8, 23.6, 23.4, 21.6. **ESI-MS** (m/z): calcd. for $C_{19}H_{30}NO$ $[M+H]^+$ 288.2322, found 288.2325.

***N,N*-Diethyl-4-methylbenzamide (4q)**



Colourless oil. 86% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.26 (d, *J* = 8.1 Hz, 2H), 7.19 (d, *J* = 7.8 Hz, 2H), 3.53 (s, 2H), 3.26 (s, 2H), 2.37 (s, 3H), 1.24 – 1.06 (m, 6H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 171.6, 139.2, 134.5, 129.1, 126.5, 43.4, 39.3, 21.5. **ESI-MS** (m/z): calcd. for $C_{12}H_{18}NO$ $[M+H]^+$ 192.1383, found 192.1384.

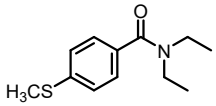
***N,N*-Diethyl-4-methoxybenzamide (4r)**



Colourless oil. 92% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.36 – 7.32 (m, 2H), 6.92 –

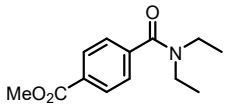
6.87 (m, 2H), 3.82 (s, 3H), 3.51 (s, 2H), 3.31 (s, 2H), 1.19 (s, 6H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 171.4, 160.4, 129.6, 128.3, 113.8, 55.4, 43.6, 39.5, 14.4, 13.1. **ESI-MS** (m/z): calcd. for $C_{12}H_{18}NO_2$ $[M+H]^+$ 208.1332, found 208.1336.

***N,N*-Diethyl-4-(methylthio)benzamide (4s)**



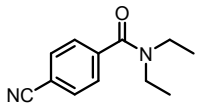
Brown oil. 80% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.4 Hz, 2H), 7.26 – 7.23 (m, 2H), 3.53 (s, 2H), 3.27 (s, 2H), 2.49 (s, 3H), 1.24 – 1.08 (m, 6H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 171.1, 140.3, 133.7, 127.1, 126.0, 43.5, 39.5, 15.6, 14.4, 13.0. **ESI-MS** (m/z): calcd. for $C_{12}H_{18}NOS$ $[M+H]^+$ 224.1104, found 224.1109.

Methyl 4-(diethylcarbamoyl)benzoate (4t)



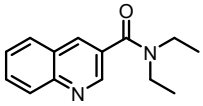
Colourless oil. 85% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.10 – 8.05 (m, 2H), 7.46 – 7.41 (m, 2H), 3.93 (s, 3H), 3.56 (q, *J* = 7.1 Hz, 2H), 3.21 (q, *J* = 7.1 Hz, 2H), 1.26 (t, *J* = 7.3 Hz, 3H), 1.09 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 170.4, 166.6, 141.7, 130.8, 130.0, 126.4, 52.5, 43.4, 39.4, 14.3, 13.0. **ESI-MS** (m/z): calcd. for $C_{13}H_{18}NO_3$ $[M+H]^+$ 236.1281, found 236.1286.

4-Cyano-*N,N*-diethylbenzamide (4u)



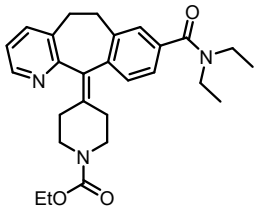
Colourless oil. 67% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.72 – 7.69 (m, 2H), 7.49 – 7.45 (m, 2H), 3.55 (q, *J* = 7.1 Hz, 2H), 3.20 (q, *J* = 7.0 Hz, 2H), 1.26 (t, *J* = 7.3 Hz, 3H), 1.11 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 169.3, 141.7, 132.6, 127.2, 118.4, 113.1, 43.4, 39.6, 14.4, 13.0. **ESI-MS** (m/z): calcd. for $C_{12}H_{15}N_2O$ $[M+H]^+$ 203.1179, found 203.1183.

***N,N*-Diethylquinoline-3-carboxamide (4v)**



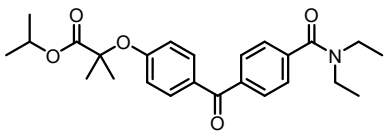
Yellow oil. 62% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.93 (d, *J* = 2.2 Hz, 1H), 8.20 (d, *J* = 2.1 Hz, 1H), 8.13 (d, *J* = 8.5 Hz, 1H), 7.85 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.77 (ddd, *J* = 8.5, 6.8, 1.5 Hz, 1H), 7.60 (ddd, *J* = 8.1, 6.7, 1.2 Hz, 1H), 3.62 (d, *J* = 7.6 Hz, 2H), 3.33 (q, *J* = 7.3 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.17 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 168.8, 148.2, 148.1, 134.2, 130.6, 130.2, 129.5, 128.3, 127.5, 127.2, 43.7, 39.8, 14.5, 13.0. **ESI-MS** (m/z): calcd. for $C_{14}H_{17}N_2O$ $[M+H]^+$ 229.1335, found 229.1340.

Ethyl 4-(3-(diethylcarbamoyl)-5,6-dihydro-11*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridin-11-ylidene)piperidine-1-carboxylate (4w)



Colourless oil. 65% yield. **^{1}H NMR** (400 MHz, CDCl3) δ 8.39 (dd, J = 4.7, 1.7 Hz, 1H), 7.43 (dd, J = 7.7, 1.7 Hz, 1H), 7.19 – 7.15 (m, 2H), 7.10 (m, 2H), 4.11 (q, J = 7.1 Hz, 2H), 3.80 (s, 2H), 3.50 (s, 2H), 3.44 – 3.29 (m, 2H), 3.23 (m, 2H), 3.09 (dt, J = 13.1, 6.6 Hz, 2H), 2.83 (m, 2H), 2.45 (m, 1H), 2.39 – 2.24 (m, 3H), 1.23 (t, J = 7.1 Hz, 6H), 1.12 – 1.04 (m, 3H). **^{13}C NMR** (100 MHz, CDCl3) δ 171.2, 157.3, 155.6, 146.7, 140.1, 138.2, 137.6, 137.5, 136.4, 134.8, 133.7, 129.2, 127.4, 123.8, 122.4, 61.4, 44.9, 44.9, 43.3, 39.2, 31.9, 31.6, 30.8, 30.6, 14.8, 14.4, 12.9. **ESI-MS** (m/z): calcd. for $C_{27}H_{34}N_3O_3$ $[M+H]^+$ 448.2595, found 448.2590.

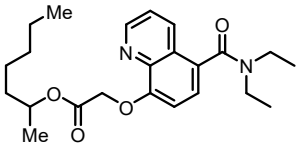
Isopropyl 2-(4-(4-(diethylcarbamoyl)benzoyl)phenoxy)-2-methylpropanoate (4x)



Colourless oil. 59% yield. **^{1}H NMR** (400 MHz, CDCl3) δ 7.75 (dd, J = 8.5, 3.3 Hz,

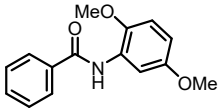
4H), 7.45 (d, J = 8.0 Hz, 2H), 6.88 – 6.82 (m, 2H), 5.07 (hept, J = 6.2 Hz, 1H), 3.55 (q, J = 7.2 Hz, 2H), 3.24 (q, J = 7.0 Hz, 2H), 1.64 (s, 6H), 1.27 – 1.23 (t, J = 7.1 Hz, 3H), 1.19 (d, J = 6.3 Hz, 6H), 1.10 (t, J = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, CDCl3) δ 195.0, 173.2, 170.4, 159.9, 140.6, 138.8, 132.2, 130.2, 129.9, 126.2, 117.3, 79.5, 69.4, 43.4, 39.4, 25.4, 21.6, 14.3, 13.0. **ESI-MS** (m/z): calcd. for $C_{25}H_{32}NO_5$ $[M+H]^+$ 426.2275, found 426.2275.

Heptan-2-yl 2-((5-(diethylcarbamoyl)quinolin-8-yl)oxy)acetate (4y)



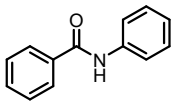
Yellow oil. 49% yield. **^{1}H NMR** (400 MHz, CDCl3) δ 8.95 (dd, J = 4.1, 1.7 Hz, 1H), 8.12 (dd, J = 8.5, 1.8 Hz, 1H), 7.46 (dd, J = 8.5, 4.1 Hz, 1H), 7.34 (d, J = 7.9 Hz, 1H), 6.91 (d, J = 7.9 Hz, 1H), 5.06 – 4.98 (m, 1H), 4.93 (s, 2H), 3.64 (s, 2H), 3.10 (q, J = 7.1 Hz, 2H), 1.60 – 1.51 (m, 1H), 1.48 – 1.40 (m, 1H), 1.32 (t, J = 7.1 Hz, 3H), 1.21 (t, J = 6.2 Hz, 9H), 0.97 (t, J = 7.1 Hz, 3H), 0.85 – 0.80 (t, J = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, CDCl3) δ 169.1, 168.3, 154.3, 149.9, 140.0, 133.4, 128.3, 126.6, 124.0, 122.5, 108.5, 72.8, 66.4, 43.3, 39.3, 35.8, 31.6, 25.0, 22.6, 20.0, 14.4, 14.0, 13.2. **ESI-MS** (m/z): calcd. for $C_{23}H_{33}N_2O_4$ $[M+H]^+$ 401.2435, found 401.2436.

***N*-(2,5-Dimethoxyphenyl)benzamide (5a)**



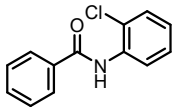
Colourless oil. 98% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.60 (s, 1H), 8.29 (d, *J* = 3.0 Hz, 1H), 7.92 – 7.87 (m, 2H), 7.58 – 7.47 (m, 3H), 6.83 (d, *J* = 8.9 Hz, 1H), 6.62 (dd, *J* = 8.9, 3.1 Hz, 1H), 3.88 (s, 3H), 3.82 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.4, 154.0, 142.4, 135.3, 131.9, 128.9, 128.5, 127.1, 110.8, 109.0, 106.0, 56.4, 55.9. **ESI-MS** (m/z): calcd. for $C_{15}H_{16}NO_3$ $[M+H]^+$ 258.1125, found 258.1122.

***N*-Phenylbenzamide (5b)**



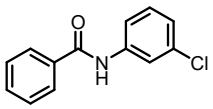
Off-white solid. 89% yield. m.p. = 164–165°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 – 7.83 (m, 3H), 7.67 – 7.62 (m, 2H), 7.55 (td, *J* = 7.3, 1.4 Hz, 1H), 7.52 – 7.45 (m, 2H), 7.37 (td, *J* = 7.9, 1.5 Hz, 2H), 7.16 (td, *J* = 7.5, 1.1 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.9, 138.0, 135.1, 132.0, 129.2, 129.0, 127.2, 124.7, 120.3. **ESI-MS** (m/z): calcd. for $C_{13}H_{12}NO$ $[M+H]^+$ 198.0913, found 198.0914.

***N*-(2-Chlorophenyl)benzamide (5c)**



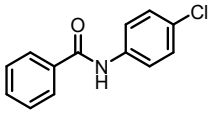
White solid. 94% yield. m.p. = 91–92°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.57 (dd, *J* = 8.3, 1.6 Hz, 1H), 8.46 (s, 1H), 7.95 – 7.90 (m, 2H), 7.61 – 7.56 (m, 1H), 7.52 (tt, *J* = 6.7, 1.8 Hz, 2H), 7.42 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.34 (ddd, *J* = 8.6, 7.6, 1.5 Hz, 1H), 7.09 (td, *J* = 7.8, 1.6 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.4, 134.8, 134.7, 132.3, 129.2, 129.1, 128.0, 127.2, 124.9, 123.2, 121.6. **ESI-MS** (m/z): calcd. for $C_{13}H_{11}ClNO$ $[M+H]^+$ 232.0524, found 232.0523.

***N*-(3-Chlorophenyl)benzamide(5d)**



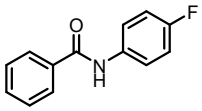
White solid. 98% yield. m.p. = 116–118°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.86 – 7.81 (m, 2H), 7.76 (t, *J* = 2.1 Hz, 1H), 7.57 – 7.52 (m, 1H), 7.50 – 7.43 (m, 3H), 7.26 (t, *J* = 8.1 Hz, 1H), 7.11 (ddd, *J* = 8.0, 2.0, 1.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.0, 139.2, 134.8, 134.6, 132.2, 130.2, 129.0, 127.2, 124.7, 120.5, 118.3. **ESI-MS** (m/z): calcd. for $C_{13}H_{11}ClNO$ $[M+H]^+$ 232.0524, found 232.0523.

***N*-(4-Chlorophenyl)benzamide (5e)**



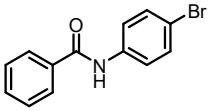
Pale-yellow solid. 71% yield. m.p. = 166–167°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.13 (s, 1H), 7.90 – 7.84 (m, 2H), 7.80 (d, *J* = 8.4 Hz, 2H), 7.64 (d, *J* = 8.7 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.1, 142.2, 134.2, 133.5, 132.6, 129.1, 127.3, 120.1, 119.0, 107.5. **ESI-MS** (m/z): calcd. for $C_{13}H_{11}ClNO$ $[M+H]^+$ 232.0524, found 232.0524.

***N*-(4-Fluorophenyl)benzamide (5f)**



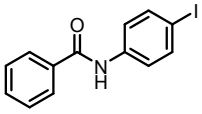
White solid. 90% yield. m.p. = 180–183°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 7.2, 1.8 Hz, 2H), 7.84 – 7.78 (m, 1H), 7.63 – 7.53 (m, 3H), 7.49 (dd, *J* = 8.2, 6.5 Hz, 2H), 7.11 – 7.03 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.8, 160.9 (d, *J* = 242.8 Hz), 134.9, 134.0, 132.1, 129.0, 127.1, 122.2 (d, *J* = 7.9 Hz), 115.9 (d, *J* = 22.5 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -117.59. **ESI-MS** (m/z): calcd. for $C_{13}H_{11}FNO$ $[M+H]^+$ 216.0819, found 216.0816.

***N*-(4-Bromophenyl)benzamide (5g)**



Off-white solid. 81% yield. m.p. = 206–208°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 – 7.84 (m, 2H), 7.81 (s, 1H), 7.60 – 7.53 (m, 3H), 7.53 – 7.46 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.8, 137.1, 134.8, 132.2, 132.2, 129.0, 127.1, 121.8, 117.3. **ESI-MS** (m/z): calcd. for $C_{13}H_{11}BrNO$ $[M+H]^+$ 276.0019, found 276.0015.

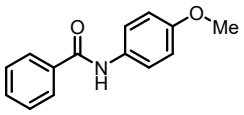
***N*-(4-Iodophenyl)benzamide (5h)**



White solid. 27% yield. m.p. = 221–223°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 – 7.83

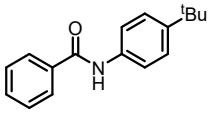
(m, 2H), 7.78 (s, 1H), 7.70 – 7.65 (m, 2H), 7.60 – 7.54 (m, 1H), 7.50 (ddt, J = 8.4, 6.6, 1.7 Hz, 2H), 7.47 – 7.42 (m, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 165.8, 138.2, 137.9, 134.8, 132.2, 129.0, 127.1, 122.1, 87.9. **ESI-MS** (m/z): calcd. for $C_{13}H_{11}INO$ $[M+H]^+$ 323.9880, found 323.9874.

***N*-(4-Methoxyphenyl)benzamide (5i)**



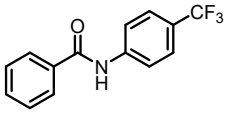
White solid. 97% yield. m.p. = 152–155°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (d, J = 6.9 Hz, 3H), 7.53 (dd, J = 8.0, 5.5 Hz, 3H), 7.46 (dd, J = 8.2, 6.6 Hz, 2H), 6.92 – 6.86 (m, 2H), 3.81 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.8, 156.7, 135.1, 131.8, 131.1, 128.9, 127.1, 122.3, 114.4, 55.6. **ESI-MS** (m/z): calcd. for $C_{14}H_{14}NO_2$ $[M+H]^+$ 228.1019, found 228.1022.

***N*-(4-(Tert-butyl)phenyl)benzamide (5j)**



White solid. 88% yield. m.p. = 139–142°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.87 – 7.82 (m, 2H), 7.60 – 7.55 (m, 2H), 7.54 – 7.49 (m, 1H), 7.46 – 7.40 (m, 2H), 7.39 – 7.34 (m, 2H), 1.33 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.0, 147.6, 135.4, 135.2, 131.8, 128.8, 127.2, 126.0, 120.3, 34.5, 31.5. **ESI-MS** (m/z): calcd. for $C_{17}H_{20}NO$ $[M+H]^+$ 254.1539, found 254.1539.

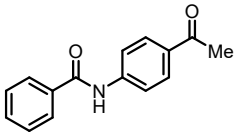
***N*-(4-(Trifluoromethyl)phenyl)benzamide (5k)**



Off-white solid. 78% yield. m.p. = 202–205°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.91 – 7.86 (m, 2H), 7.79 (d, J = 8.5 Hz, 2H), 7.64 (d, J = 8.5 Hz, 2H), 7.60 – 7.56 (m, 1H), 7.55 – 7.49 (m, 2H). **^{13}C NMR** (100 MHz, d_6-DMSO) δ 166.5, 143.3, 135.0, 132.3, 128.9, 128.3, 126.4 (q, J = 3.8 Hz), 124.9 (q, J = 270.0 Hz), 124.1 (q, J = 31.8

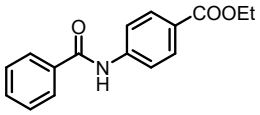
Hz), 120.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.11. **ESI-MS** (m/z): calcd. for $C_{14}H_{11}F_3NO$ $[M+H]^+$ 266.0787, found 266.0781.

***N*-(4-Acetylphenyl)Benzamide (5l)**



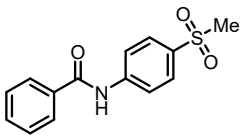
White solid. 84% yield. m.p. = 207–209°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 – 7.97 (m, 3H), 7.92 – 7.85 (m, 2H), 7.80 – 7.75 (m, 2H), 7.62 – 7.56 (m, 1H), 7.52 (dd, *J* = 8.3, 6.6 Hz, 2H), 2.60 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 197.1, 165.9, 142.4, 134.6, 133.3, 132.5, 130.0, 129.1, 127.2, 119.4, 26.6. **ESI-MS** (m/z): calcd. for $C_{15}H_{14}NO_2$ $[M+H]^+$ 240.1019, found 240.1021.

Ethyl 4-benzamidobenzoate (5m)



White solid. 92% yield. m.p. = 145–146°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.30 (s, 1H), 8.05 – 7.99 (m, 2H), 7.88 – 7.83 (m, 2H), 7.78 – 7.72 (m, 2H), 7.57 – 7.51 (m, 1H), 7.45 (dd, *J* = 8.3, 6.8 Hz, 2H), 4.35 (q, *J* = 7.1 Hz, 2H), 1.38 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.3, 166.1, 142.3, 134.6, 132.3, 130.9, 128.9, 127.2, 126.2, 119.4, 61.0, 14.5. **ESI-MS** (m/z): calcd. for $C_{16}H_{16}NO_3$ $[M+H]^+$ 270.1125, found 270.1127.

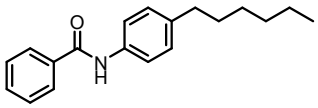
***N*-(4-(Methylsulfonyl)phenyl)benzamide (5n)**



White solid. 78% yield. m.p. = 189–191°C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.97 – 7.93 (m, 2H), 7.91 – 7.86 (m, 4H), 7.63 – 7.59 (m, 1H), 7.53 (t, *J* = 7.7 Hz, 2H), 3.06 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 166.0, 142.9, 135.7, 134.12, 132.7, 129.2,

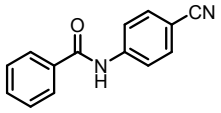
129.0, 127.3, 120.1, 44.9. **ESI-MS** (m/z): calcd. for $C_{14}H_{14}NO_3S$ $[M+H]^+$ 276.0689, found 276.0694.

***N*-(4-Hexylphenyl)benzamide (5o)**



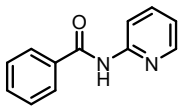
White solid. 67% yield. m.p. = 175–177°C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.89 – 7.84 (m, 2H), 7.80 (s, 1H), 7.54 (d, *J* = 7.8 Hz, 3H), 7.48 (dd, *J* = 8.4, 6.9 Hz, 2H), 7.20 – 7.15 (m, 2H), 2.59 (t, *J* = 7.7 Hz, 2H), 1.61 – 1.57 (m, 2H), 1.36 – 1.27 (m, 6H), 0.91-0.85 (m, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 165.8, 139.5, 135.6, 135.2, 131.9, 129.1, 128.9, 127.1, 120.3, 35.5, 31.9, 31.6, 29.0, 22.8, 14.3. **ESI-MS** (m/z): calcd. for $C_{19}H_{24}NO$ $[M+H]^+$ 282.1852, found 282.1854.

***N*-(4-Cyanophenyl)benzamide (5p)**



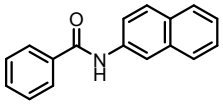
White solid. 57% yield. m.p. = 169–171°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 – 7.84 (m, 2H), 7.83 (s, 1H), 7.63 – 7.54 (m, 3H), 7.52 – 7.47 (m, 2H), 7.36 – 7.31 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.8, 136.6, 134.8, 132.2, 129.7, 129.3, 129.0, 127.1, 121.5. **ESI-MS** (m/z): calcd. for $C_{14}H_{11}N_2O$ $[M+H]^+$ 223.0866, found 223.0865.

***N*-(Pyridin-2-yl)benzamide (5q)**



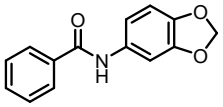
Yellow oil. 97% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.19 (s, 1H), 8.40 (dt, *J* = 8.4, 1.0 Hz, 1H), 8.13 (ddd, *J* = 5.0, 2.0, 1.0 Hz, 1H), 7.95 – 7.90 (m, 2H), 7.73 (ddd, *J* = 8.4, 7.4, 1.9 Hz, 1H), 7.57 – 7.52 (m, 1H), 7.49 – 7.43 (m, 2H), 7.02 (ddd, *J* = 7.4, 4.9, 1.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.1, 151.8, 147.9, 138.6, 134.5, 132.3, 128.9, 128.6, 127.4, 120.0, 114.4. **ESI-MS** (m/z): calcd. for $C_{12}H_{14}N_2O$ $[M+H]^+$ 199.0866, found 199.0865.

***N*-(Naphthalen-2-yl)benzamide (5r)**



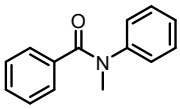
White solid. 81% yield. m.p. = 161–165°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.34 (d, *J* = 2.1 Hz, 1H), 8.15 (s, 1H), 7.93 – 7.88 (m, 2H), 7.84 – 7.77 (m, 3H), 7.60 (dd, *J* = 8.7, 2.2 Hz, 1H), 7.58 – 7.52 (m, 1H), 7.51 – 7.40 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.1, 135.5, 135.0, 134.0, 132.0, 130.9, 128.9, 128.9, 127.8, 127.7, 127.2, 126.7, 125.3, 120.3, 117.2. **ESI-MS** (m/z): calcd. for $C_{17}H_{14}NO$ $[M+H]^+$ 248.1070, found 248.1069.

***N*-(Benzo[*d*][1,3]dioxol-5-yl)benzamide (5s)**



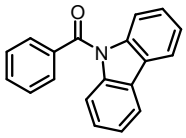
White solid. 91% yield. m.p. = 133–134°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.85 – 7.80 (m, 2H), 7.54 – 7.49 (m, 1H), 7.46–7.40 (m, 2H), 7.33 (d, *J* = 2.2 Hz, 1H), 6.90 (dd, *J* = 8.3, 2.1 Hz, 1H), 6.75 (d, *J* = 8.3 Hz, 1H), 5.95 (s, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.9, 147.9, 144.6, 134.9, 132.2, 131.9, 128.8, 127.1, 113.8, 108.2, 103.4, 101.4. **ESI-MS** (m/z): calcd. for $C_{14}H_{12}NO_3$ $[M+H]^+$ 242.0812, found 242.0808.

***N*-Methyl-*N*-phenylbenzamide (5t)**



Pale-yellow oil. 94% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 – 7.27 (m, 2H), 7.25 – 7.19 (m, 3H), 7.19 – 7.11 (m, 3H), 7.06 – 7.01 (m, 2H), 3.50 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.8, 145.0, 136.0, 129.7, 129.3, 128.8, 127.8, 127.0, 126.6, 38.5. **ESI-MS** (m/z): calcd. for $C_{14}H_{14}NO$ $[M+Na]^+$ 234.0889, found 234.0889.

(9*H*-carbazol-9-yl)(phenyl)methanone (5u)

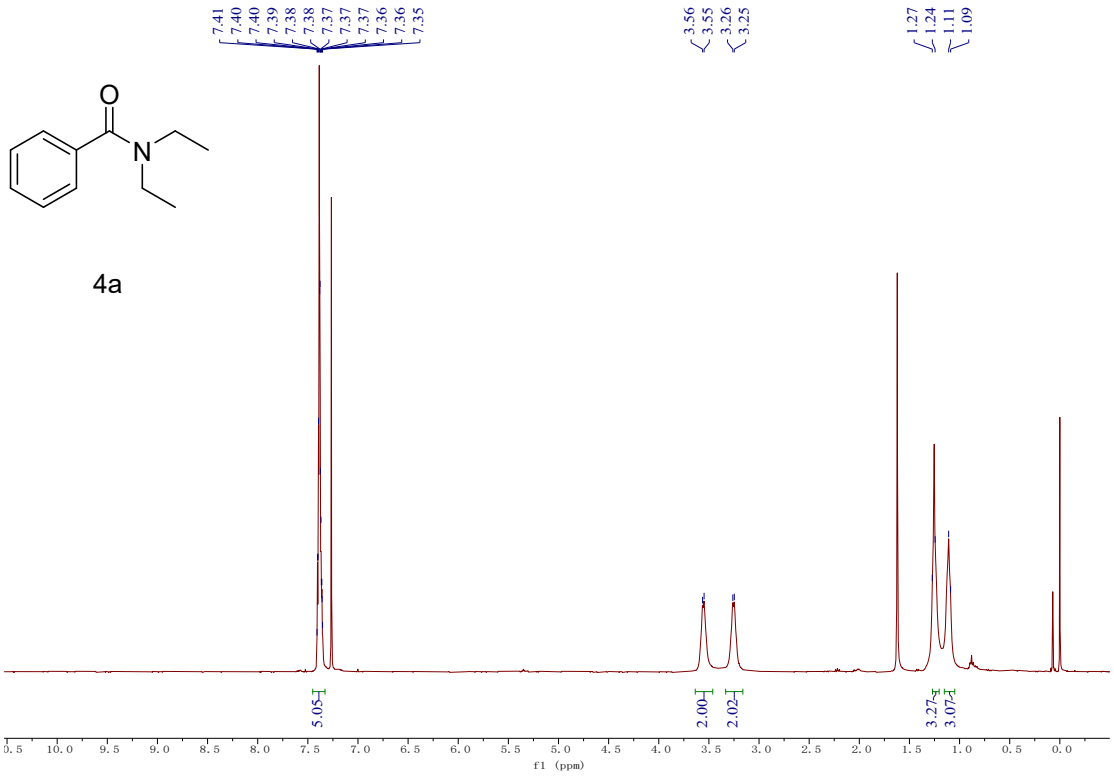


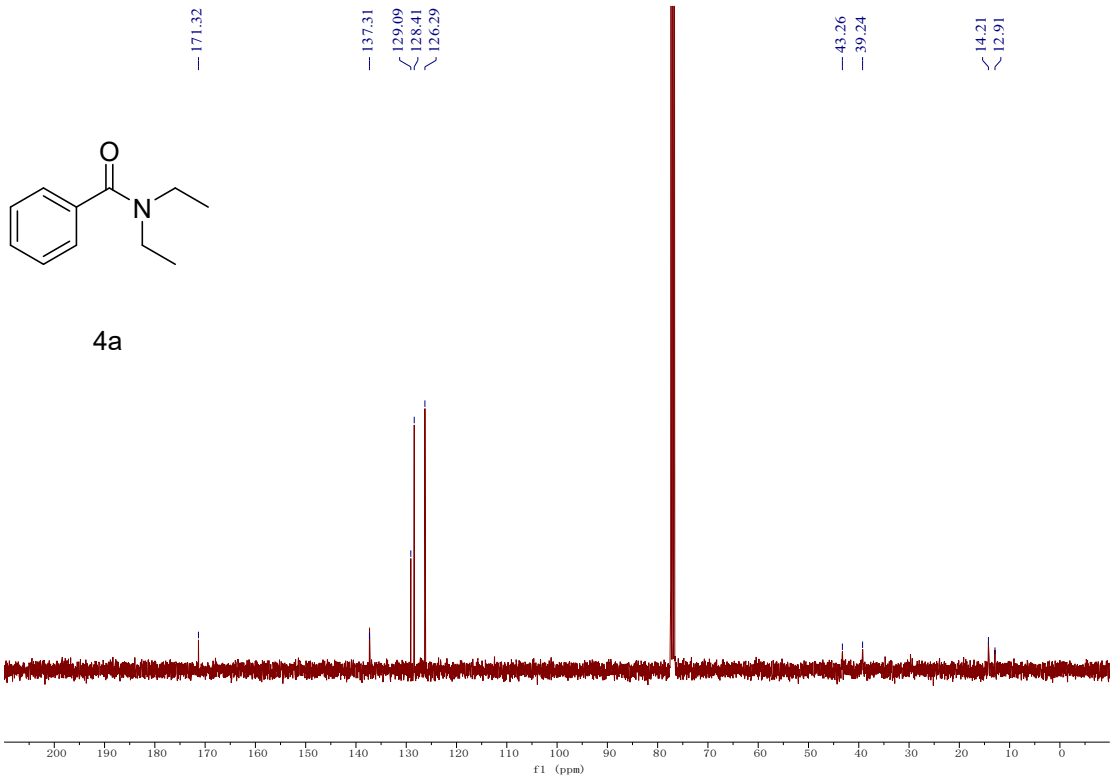
Off-white solid. 20% yield. m.p. = 100–101℃. **^{1}H NMR** (400 MHz, $CDCl_3$) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 7.98 (m, 2H), 7.74 – 7.70 (m, 2H), 7.68 – 7.63 (m, 1H), 7.56 – 7.49 (m, 4H), 7.34 – 7.28 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.8, 139.2, 135.8, 132.5, 129.2, 129.1, 129.0, 126.9, 126.1, 123.5, 120.0, 115.9. **ESI-MS** (m/z): calcd. for $C_{19}H_{13}NO$ $[M+H]^+$ 272. 1070, found 272.1068.

8. Reference

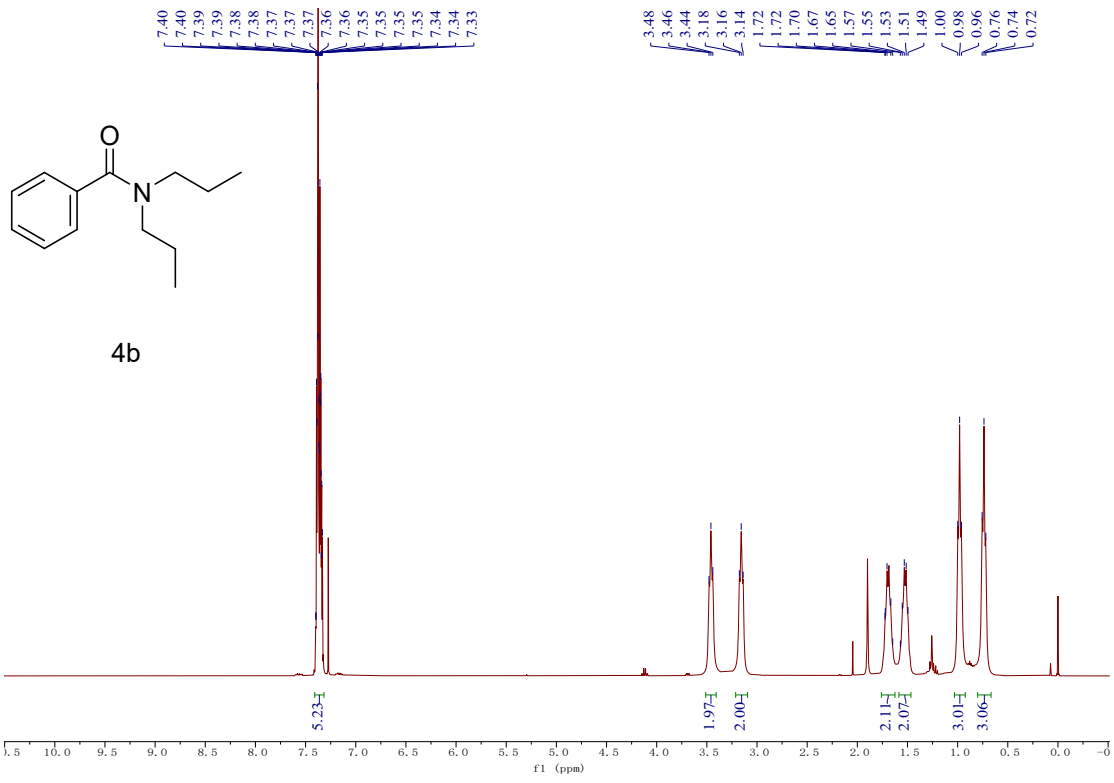
1. X. -Y. Chen, Y. -N. Li, Y. Wu, J. Bai, Y. Guo, P. Wang, *J. Am. Chem. Soc.,* 2023, **145**, 10431-10440.

9. NMR Spectra

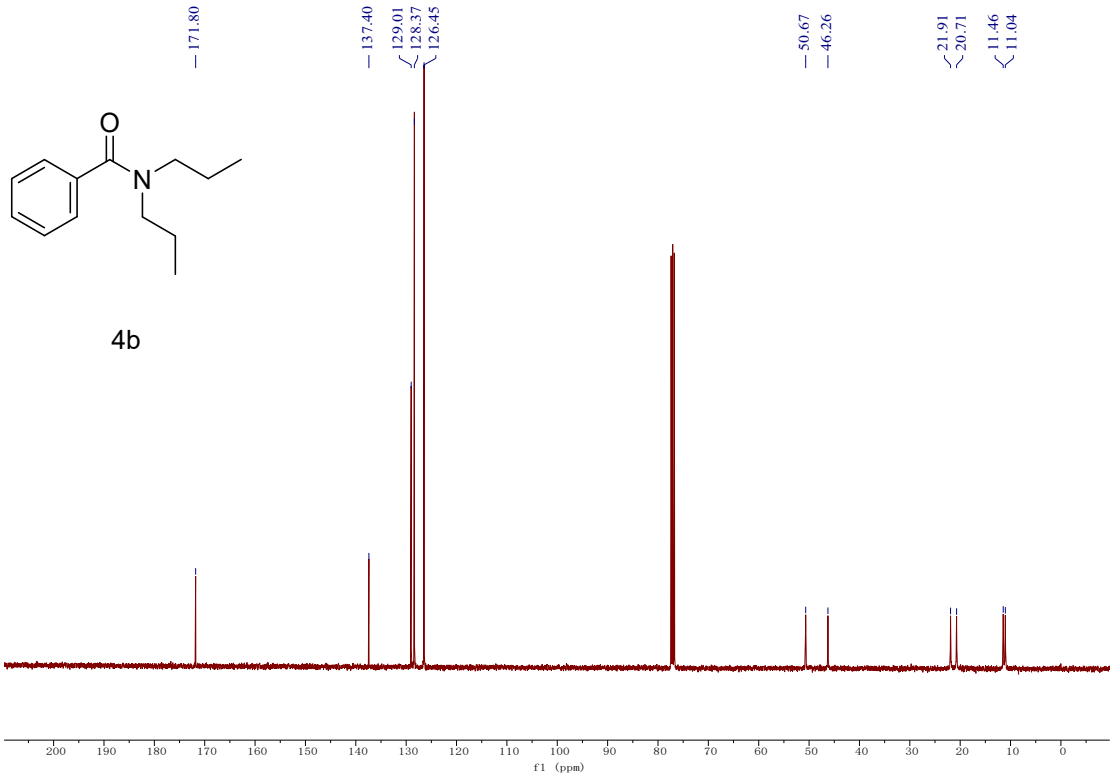


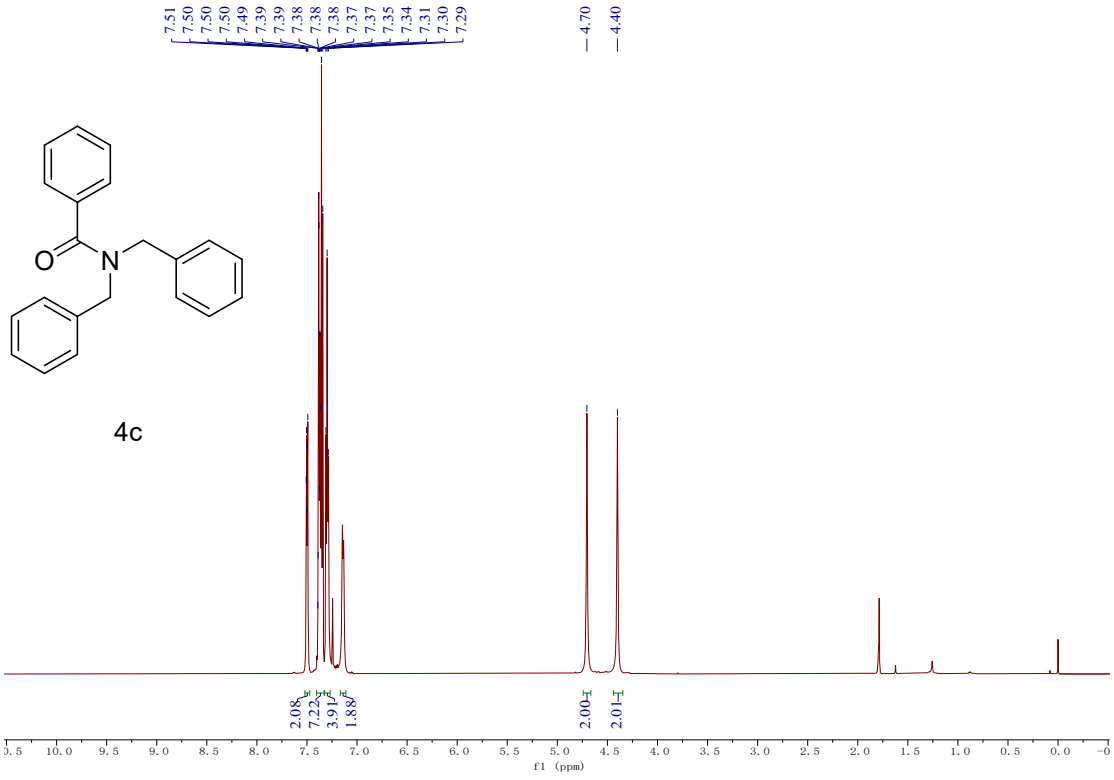

171.32
137.31
129.09
128.41
126.29
43.26
39.24
14.21
12.91
O
N
f1 (ppm)

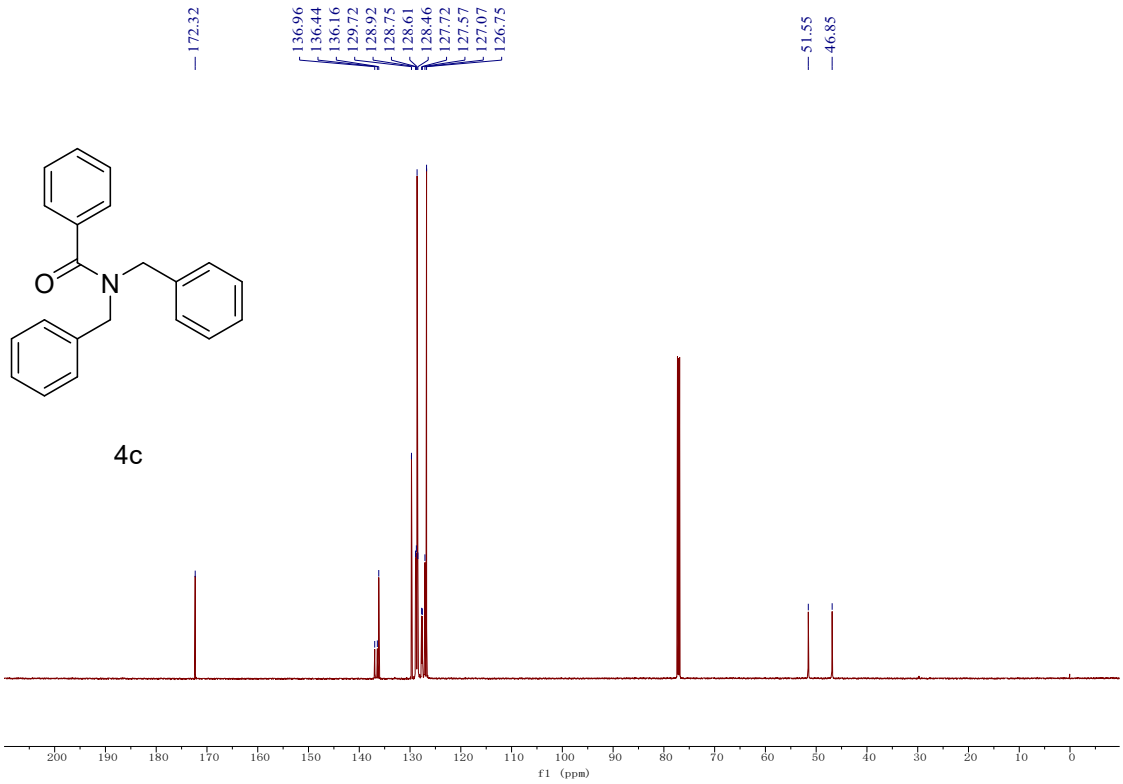
4a

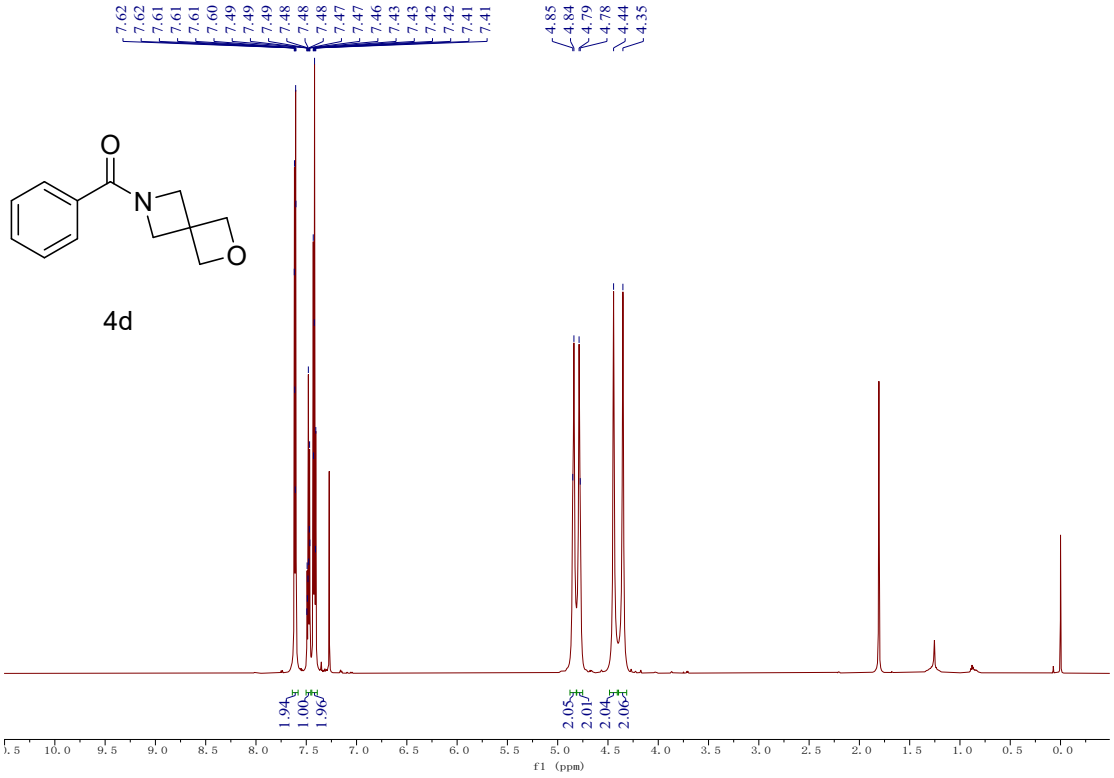

7.40
7.40
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.35
7.35
7.35
7.35
7.34
7.34
7.33
3.48
3.46
3.44
3.18
3.16
3.14
1.72
1.72
1.70
1.67
1.65
1.57
1.55
1.53
1.51
1.49
1.00
0.98
0.96
0.76
0.74
0.72
5.23
1.97
2.00
2.11
2.07
3.01
3.06
O
N
f1 (ppm)

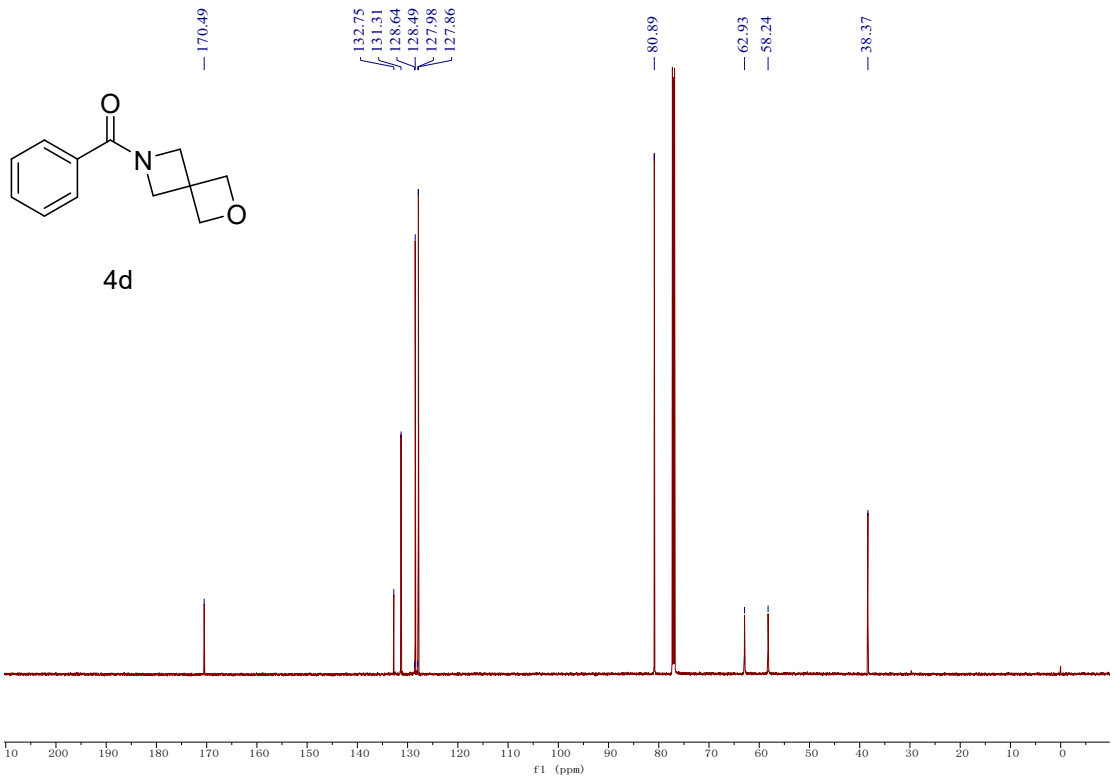
4b

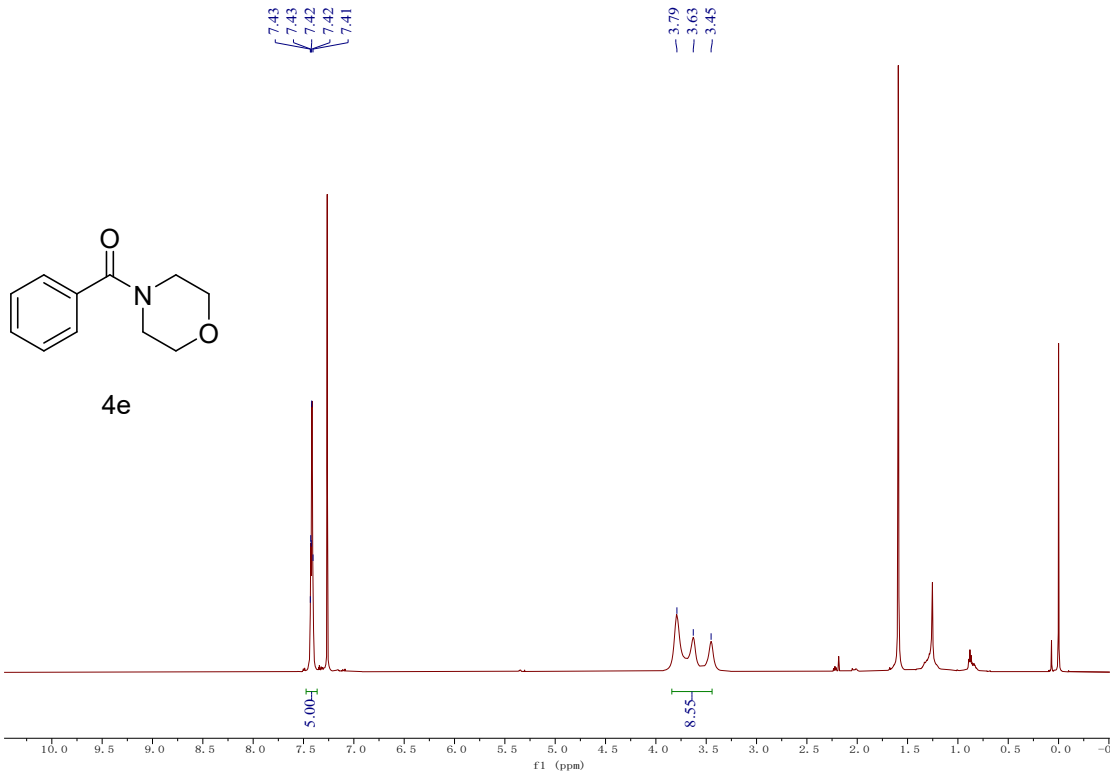

171.80
137.40
129.01
128.37
126.45
50.67
46.26
21.91
20.71
11.46
11.04
O
N
4b
f1 (ppm)

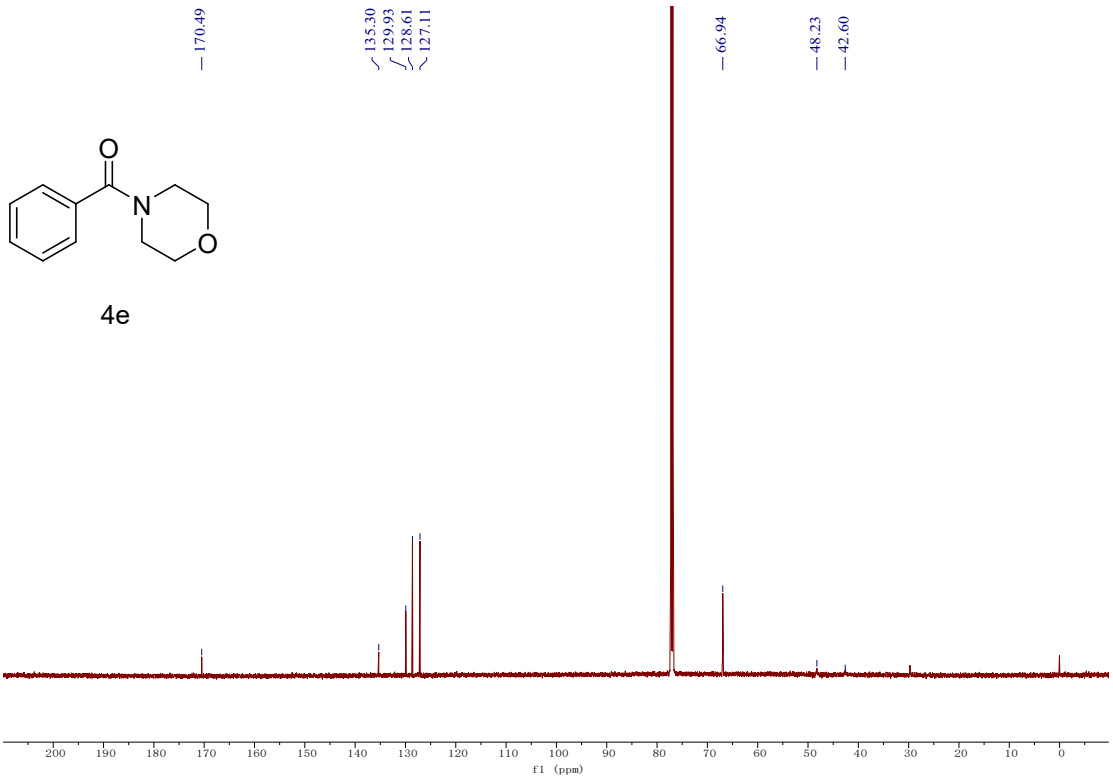

7.51
7.50
7.50
7.50
7.49
7.39
7.39
7.38
7.38
7.37
7.37
7.35
7.34
7.31
7.30
7.29
4.70
4.40
O
N
4c
2.08
7.22
3.91
1.88
2.00
2.01
f1 (ppm)


172.32
136.96
136.44
136.16
129.72
128.92
128.75
128.61
128.46
127.72
127.57
127.07
126.75
51.55
46.85
O
N
4c
f1 (ppm)

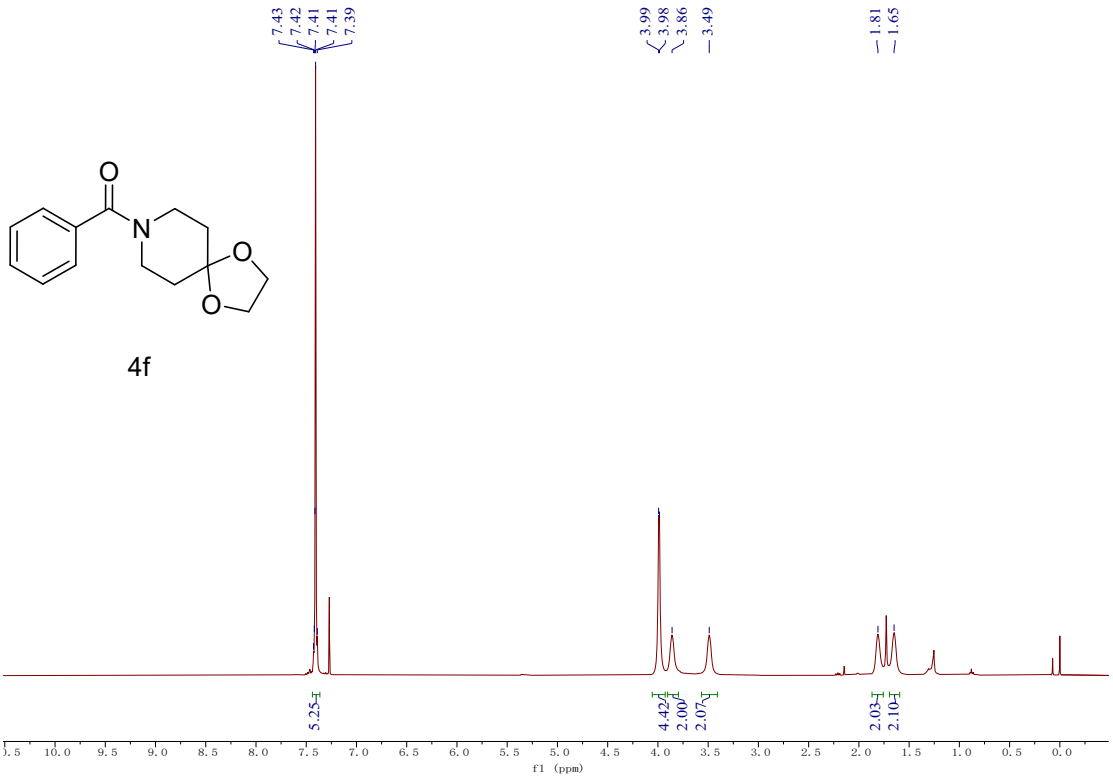

7.62
7.62
7.61
7.61
7.61
7.61
7.60
7.49
7.49
7.49
7.48
7.48
7.48
7.47
7.47
7.46
7.43
7.43
7.42
7.42
7.41
7.41
4.85
4.84
4.79
4.78
4.44
4.35
O
N
O
4d
1.94
1.00
1.96
2.05
2.01
2.04
2.06
f1 (ppm)


170.49
132.75
131.31
128.64
128.49
127.98
127.86
80.89
62.93
58.24
38.37
O
N
O
4d
f1 (ppm)

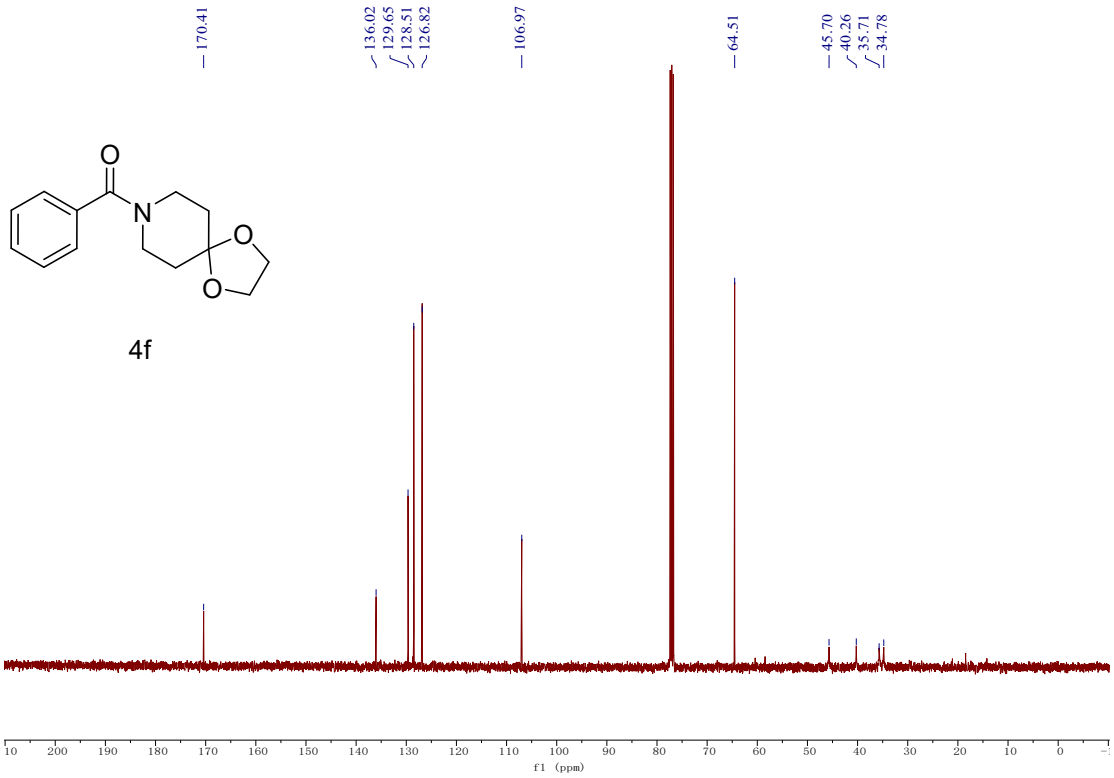

7.43
7.43
7.42
7.42
7.41
3.79
3.63
3.45
O
N
O
4e
5.00
8.55
f1 (ppm)

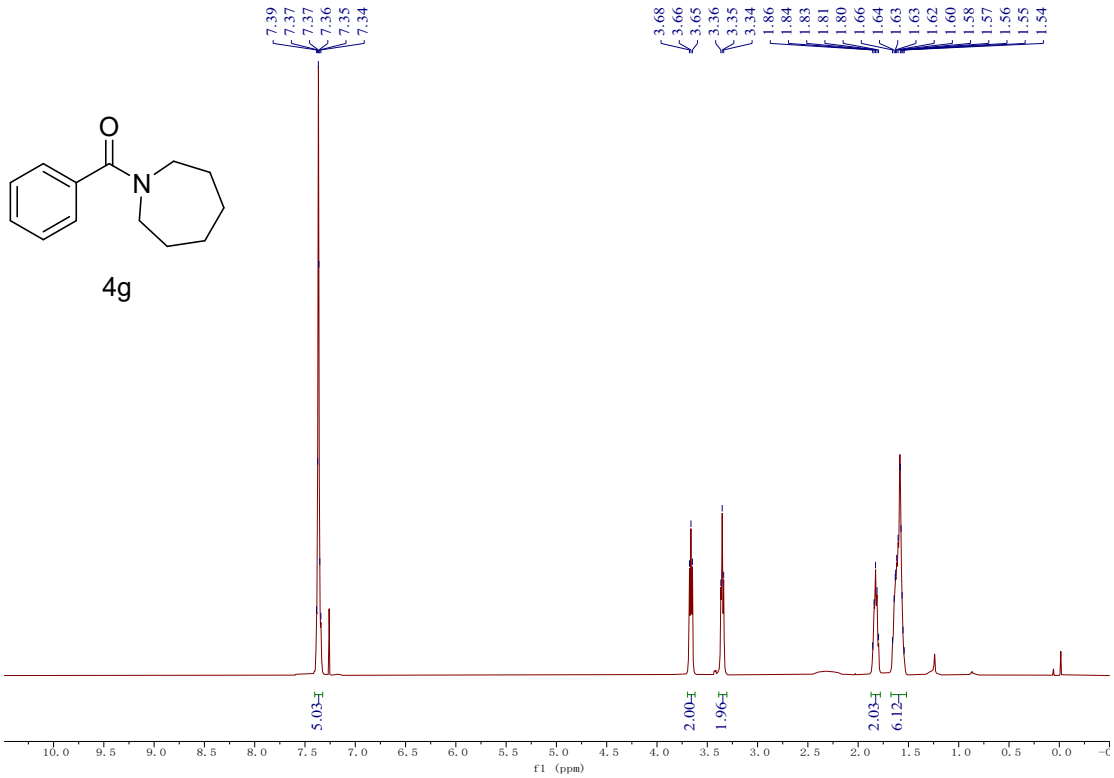

170.49
135.30
129.93
128.61
127.11
66.94
48.23
42.60
O
N
O
f1 (ppm)

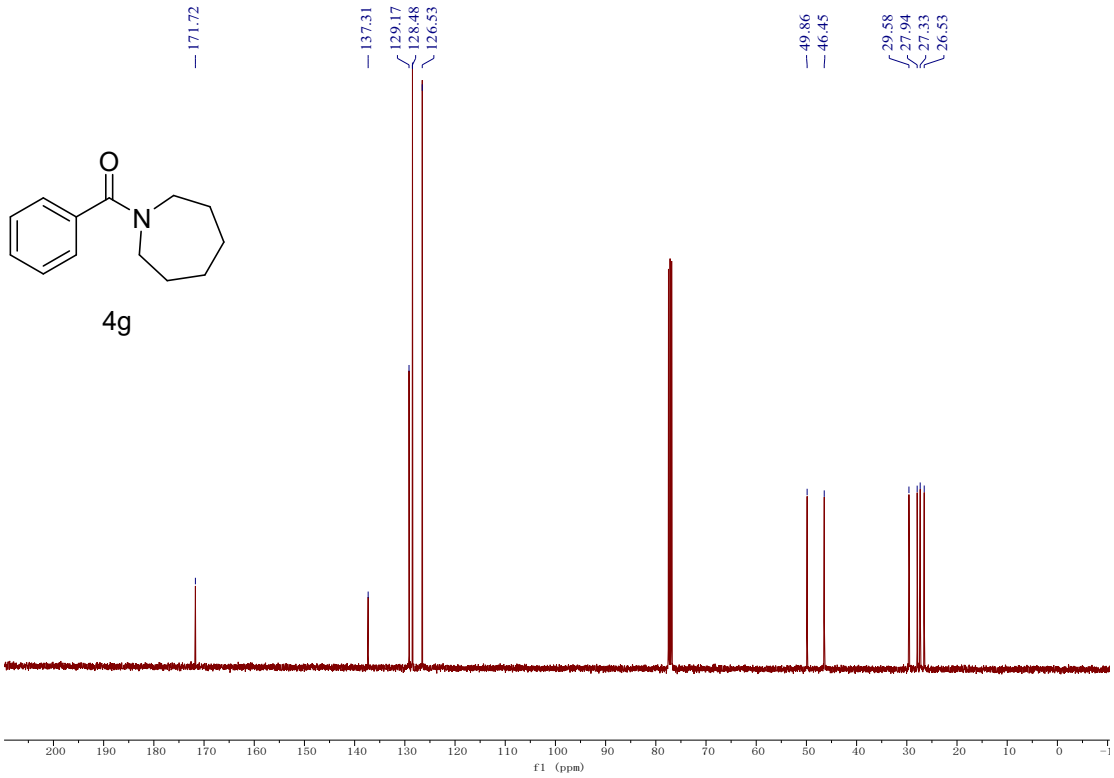
4e


7.43
7.42
7.41
7.41
7.39
3.99
3.98
3.86
3.49
1.81
1.65
O
N
O
O
5.25
4.42
2.00
2.07
2.03
2.10
f1 (ppm)

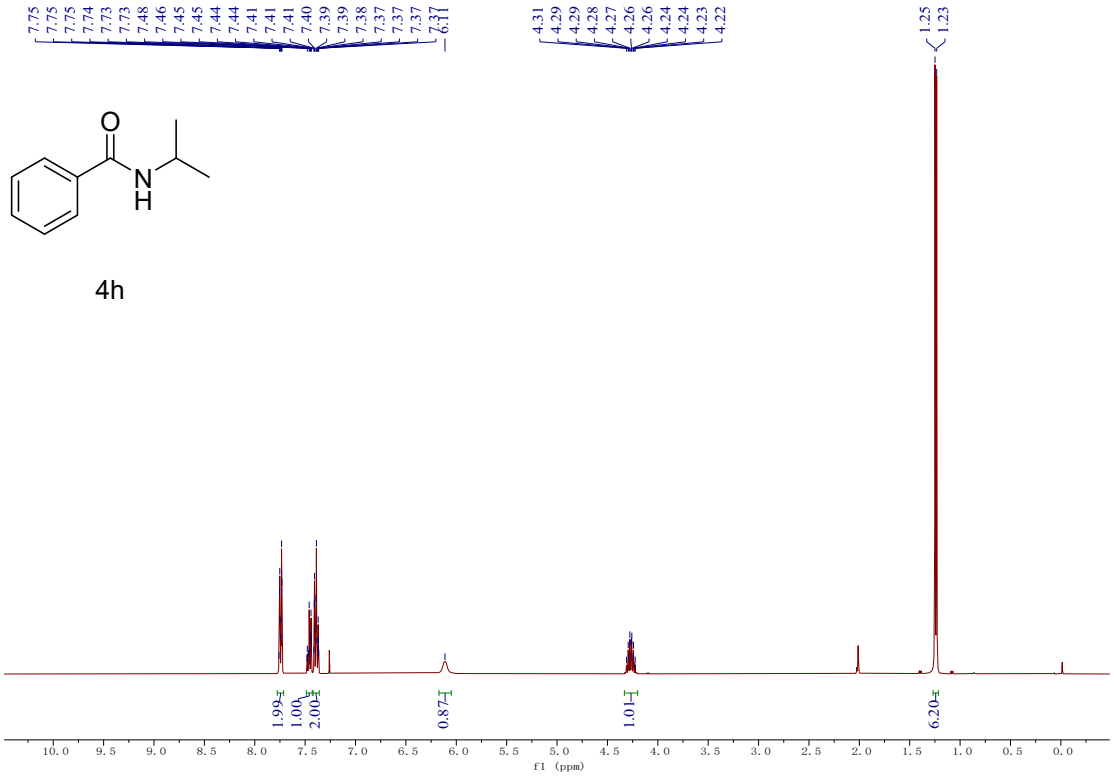
4f


170.41
136.02
129.65
128.51
126.82
106.97
64.51
45.70
40.26
35.71
34.78
O
N
O
O
4f
f1 (ppm)

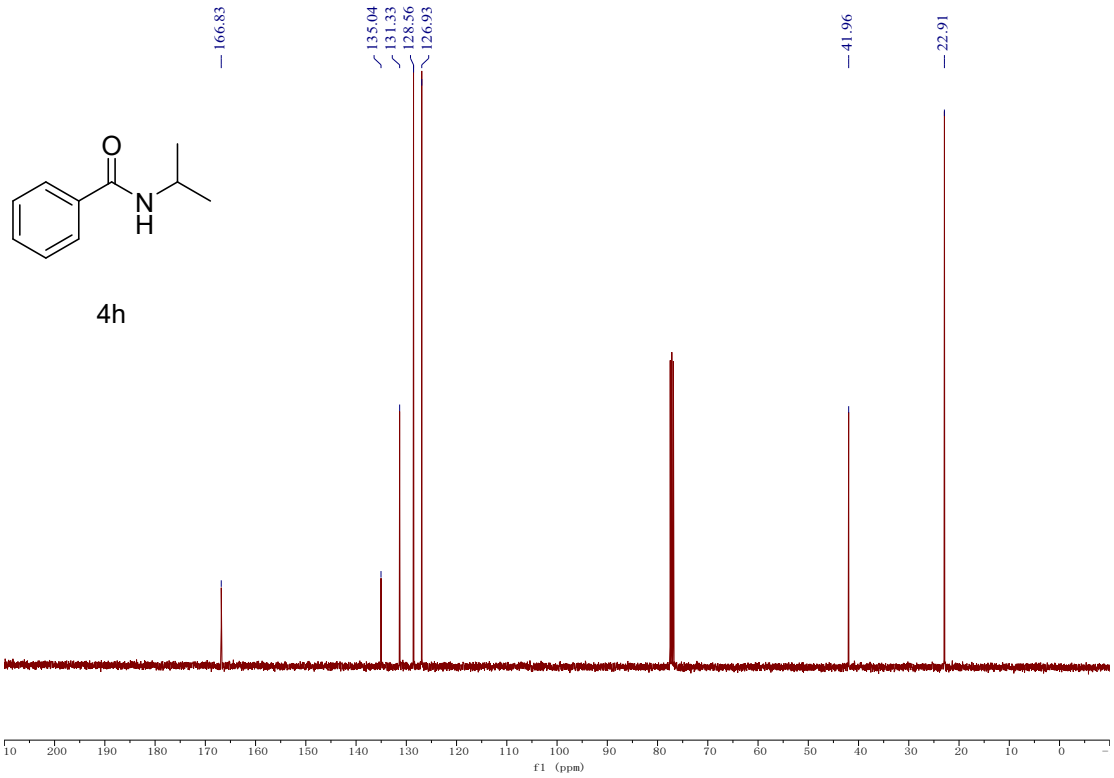

7.39
7.37
7.37
7.36
7.35
7.34
3.68
3.66
3.65
3.36
3.35
3.34
1.86
1.84
1.83
1.81
1.80
1.66
1.64
1.63
1.63
1.62
1.60
1.58
1.57
1.56
1.55
1.54
O
N
4g
5.03
2.00
1.96
2.03
6.12
f1 (ppm)

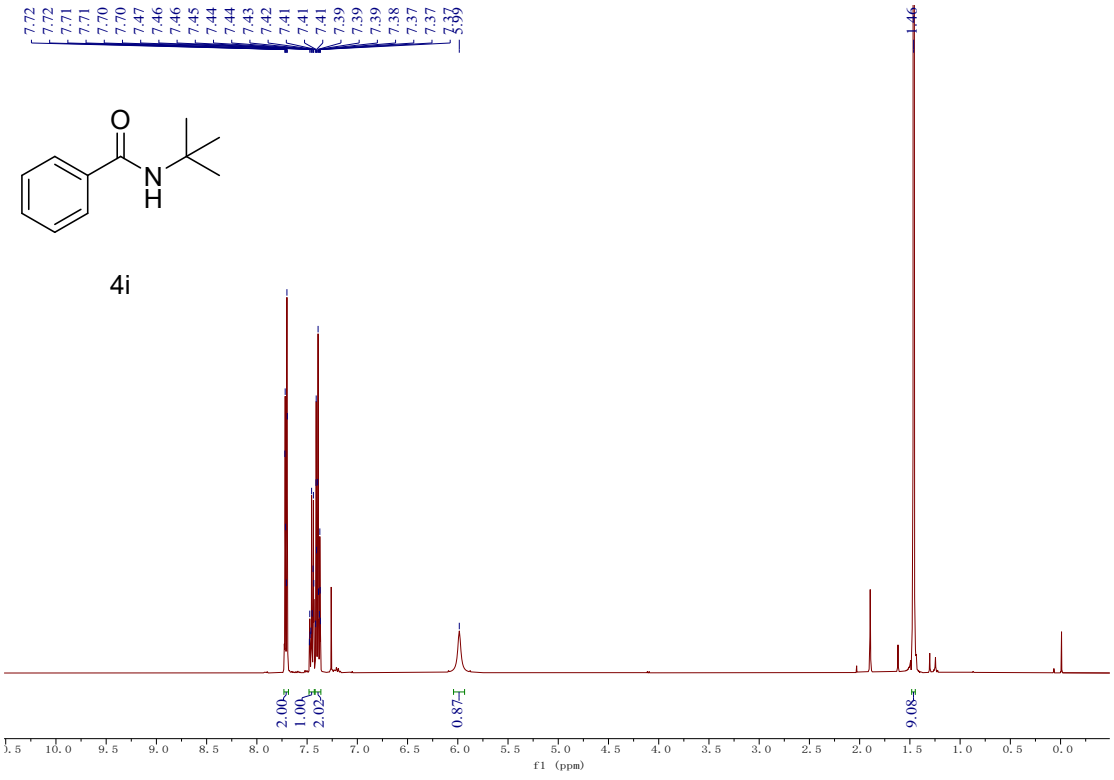

171.72
137.31
129.17
128.48
126.53
49.86
46.45
29.58
27.94
27.33
26.53
O
N
f1 (ppm)

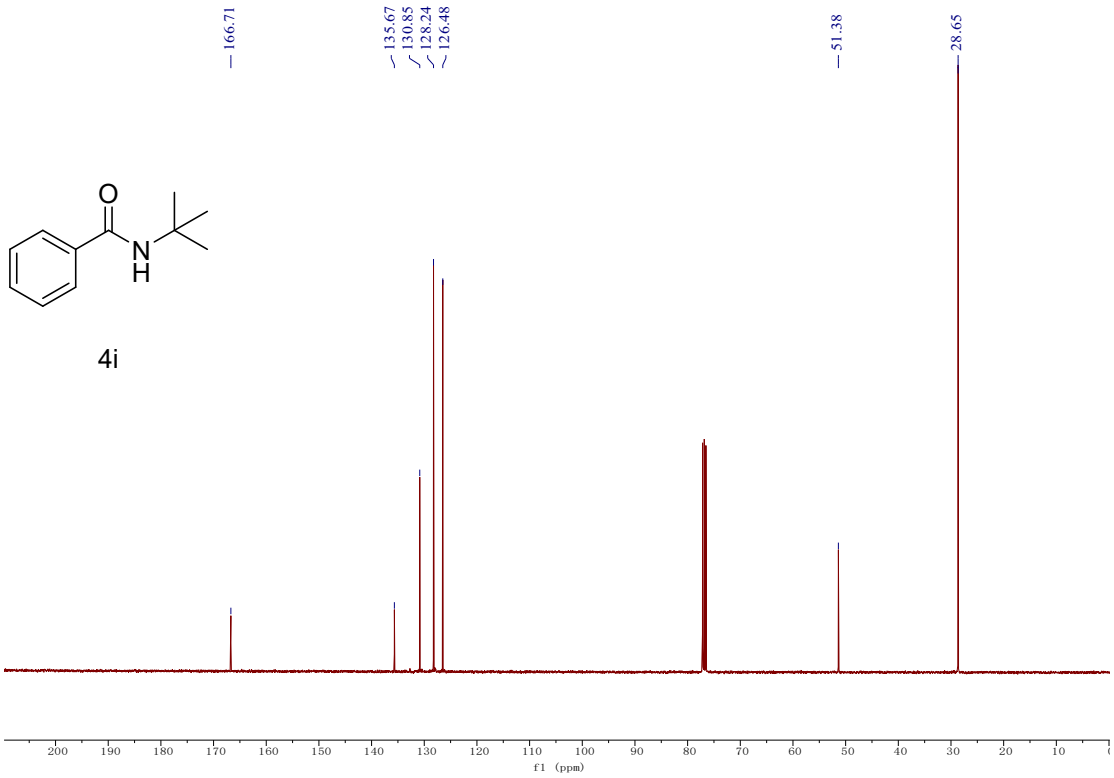
4g

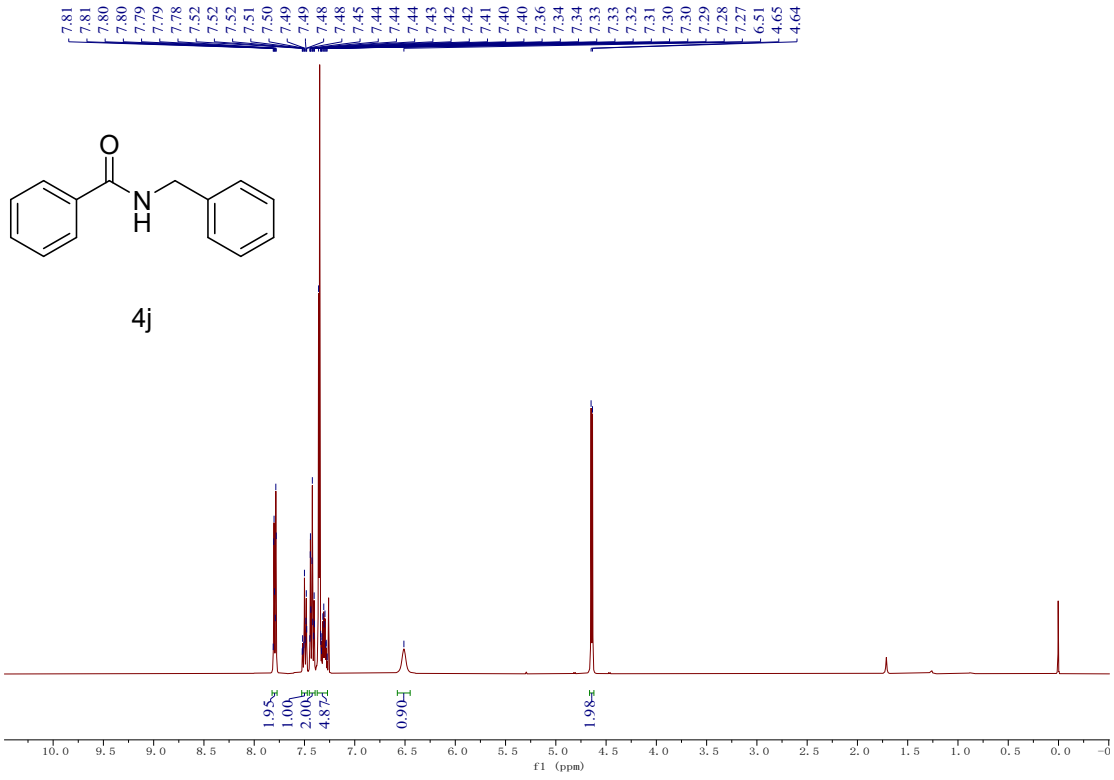

7.75
7.75
7.75
7.74
7.73
7.73
7.48
7.46
7.45
7.45
7.44
7.44
7.41
7.41
7.41
7.40
7.39
7.39
7.38
7.37
7.37
7.37
7.37
6.11
4.31
4.29
4.29
4.28
4.27
4.26
4.26
4.24
4.24
4.23
4.22
1.25
1.23
1.99
1.00
2.00
0.87
1.01
6.20
O
N
H
f1 (ppm)

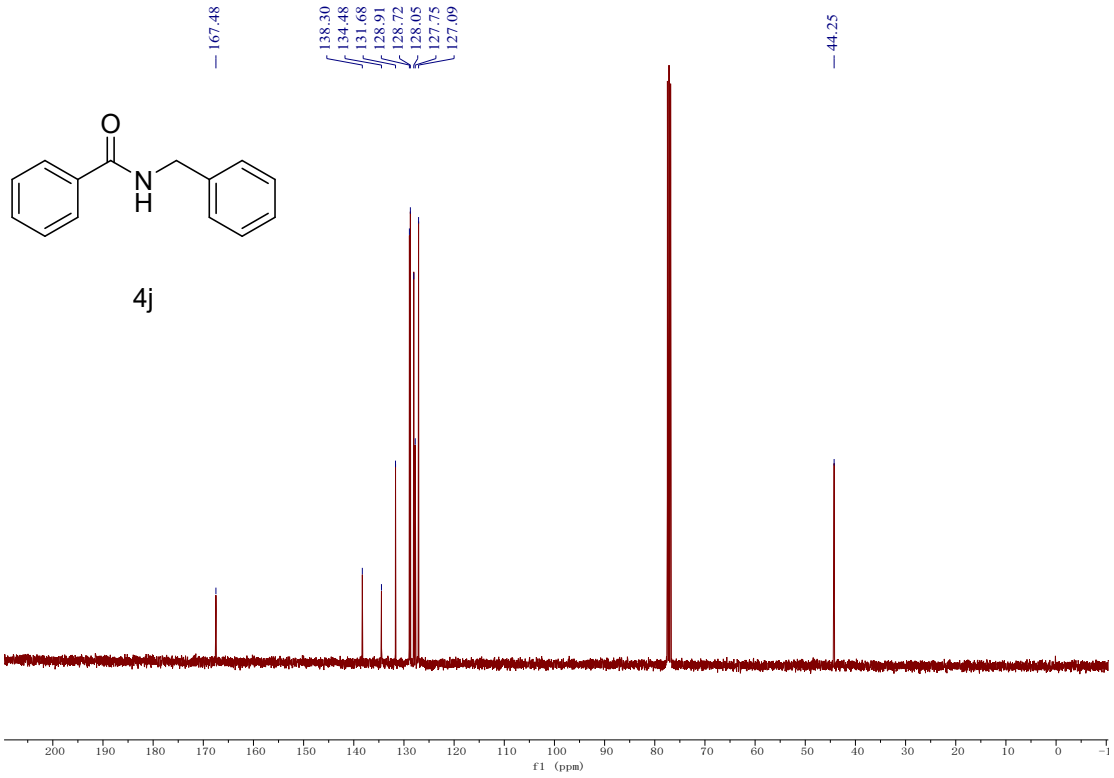
4h

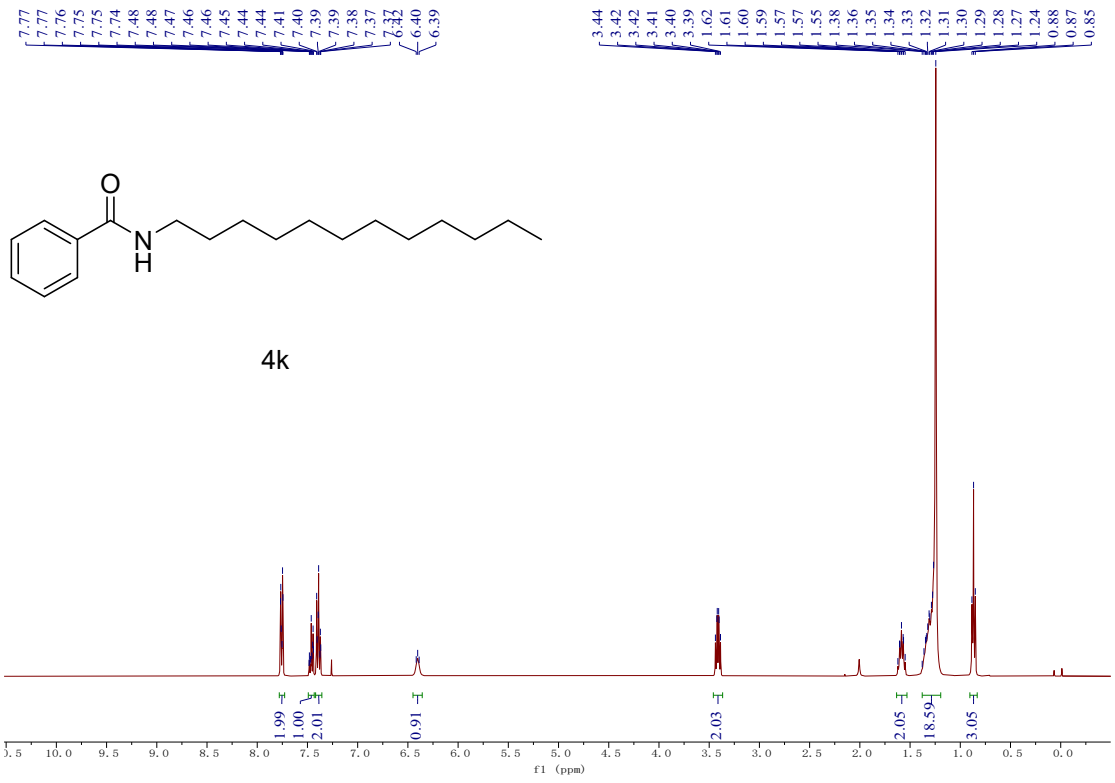

166.83
135.04
131.33
128.56
126.93
41.96
22.91
O
N
H
4h
f1 (ppm)

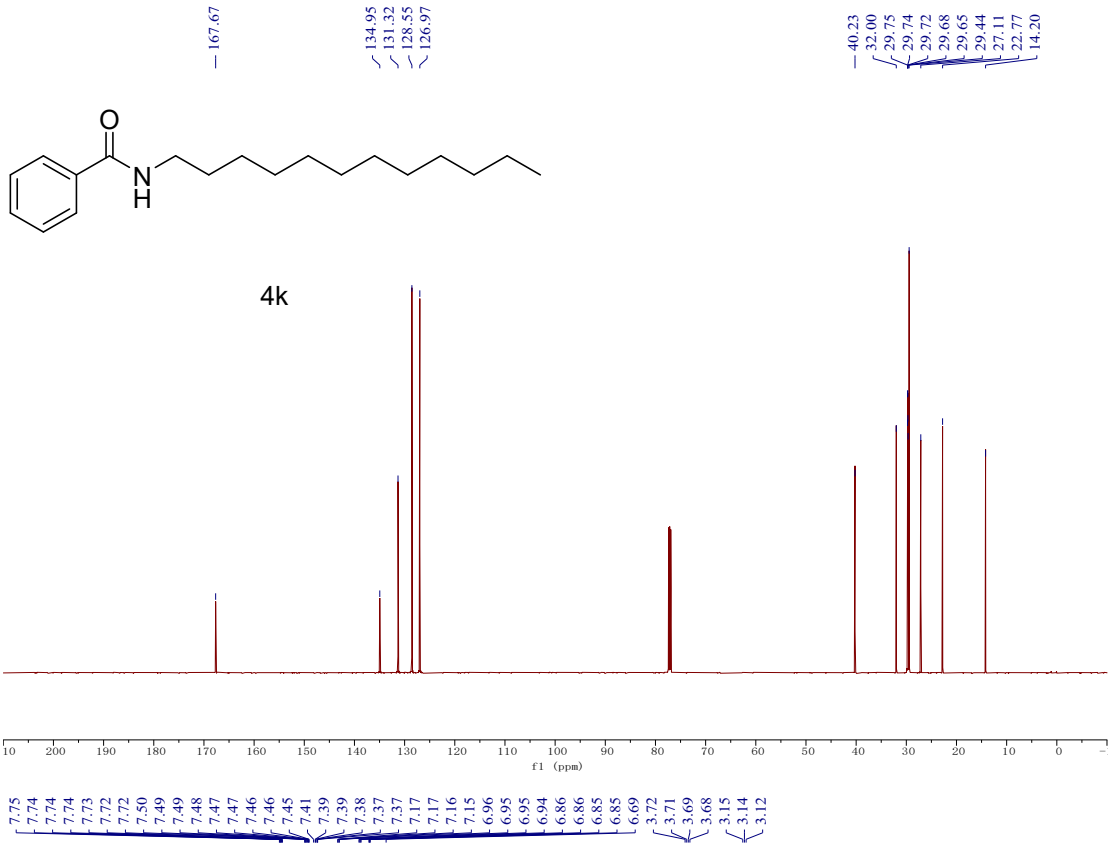

7.72
7.72
7.71
7.71
7.70
7.70
7.47
7.46
7.46
7.45
7.44
7.44
7.43
7.42
7.41
7.41
7.41
7.39
7.39
7.39
7.38
7.37
7.37
7.37
5.99
1.46
O
N
H
4i
2.00
1.00
2.02
0.87
9.08
f1 (ppm)

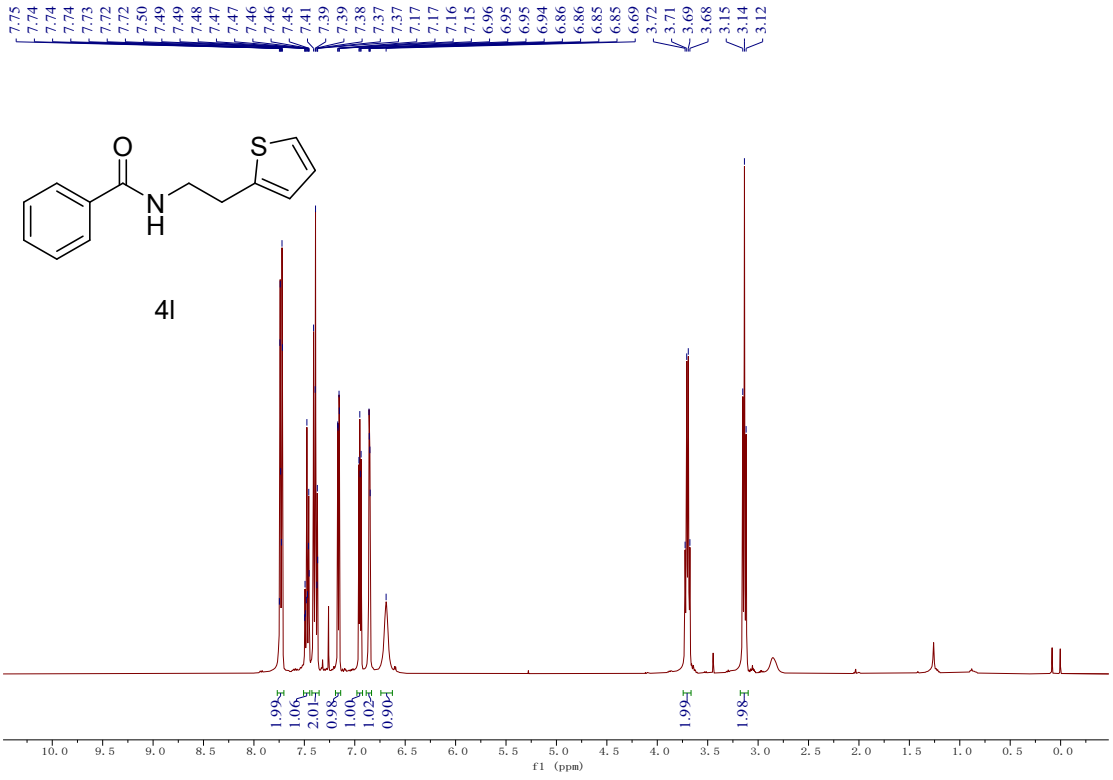

166.71
135.67
130.85
128.24
126.48
51.38
28.65
O
N
H
4i
f1 (ppm)

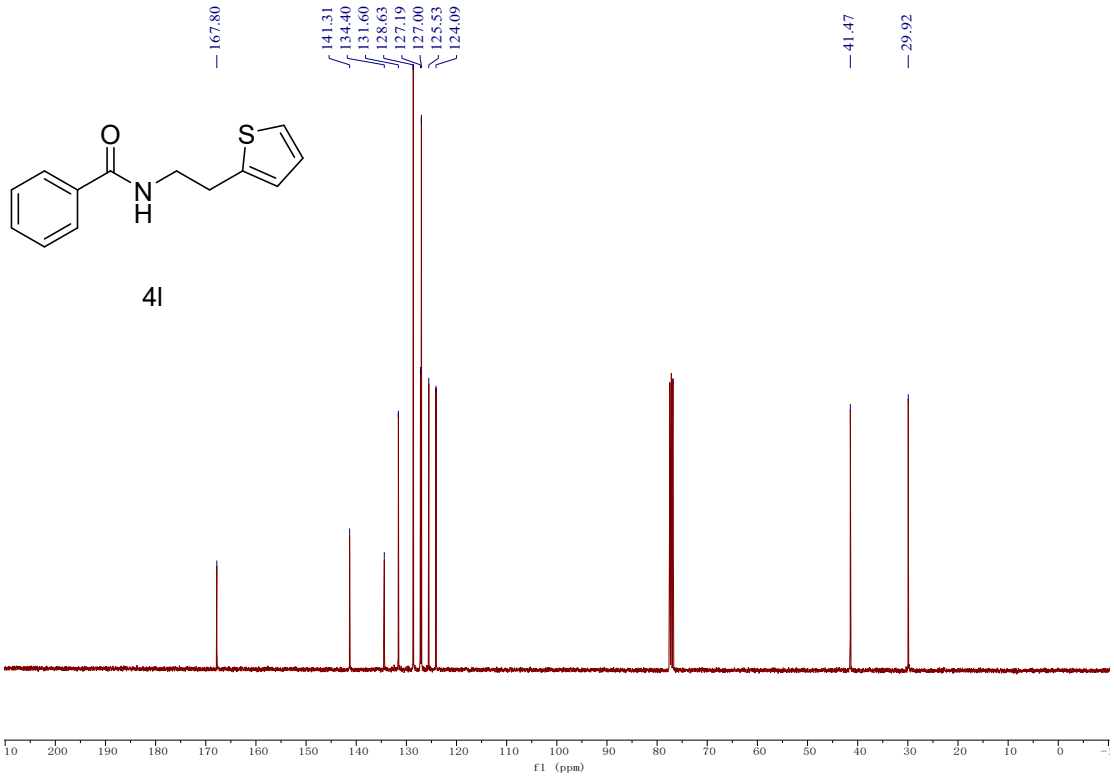

7.81
7.81
7.80
7.80
7.79
7.79
7.78
7.52
7.52
7.52
7.51
7.50
7.49
7.49
7.48
7.48
7.45
7.44
7.44
7.44
7.43
7.42
7.42
7.41
7.40
7.40
7.36
7.34
7.34
7.33
7.33
7.32
7.31
7.30
7.30
7.29
7.28
7.27
6.51
4.65
4.64
O
N
H
4j
1.95
1.00
2.00
4.87
0.90
1.98
f1 (ppm)

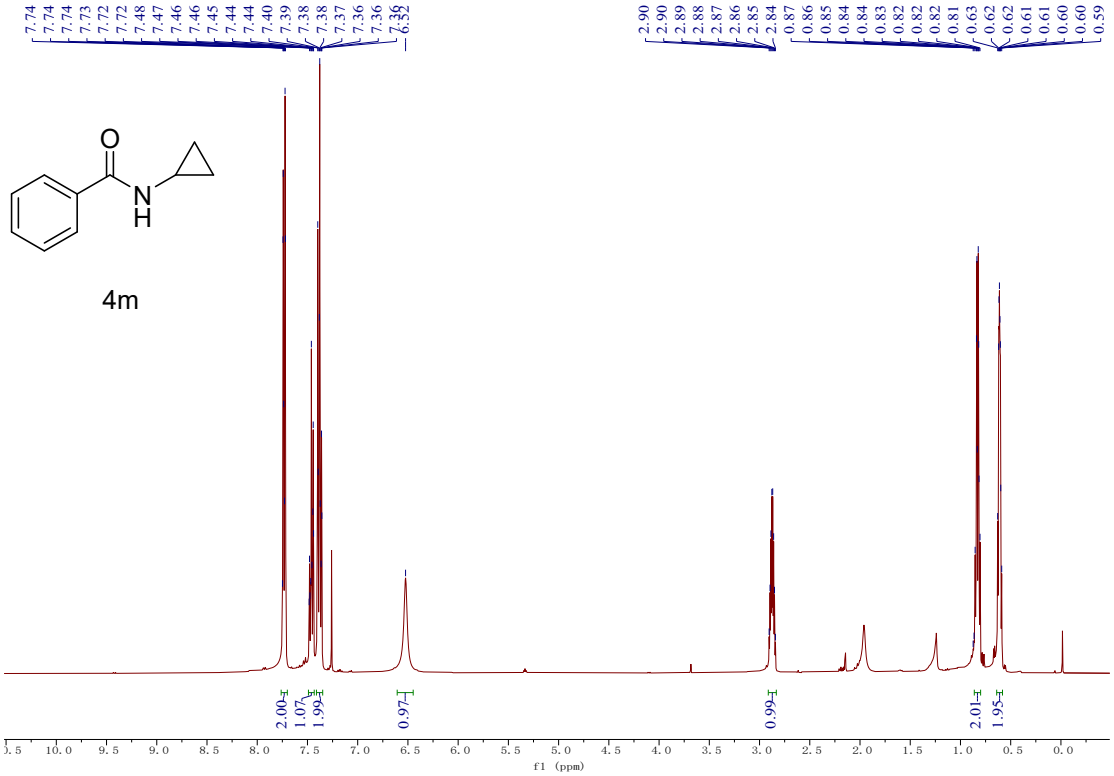

167.48
138.30
134.48
131.68
128.91
128.72
128.05
127.75
127.09
44.25
O
N
H
4j
f1 (ppm)

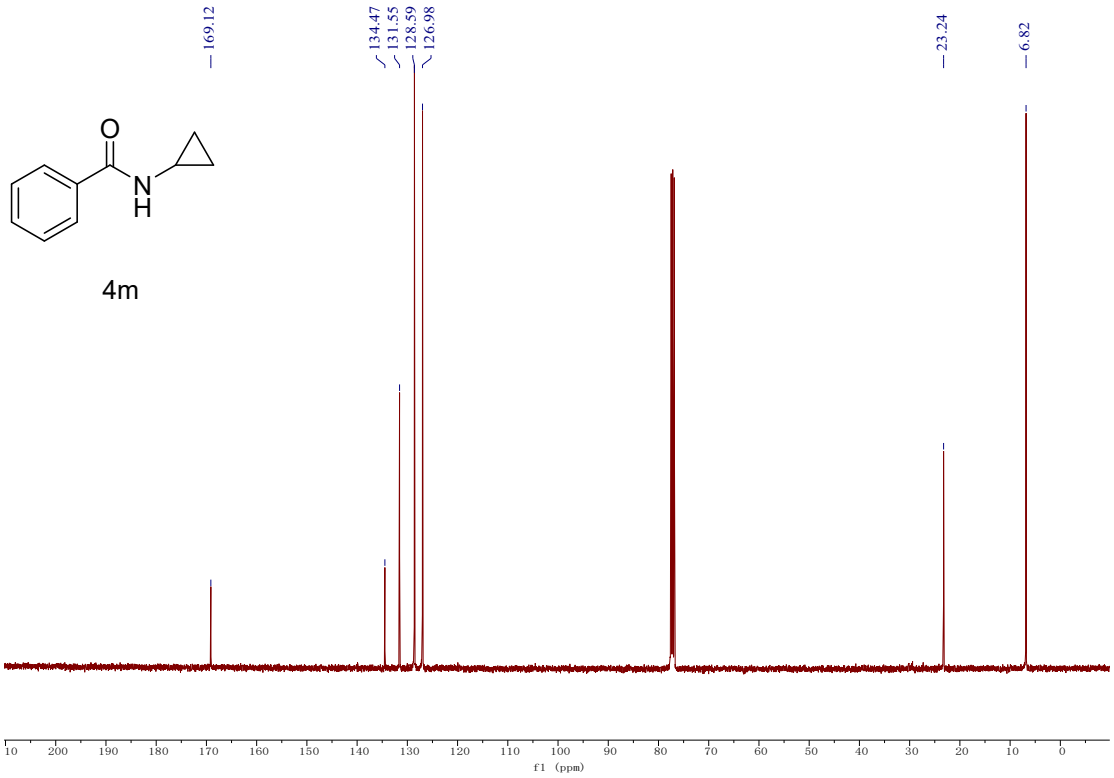

7.77
7.77
7.76
7.75
7.75
7.74
7.48
7.48
7.47
7.46
7.46
7.45
7.44
7.44
7.41
7.40
7.39
7.39
7.38
7.37
7.37
6.42
6.40
6.39
3.44
3.42
3.42
3.41
3.40
3.39
1.62
1.61
1.60
1.59
1.57
1.57
1.55
1.38
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.29
1.28
1.27
1.24
0.88
0.87
0.85
O
N
H
4k
1.99
1.00
2.01
0.91
2.03
2.05
18.59
3.05
f1 (ppm)

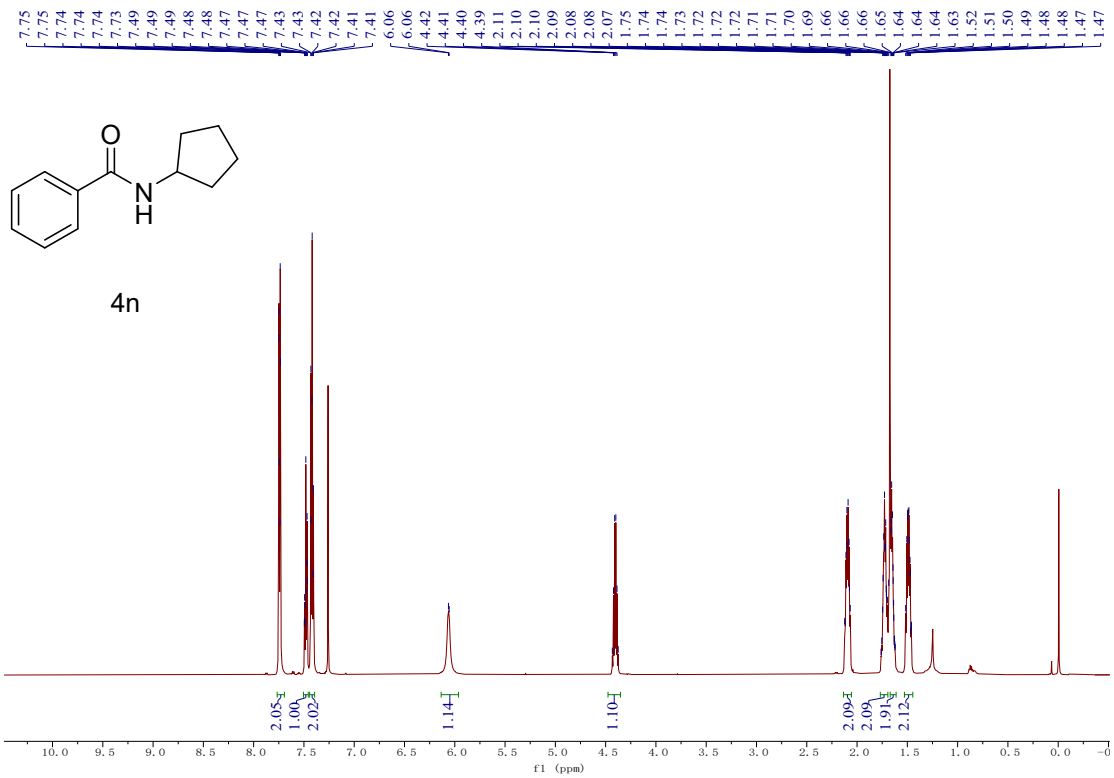

167.67
134.95
131.32
128.55
126.97
40.23
32.00
29.75
29.74
29.72
29.68
29.65
29.44
27.11
22.77
14.20
O
N
H
4k
f1 (ppm)

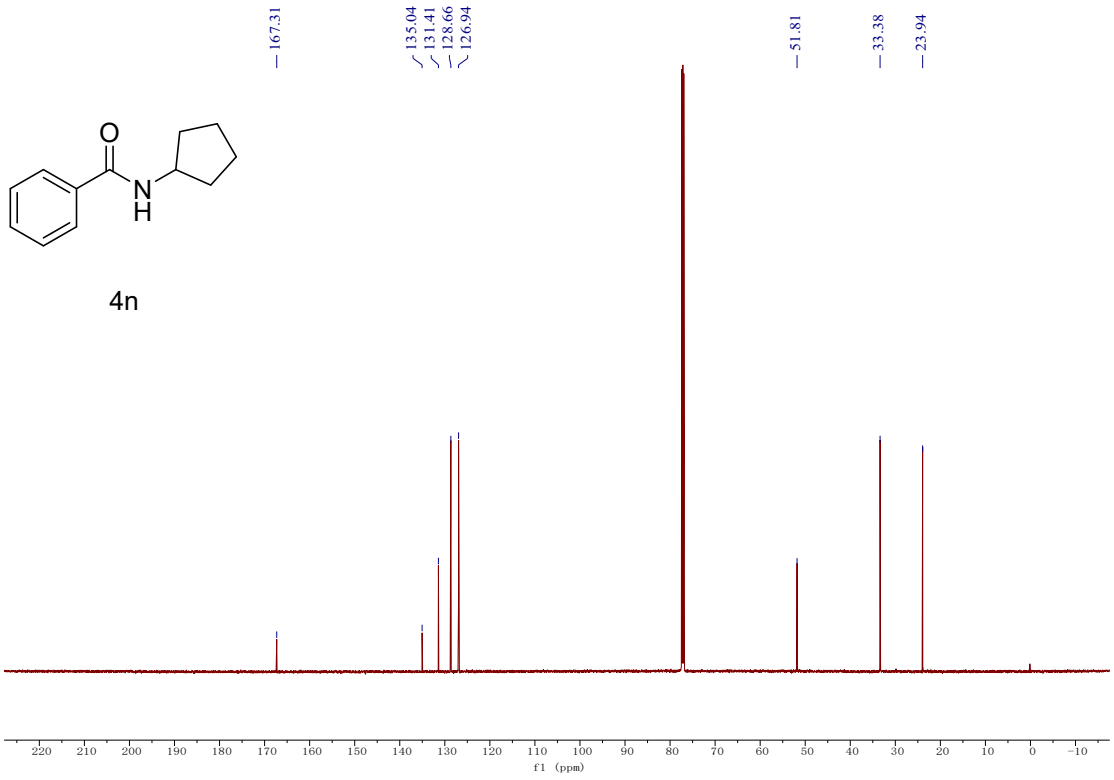

7.75
7.74
7.74
7.74
7.73
7.72
7.72
7.50
7.49
7.49
7.48
7.47
7.47
7.46
7.46
7.45
7.41
7.39
7.39
7.38
7.37
7.37
7.17
7.17
7.16
7.15
6.96
6.95
6.95
6.94
6.86
6.86
6.85
6.85
6.69
3.72
3.71
3.69
3.68
3.15
3.14
3.12
O
S
N
H
4l
1.99
1.06
2.01
0.98
1.00
1.02
0.90
1.99
1.98
f1 (ppm)


167.80
141.31
134.40
131.60
128.63
127.19
127.00
125.53
124.09
41.47
29.92
O
S
N
H
4l
f1 (ppm)

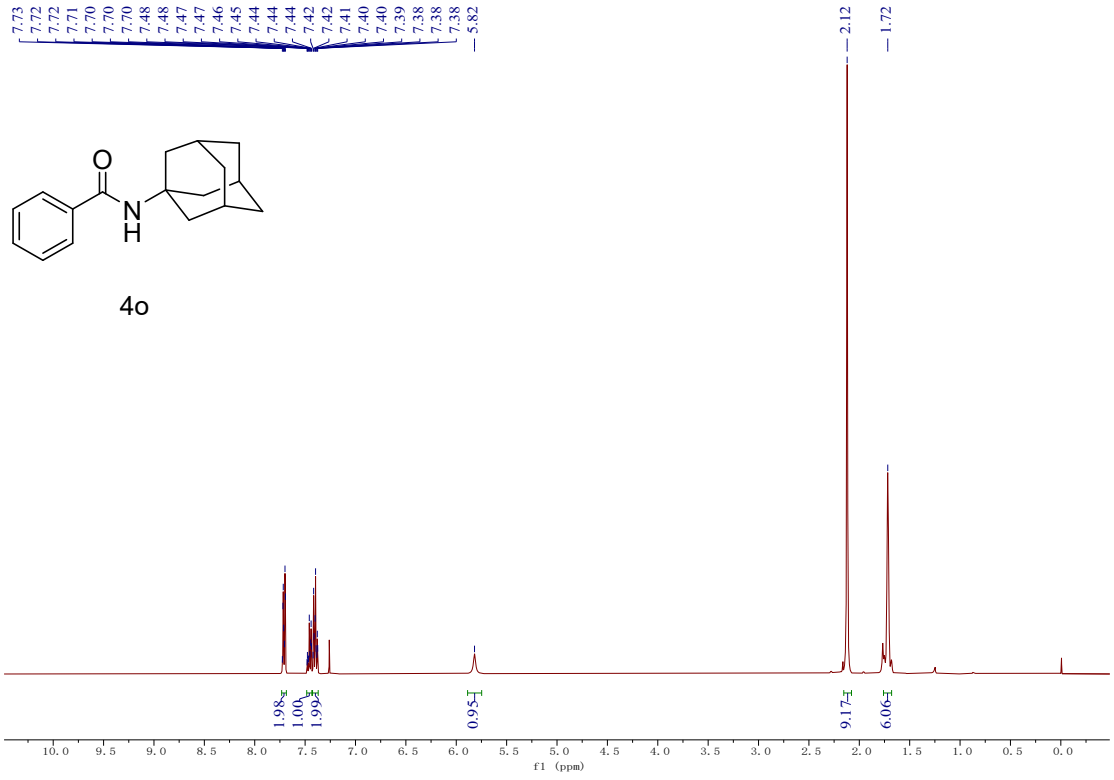

7.74
7.74
7.74
7.73
7.72
7.72
7.48
7.47
7.46
7.46
7.45
7.44
7.44
7.40
7.39
7.38
7.38
7.37
7.36
7.36
7.35
6.32
2.90
2.90
2.89
2.88
2.87
2.86
2.85
2.84
0.87
0.86
0.85
0.84
0.84
0.83
0.82
0.82
0.82
0.81
0.63
0.62
0.62
0.61
0.61
0.60
0.60
0.59
2.00
1.07
1.99
0.97
0.99
2.01
1.95
O
N
H
4m
f1 (ppm)


169.12
134.47
131.55
128.59
126.98
23.24
6.82
O
N
H
4m
f1 (ppm)

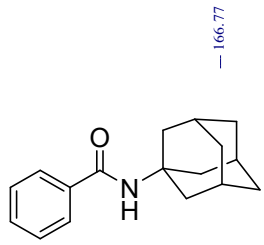

7.75
7.74
7.49
7.48
7.47
7.43
7.42
7.41
6.06
4.42
4.41
4.40
4.39
2.11
2.10
2.09
2.08
2.07
1.75
1.74
1.73
1.72
1.71
1.70
1.69
1.66
1.65
1.64
1.63
1.52
1.51
1.50
1.49
1.48
1.47
O
N
H
4n
2.05
1.00
2.02
1.14
1.10
2.09
2.09
1.91
2.12
f1 (ppm)


167.31
135.04
131.41
128.66
126.94
51.81
33.38
23.94
O
N
H
f1 (ppm)

4n

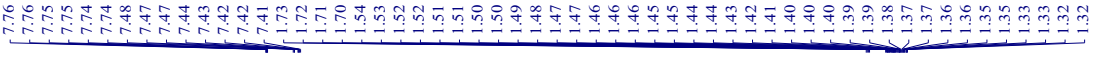

7.73
7.72
7.72
7.71
7.70
7.70
7.70
7.48
7.47
7.47
7.46
7.45
7.44
7.44
7.44
7.42
7.41
7.40
7.40
7.39
7.38
7.38
7.38
5.82
2.12
1.72
1.98
1.00
1.99
0.95
9.17
6.06
O
N
H
f1 (ppm)

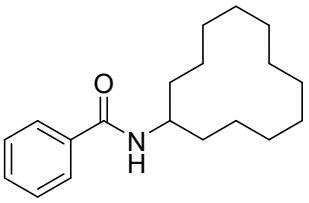
4o



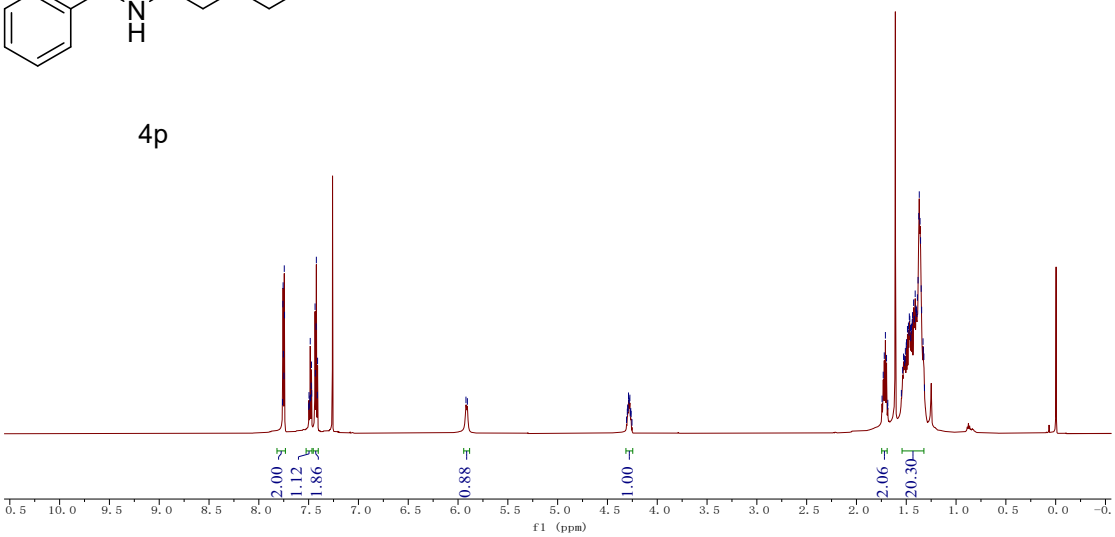
4o

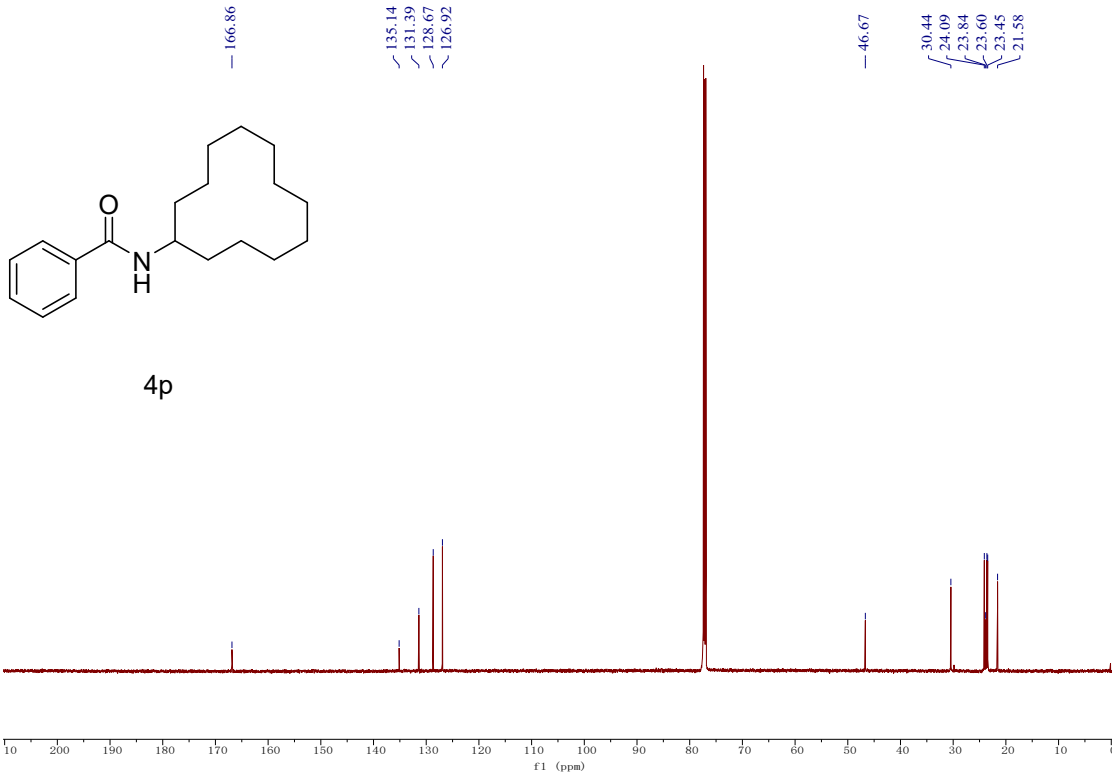
166.77
136.13
131.15
128.57
126.82
52.39
41.77
36.49
36.33
29.74
29.60
29.43
f1 (ppm)


7.76
7.76
7.75
7.75
7.74
7.74
7.48
7.47
7.47
7.44
7.43
7.42
7.42
7.41
1.73
1.72
1.71
1.70
1.54
1.53
1.52
1.52
1.51
1.51
1.50
1.50
1.49
1.48
1.47
1.47
1.46
1.46
1.46
1.45
1.45
1.44
1.44
1.43
1.42
1.41
1.40
1.40
1.40
1.39
1.39
1.38
1.37
1.37
1.36
1.36
1.35
1.35
1.33
1.33
1.32
1.32

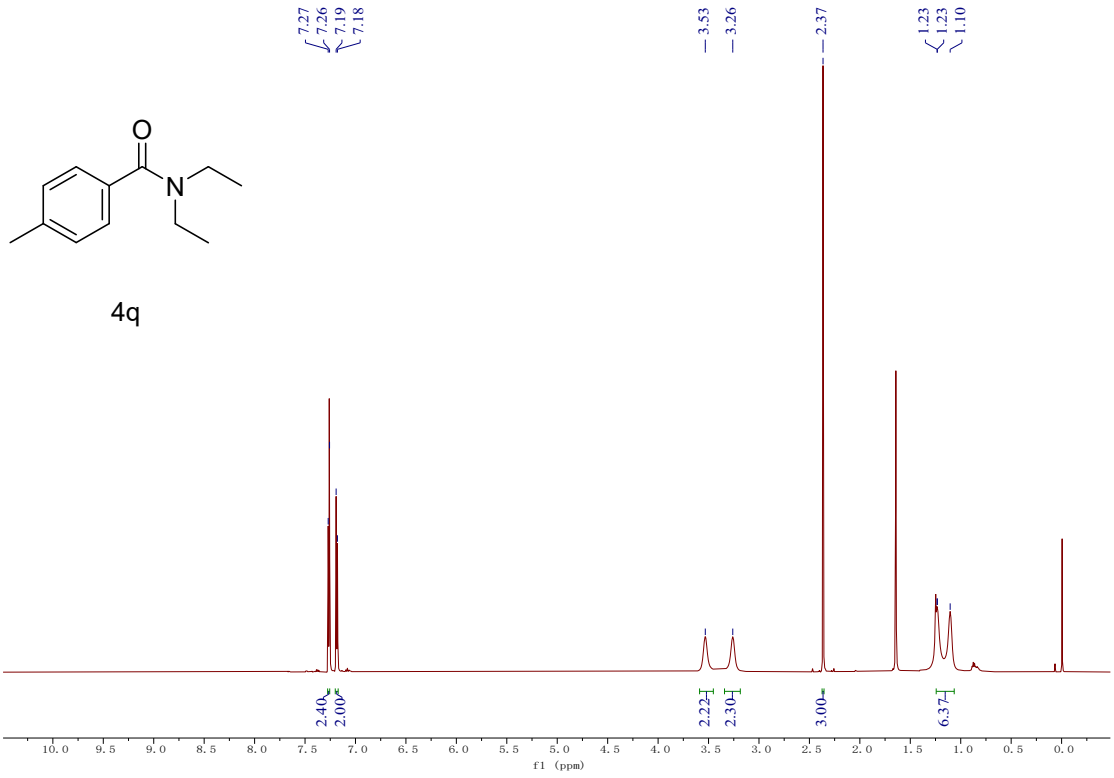


4p

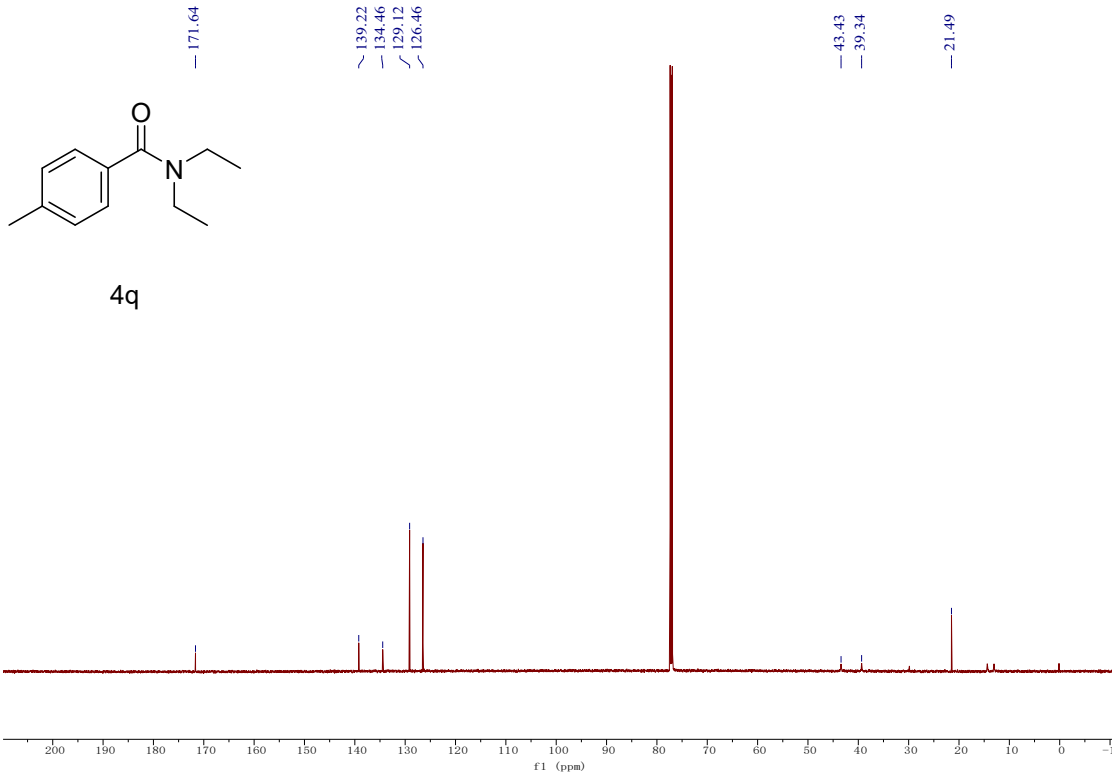

2.00
1.12
1.86
0.88
1.00
2.06
20.30
f1 (ppm)

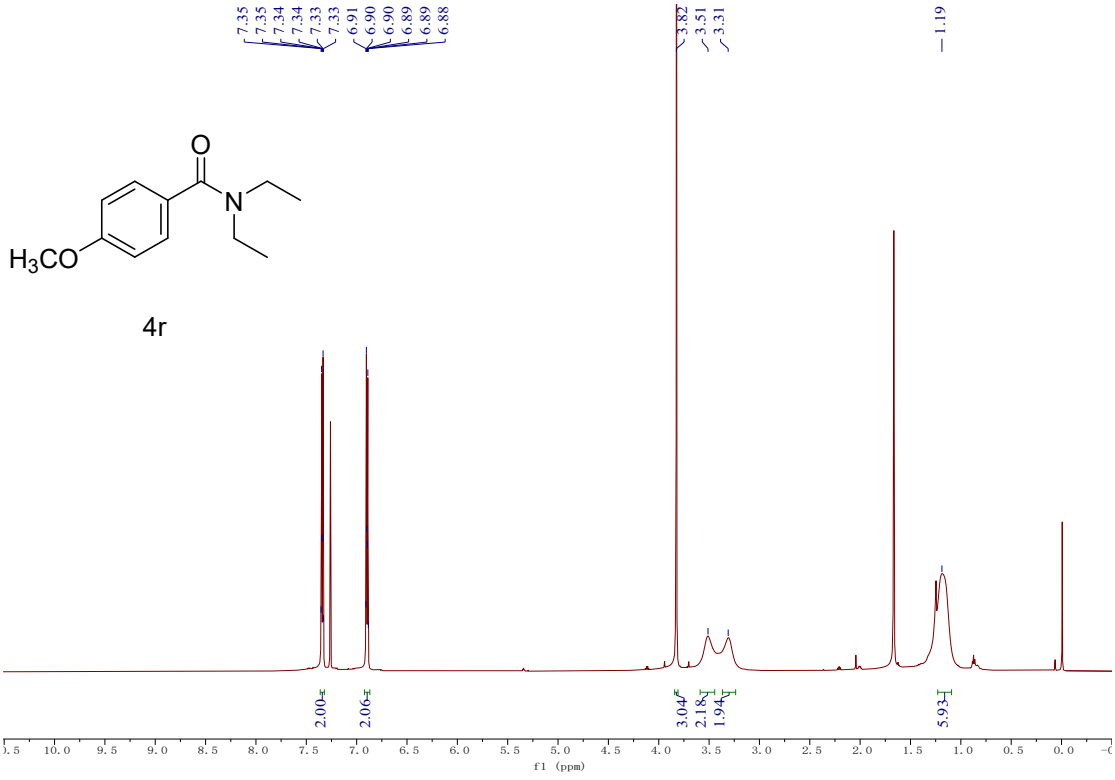

166.86
135.14
131.39
128.67
126.92
46.67
30.44
24.09
23.84
23.60
23.45
21.58
O
N
H
f1 (ppm)

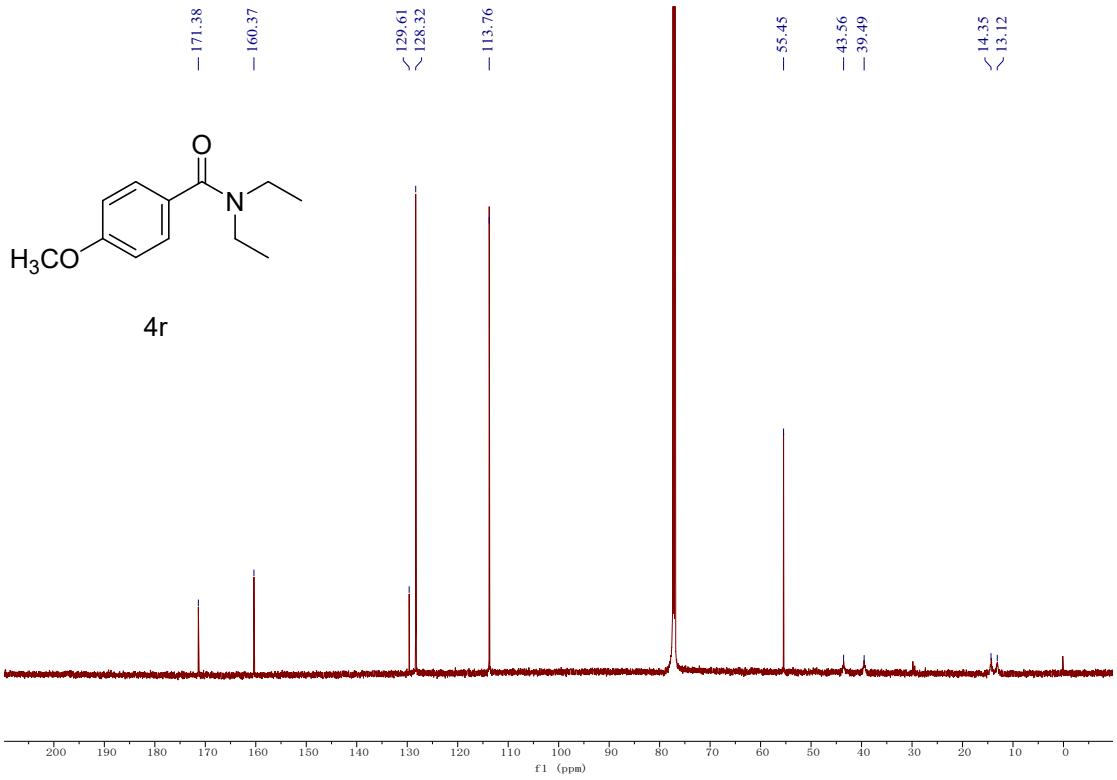
4p


7.27
7.26
7.19
7.18
3.53
3.26
2.37
1.23
1.23
1.10
O
N
2.40
2.00
2.22
2.30
3.00
6.37
f1 (ppm)

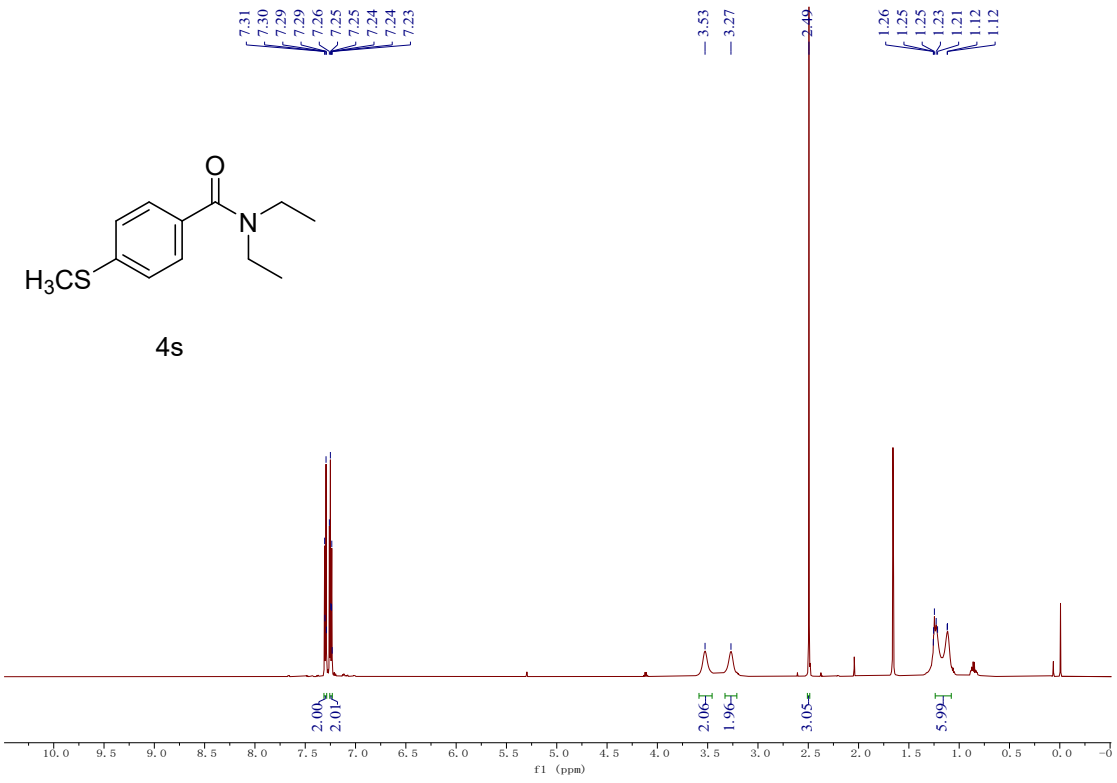
4q


171.64
139.22
134.46
129.12
126.46
43.43
39.34
21.49
O
N
4q
f1 (ppm)

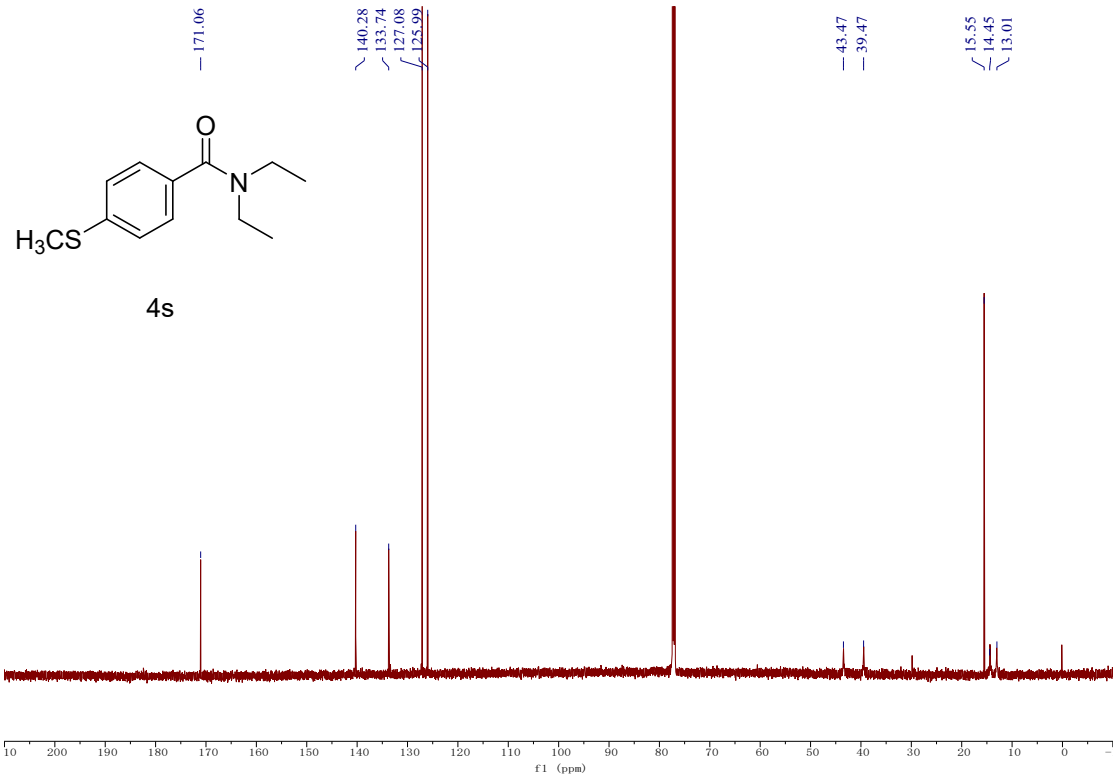

7.35
7.35
7.34
7.34
7.33
7.33
6.91
6.90
6.90
6.89
6.89
6.88
3.82
3.51
3.31
1.19
O
N
H3CO
4r
2.00
2.06
3.04
2.18
1.94
5.93
f1 (ppm)


171.38
160.37
129.61
128.32
113.76
55.45
43.56
39.49
14.35
13.12
O
N
H3CO
f1 (ppm)

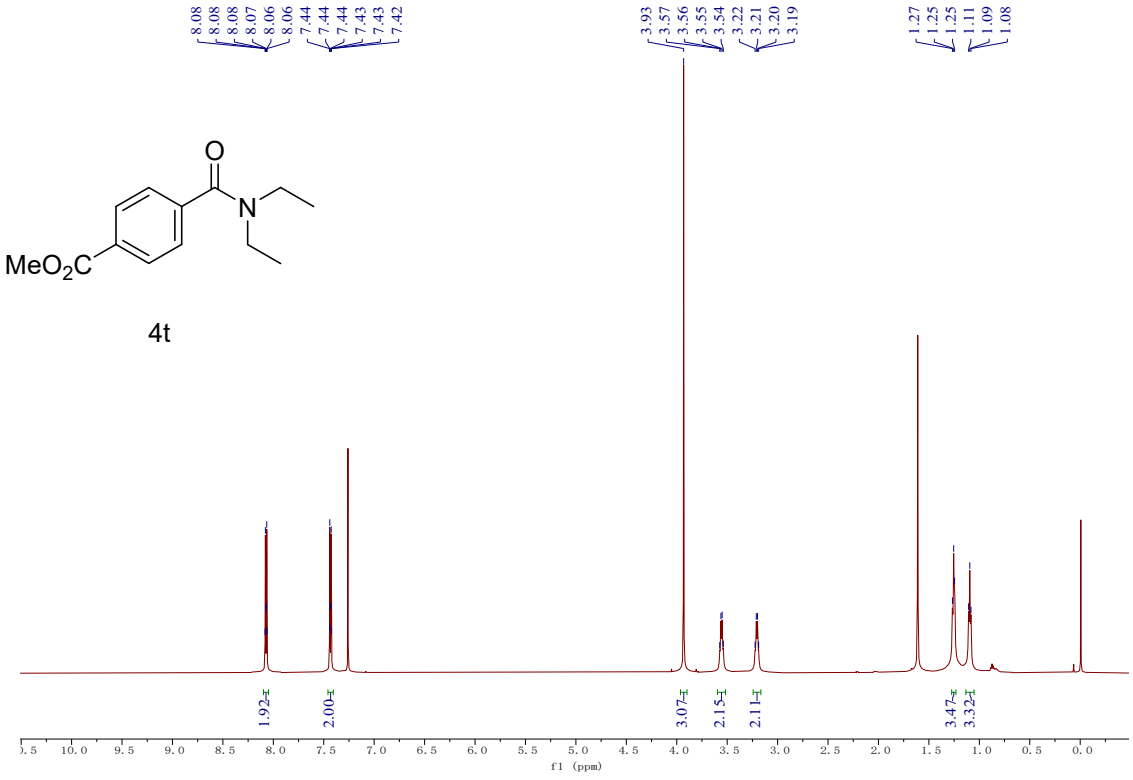
4r


7.31
7.30
7.29
7.29
7.26
7.25
7.25
7.24
7.24
7.23
3.53
3.27
2.49
1.26
1.25
1.25
1.23
1.21
1.12
1.12
O
N
H3CS
2.00
2.01
2.06
1.96
3.05
5.99
f1 (ppm)

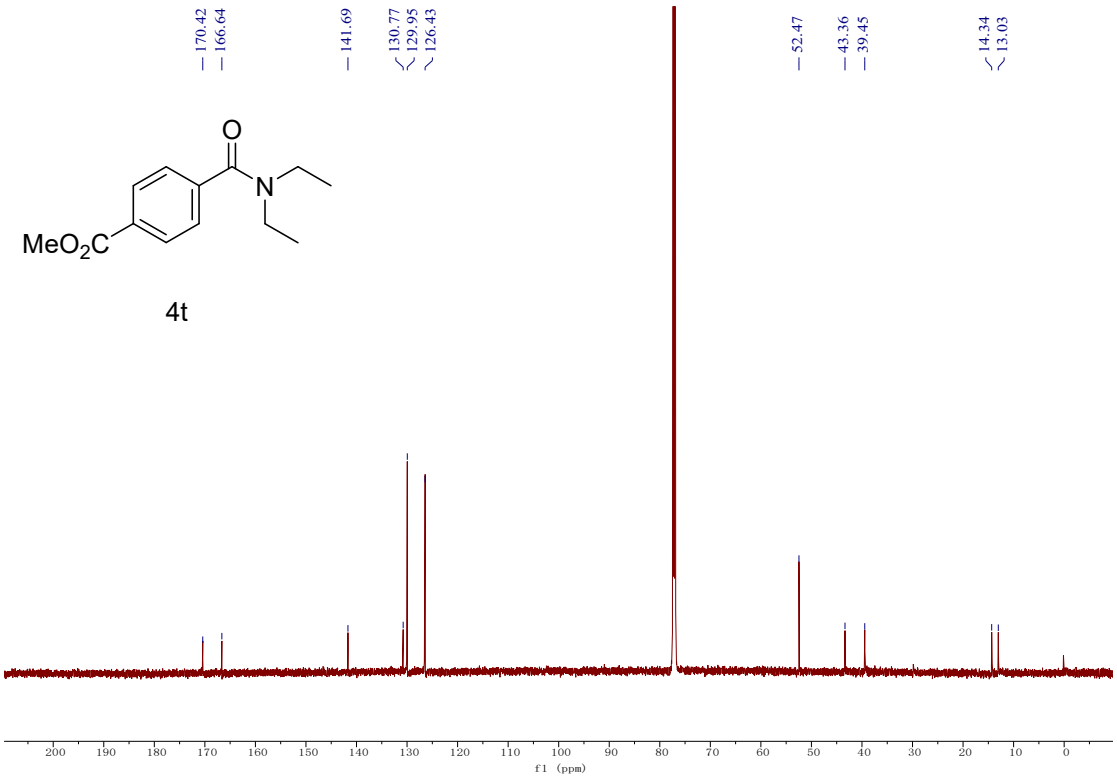
4s


171.06
140.28
133.74
127.08
125.99
43.47
39.47
15.55
14.45
13.01
O
N
H3CS
f1 (ppm)

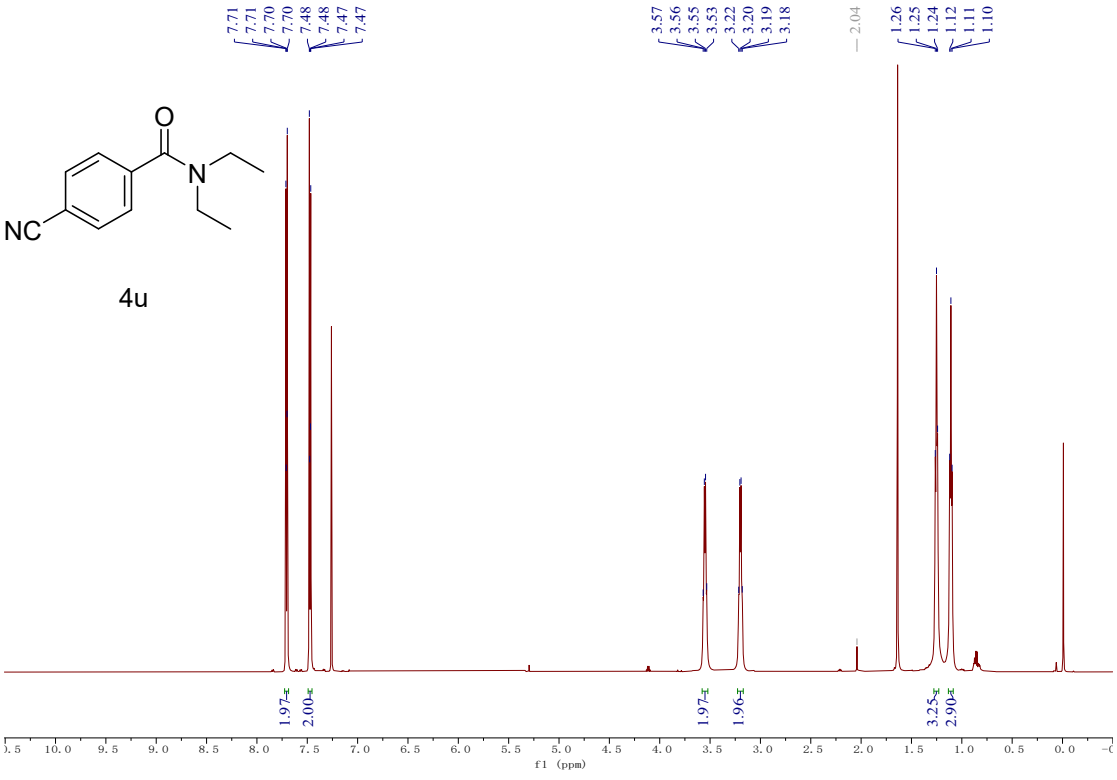
4s


8.08
8.08
8.08
8.07
8.06
8.06
7.44
7.44
7.44
7.43
7.43
7.42
3.93
3.57
3.56
3.55
3.54
3.22
3.21
3.20
3.19
1.27
1.25
1.25
1.11
1.09
1.08
O
N
MeO2C
1.92
2.00
3.07
2.15
2.11
3.47
3.32
f1 (ppm)

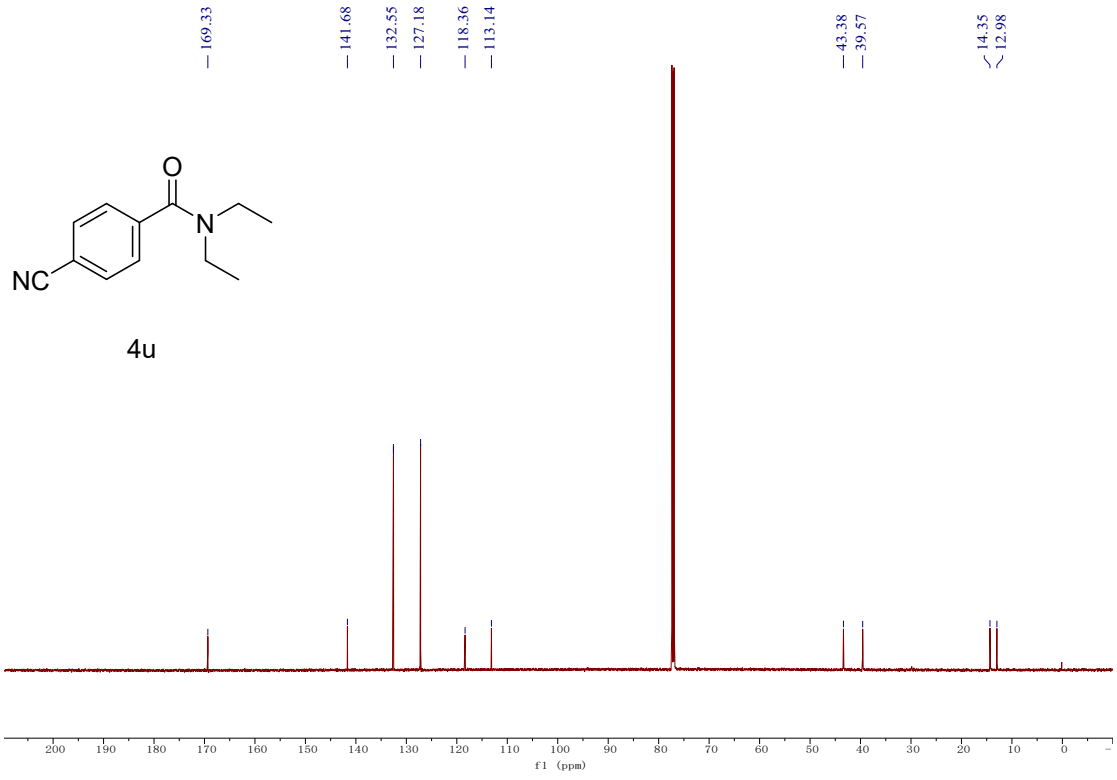
4t


170.42
166.64
141.69
130.77
129.95
126.43
52.47
43.36
39.45
14.34
13.03
O
N
MeO2C
f1 (ppm)

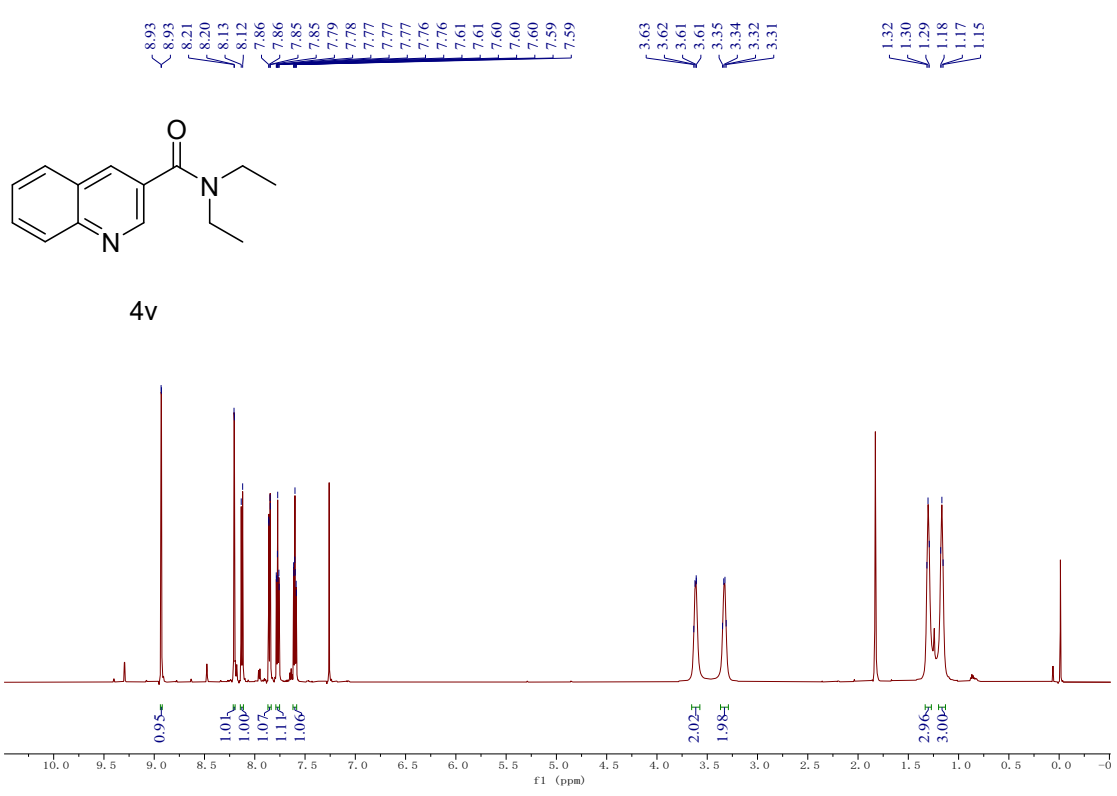
4t


7.71
7.71
7.70
7.70
7.48
7.48
7.47
7.47
3.57
3.56
3.55
3.53
3.22
3.20
3.19
3.18
2.04
1.26
1.25
1.24
1.12
1.11
1.10
O
N
NC
1.97
2.00
1.97
1.96
3.25
2.90
f1 (ppm)

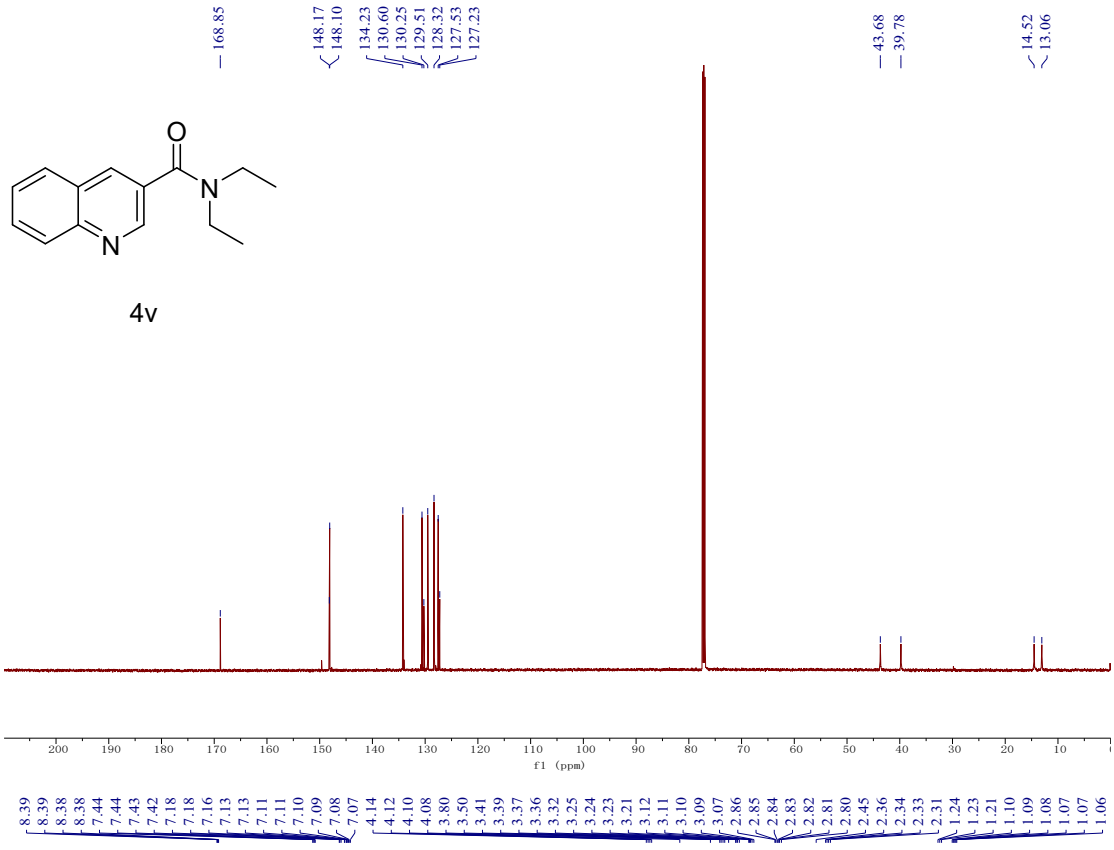
4u


169.33
141.68
132.55
127.18
118.36
113.14
43.38
39.57
14.35
12.98
O
N
NC
f1 (ppm)

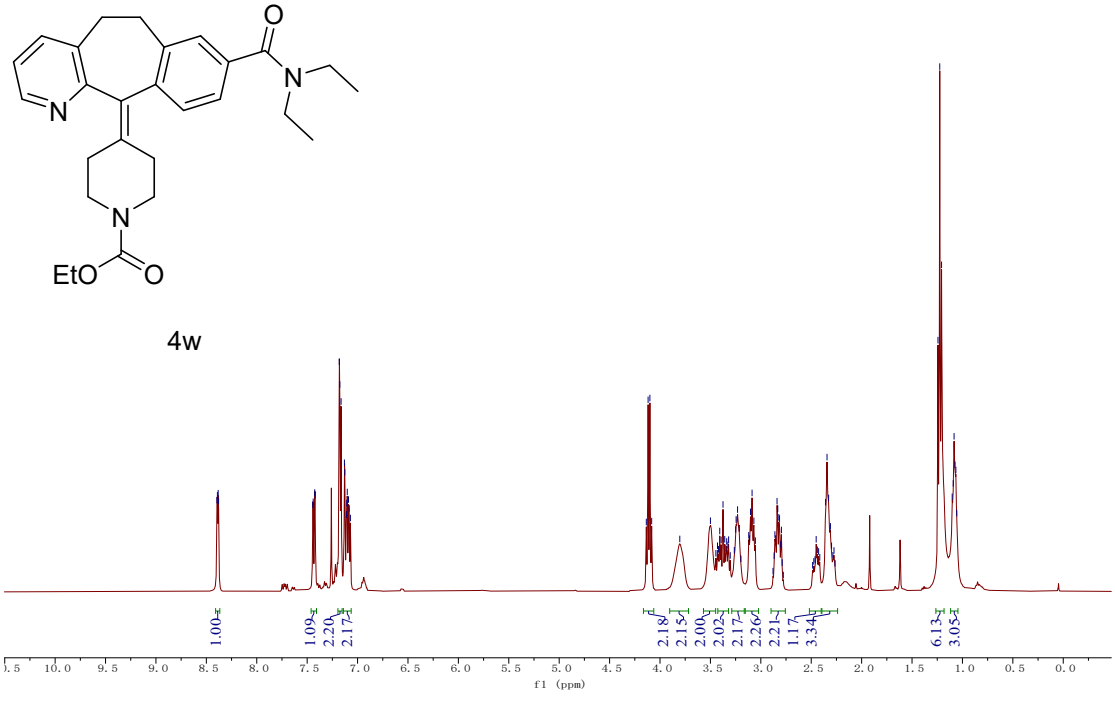
4u


O
N
N
f1 (ppm)

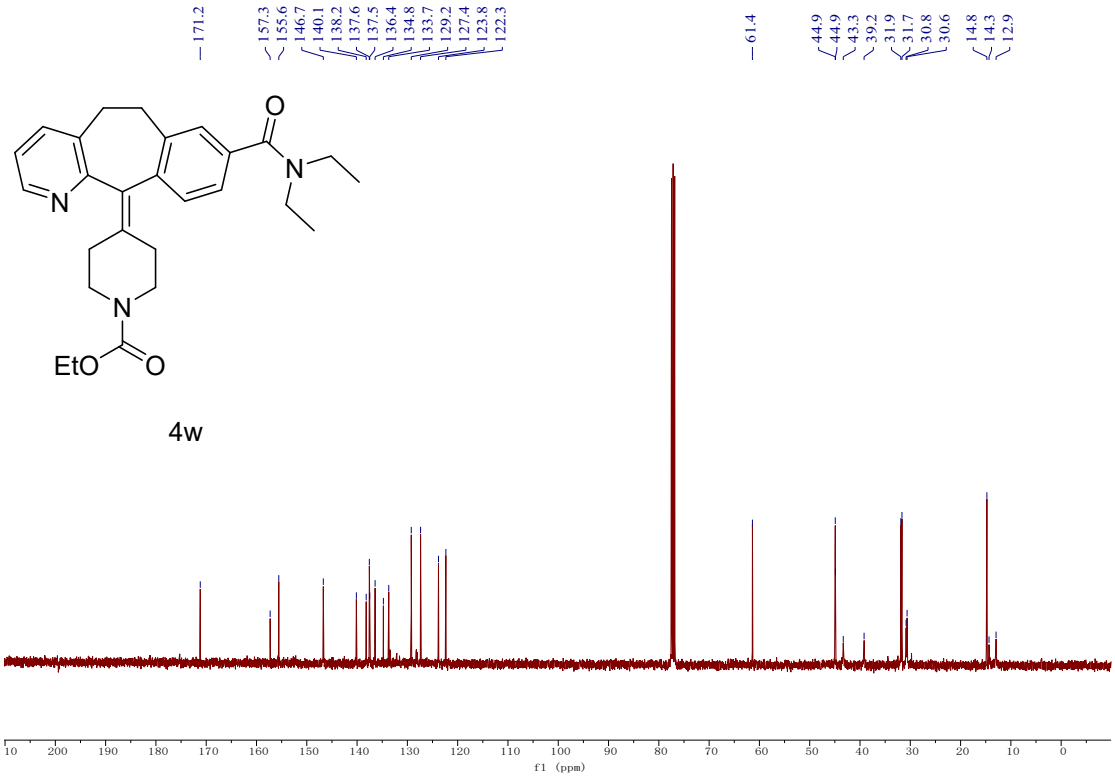
4v


168.85
148.17
148.10
134.23
130.60
130.25
129.51
128.32
127.53
127.23
43.68
39.78
14.52
13.06
O
N
N
f1 (ppm)

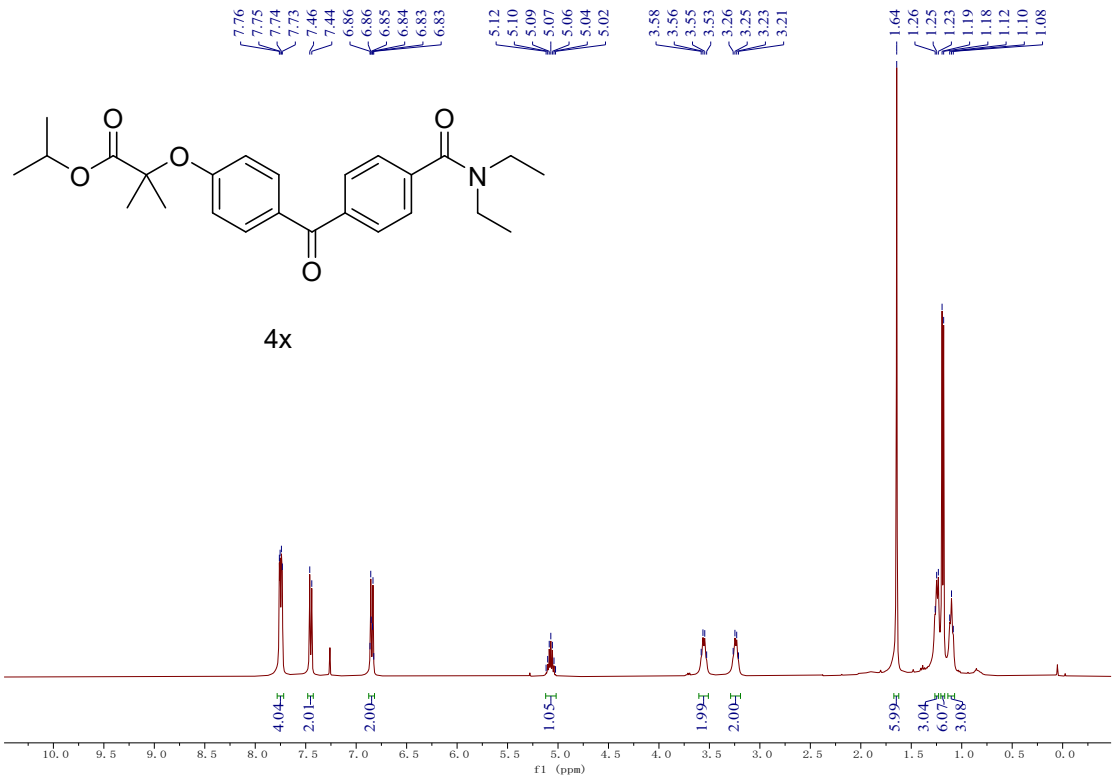
4v


O
N
N
N
EtO
O
1.00
1.09
2.20
2.17
2.18
2.15
2.00
2.02
2.17
2.26
2.21
1.17
3.34
6.13
3.05
f1 (ppm)

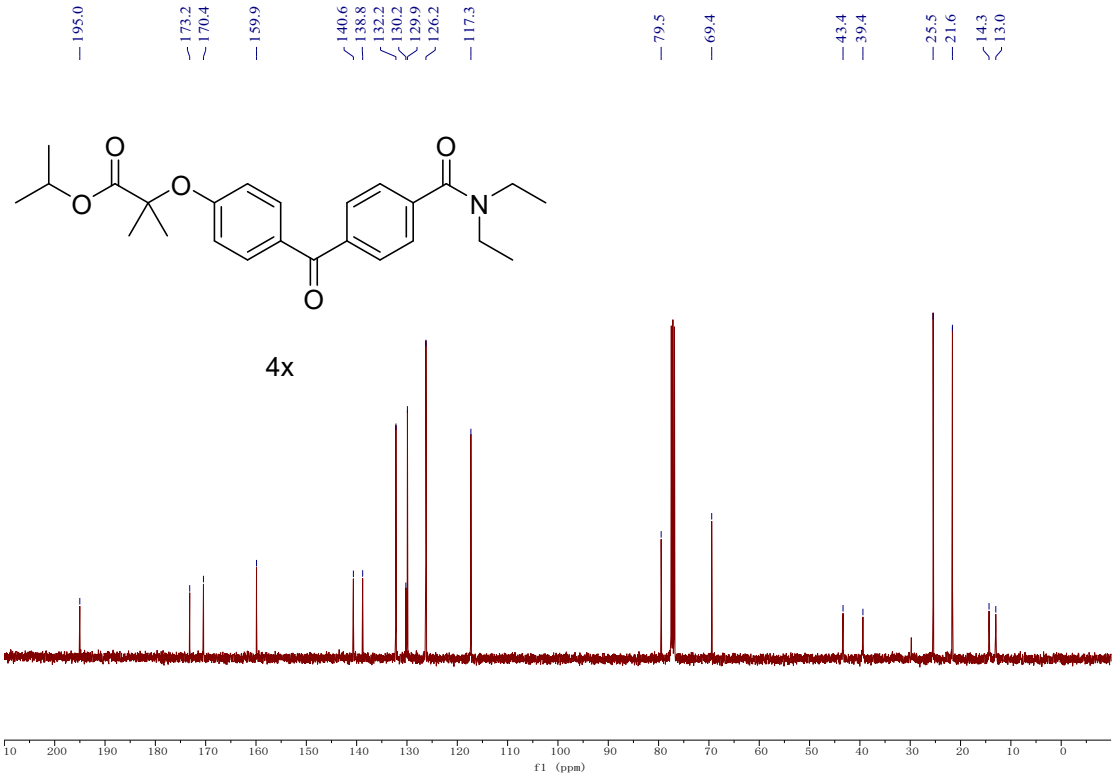
4w

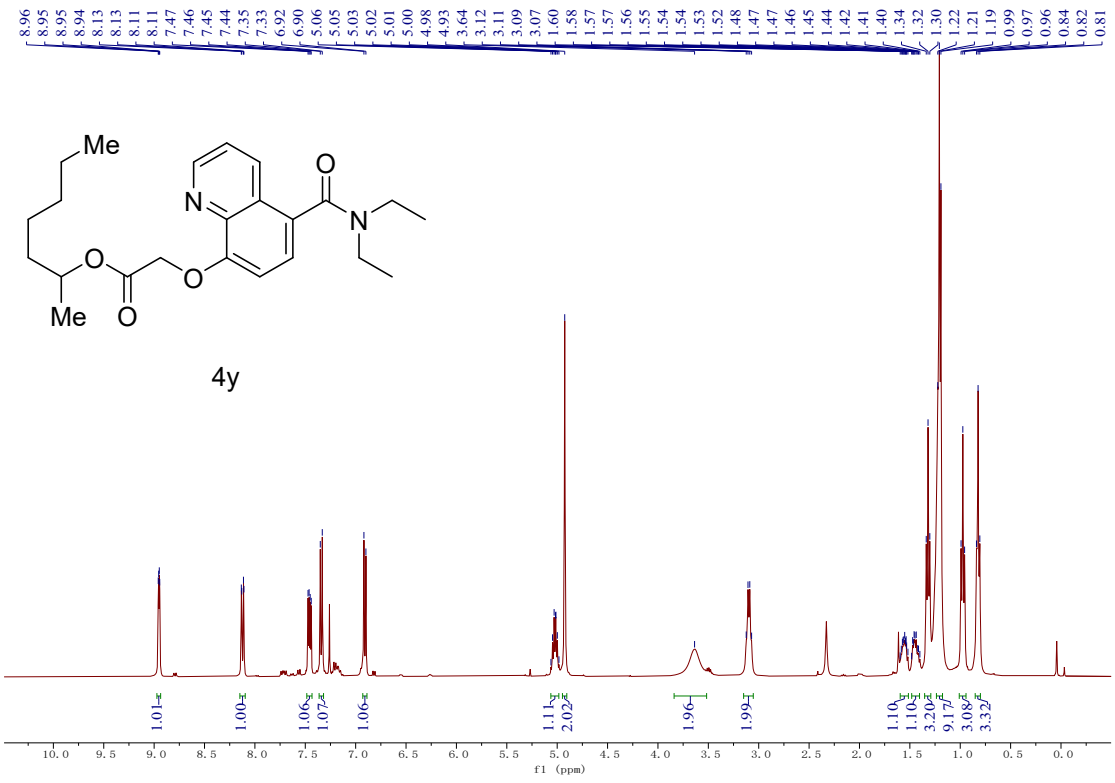

171.2
157.3
155.6
146.7
140.1
138.2
137.6
137.5
136.4
134.8
133.7
129.2
127.4
123.8
122.3
61.4
44.9
44.9
43.3
39.2
31.9
31.7
30.8
30.6
14.8
14.3
12.9
O
N
N
EtO
O
f1 (ppm)

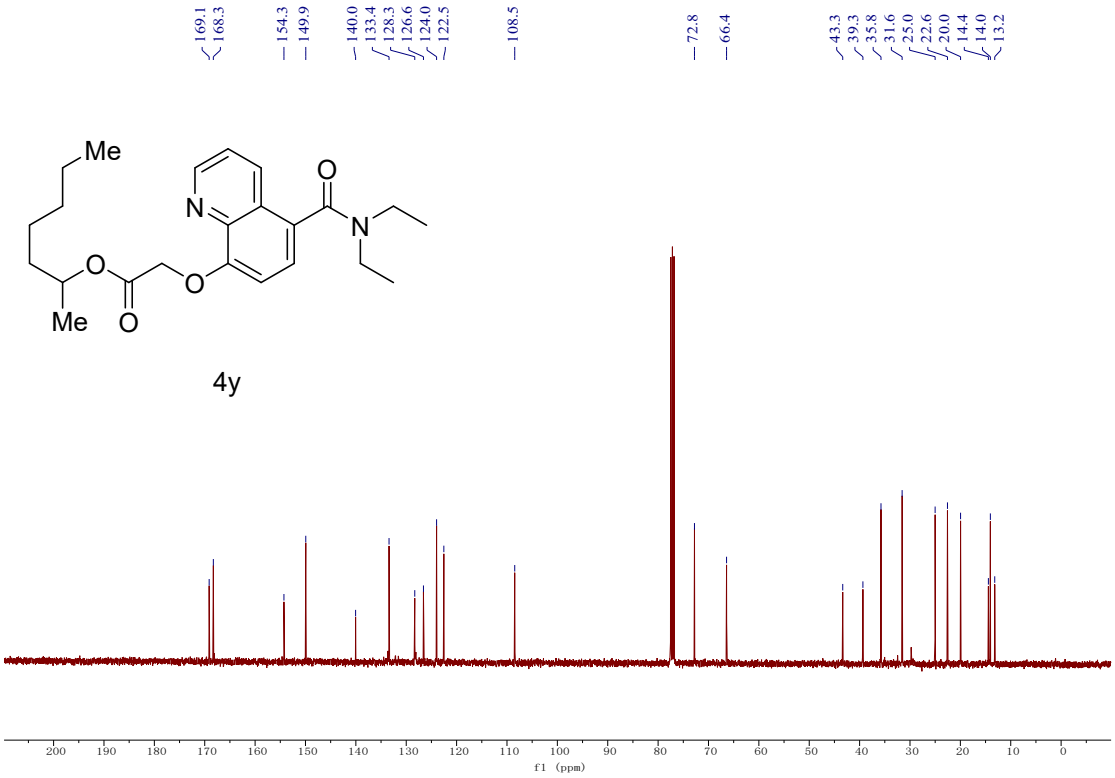
4w

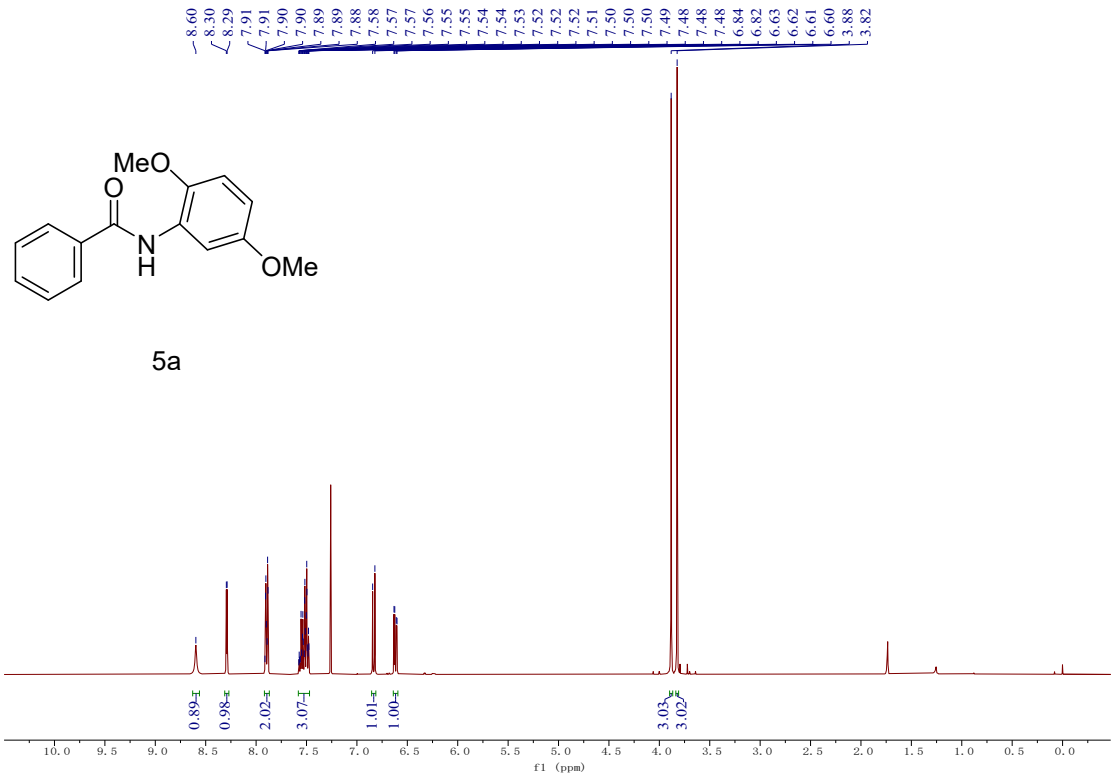

7.76
7.75
7.74
7.73
7.46
7.44
6.86
6.86
6.85
6.84
6.83
6.83
5.12
5.10
5.09
5.07
5.06
5.04
5.02
3.58
3.56
3.55
3.53
3.26
3.25
3.23
3.21
1.64
1.26
1.25
1.23
1.19
1.18
1.12
1.10
1.08
O
O
O
O
N
O
4.04
2.01
2.00
1.05
1.99
2.00
5.99
3.04
6.07
3.08
f1 (ppm)

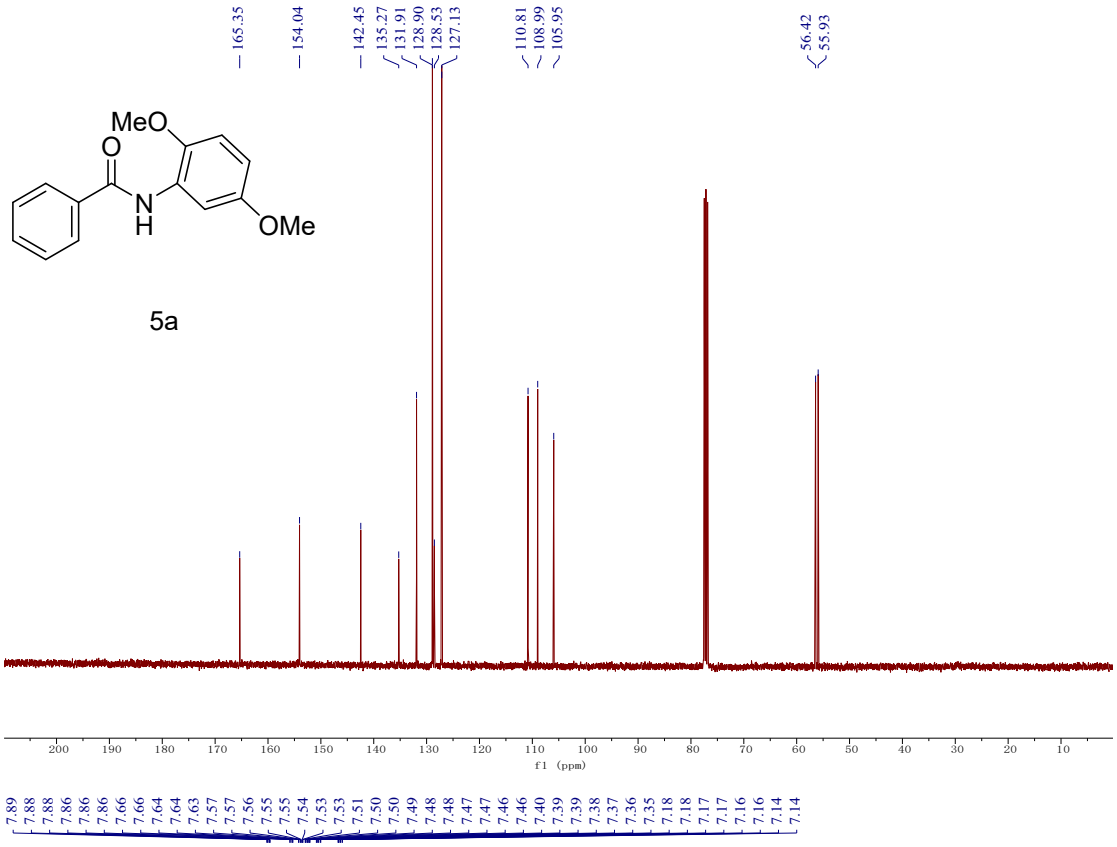
4x

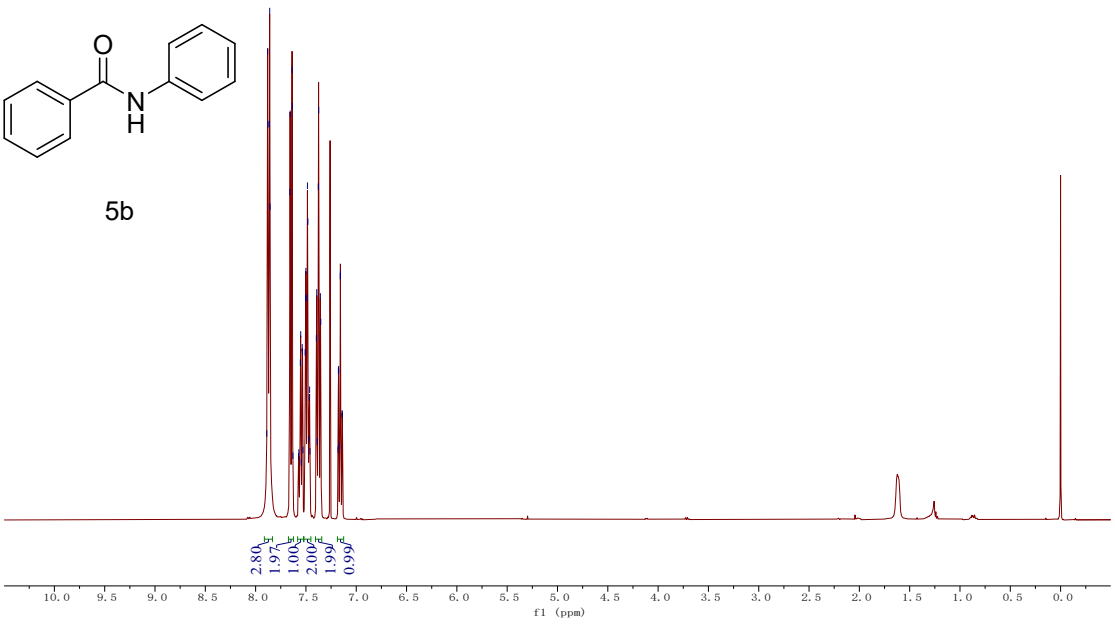

195.0
173.2
170.4
159.9
140.6
138.8
132.2
130.2
129.9
126.2
117.3
79.5
69.4
43.4
39.4
25.5
21.6
14.3
13.0
4x
f1 (ppm)

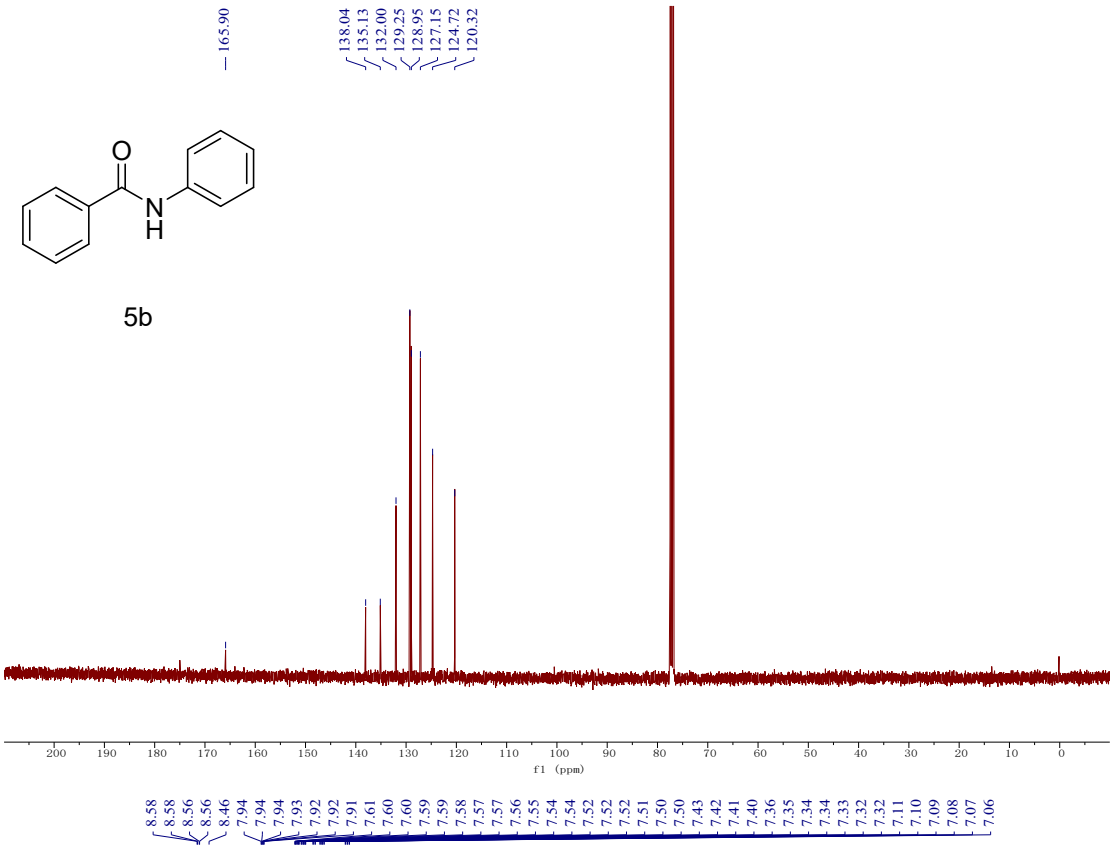

Me
Me
4y
f1 (ppm)

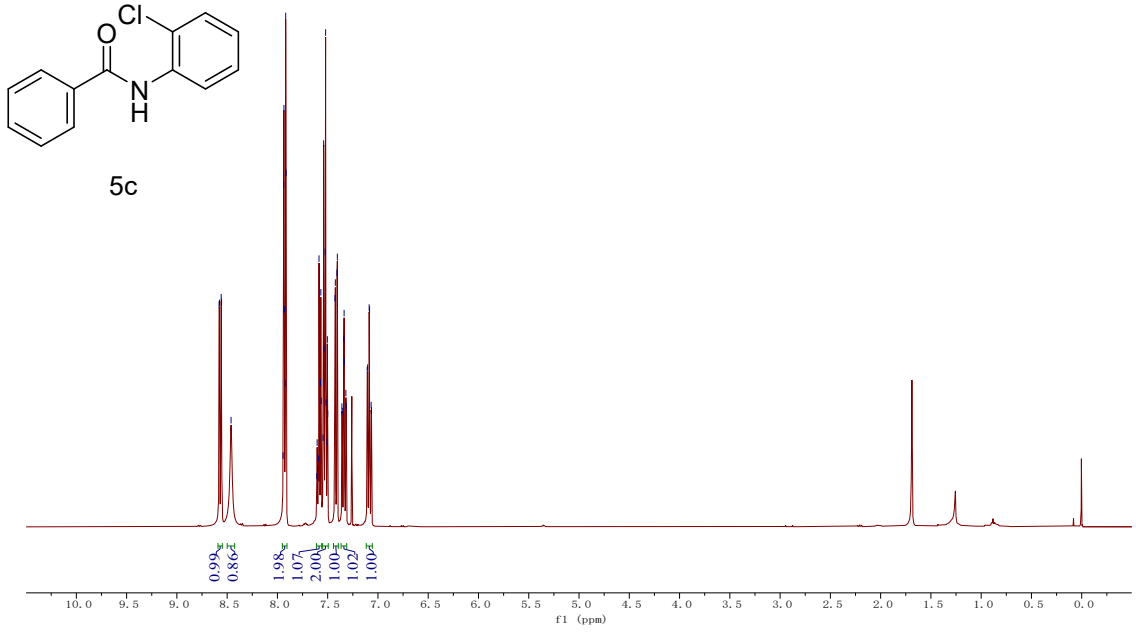

169.1
168.3
154.3
149.9
140.0
133.4
128.3
126.6
124.0
122.5
108.5
72.8
66.4
43.3
39.3
35.8
31.6
25.0
22.6
20.0
14.4
14.0
13.2
Me
O
N
N
O
O
O
Me
4y
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

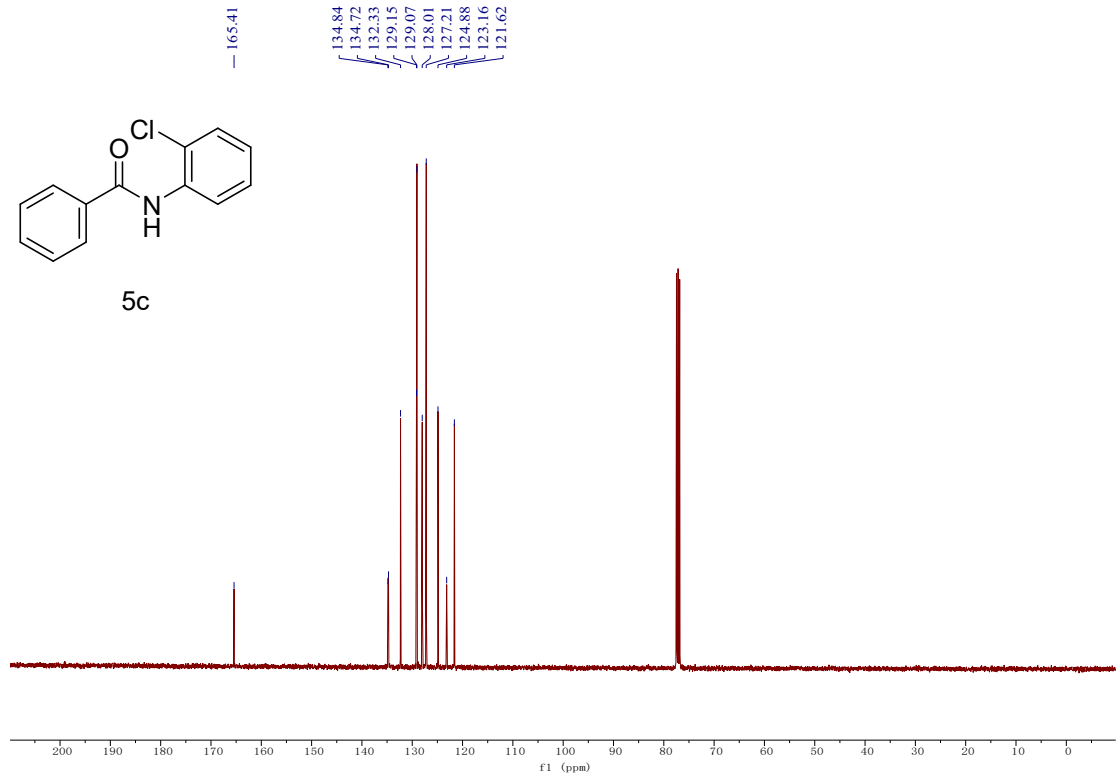

8.60
8.30
8.29
7.91
7.91
7.90
7.90
7.89
7.89
7.88
7.58
7.57
7.57
7.56
7.55
7.55
7.54
7.54
7.53
7.52
7.52
7.52
7.51
7.50
7.50
7.50
7.49
7.48
7.48
6.84
6.82
6.63
6.62
6.61
6.60
3.88
3.82
MeO
O
N
H
OMe
5a
0.89
0.98
2.02
3.07
1.01
1.00
3.03
3.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

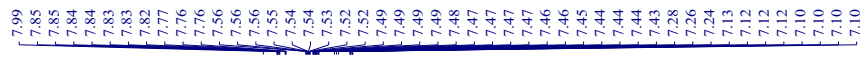

165.35
154.04
142.45
135.27
131.91
128.90
128.53
127.13
110.81
108.99
105.95
56.42
55.93
MeO
O
N
H
OMe
5a
f1 (ppm)

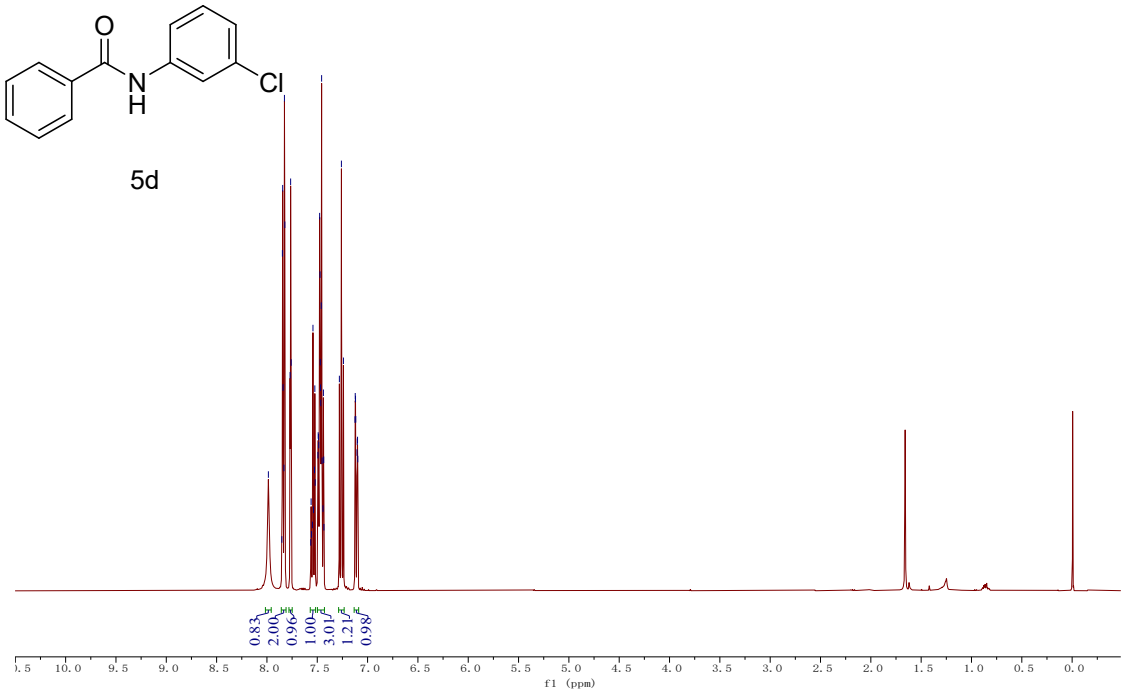

O
N
H
5b
2.80
1.97
1.00
2.00
1.99
0.99
f1 (ppm)

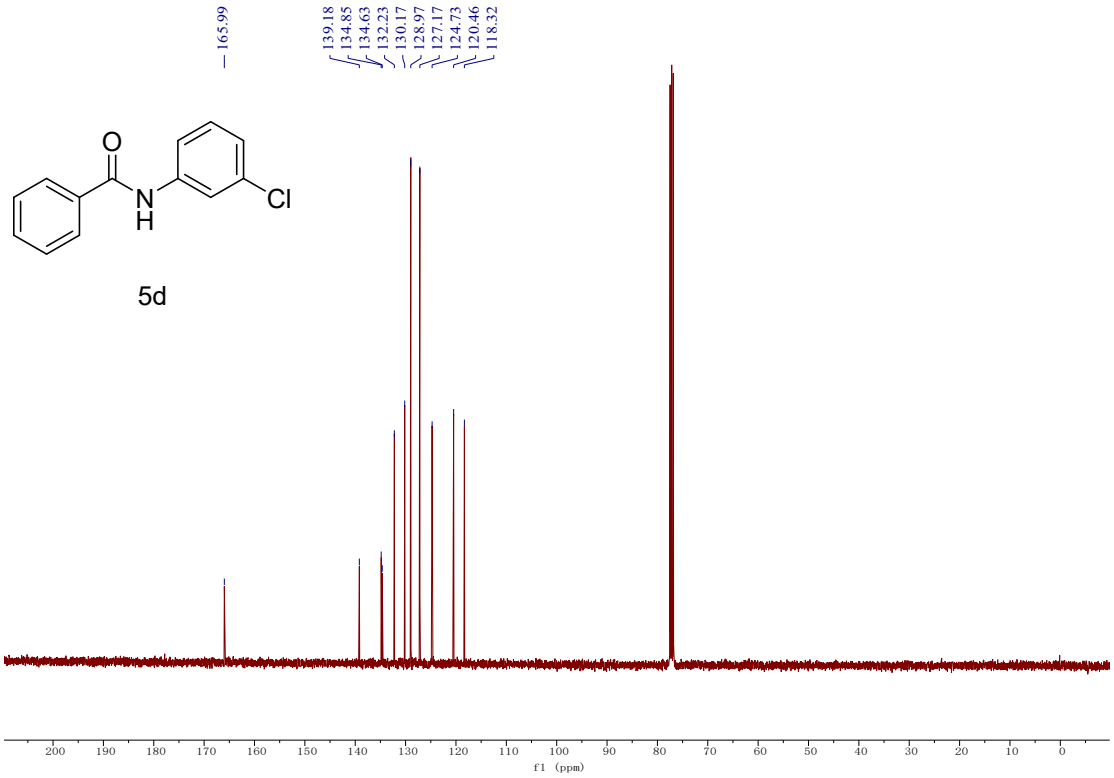

165.90
138.04
135.13
132.00
129.25
128.95
127.15
124.72
120.32
O
N
H
5b
f1 (ppm)
8.58
8.58
8.56
8.56
8.46
7.94
7.94
7.94
7.93
7.92
7.92
7.91
7.61
7.60
7.60
7.59
7.59
7.58
7.57
7.57
7.56
7.55
7.54
7.54
7.52
7.52
7.52
7.51
7.50
7.50
7.43
7.42
7.41
7.40
7.36
7.35
7.34
7.34
7.33
7.32
7.32
7.11
7.10
7.09
7.08
7.07
7.06

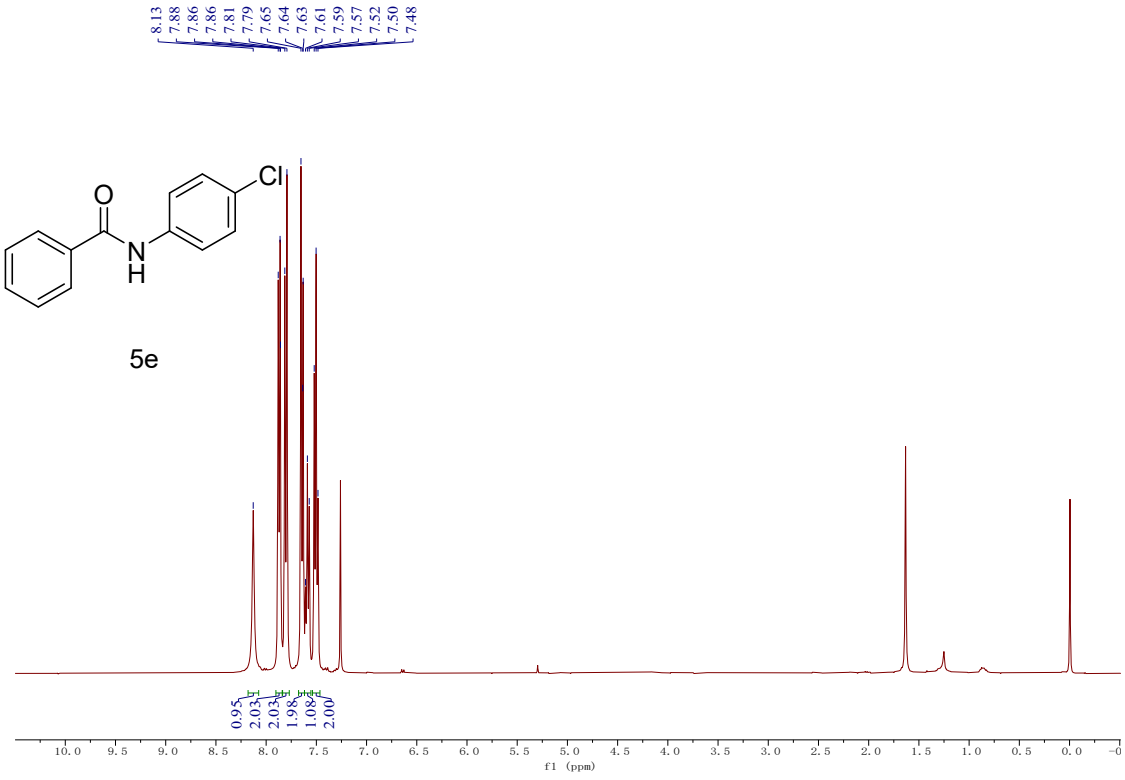

Cl
O
N
H
5c
0.99
0.86
1.98
1.07
2.00
1.00
1.02
1.00
f1 (ppm)

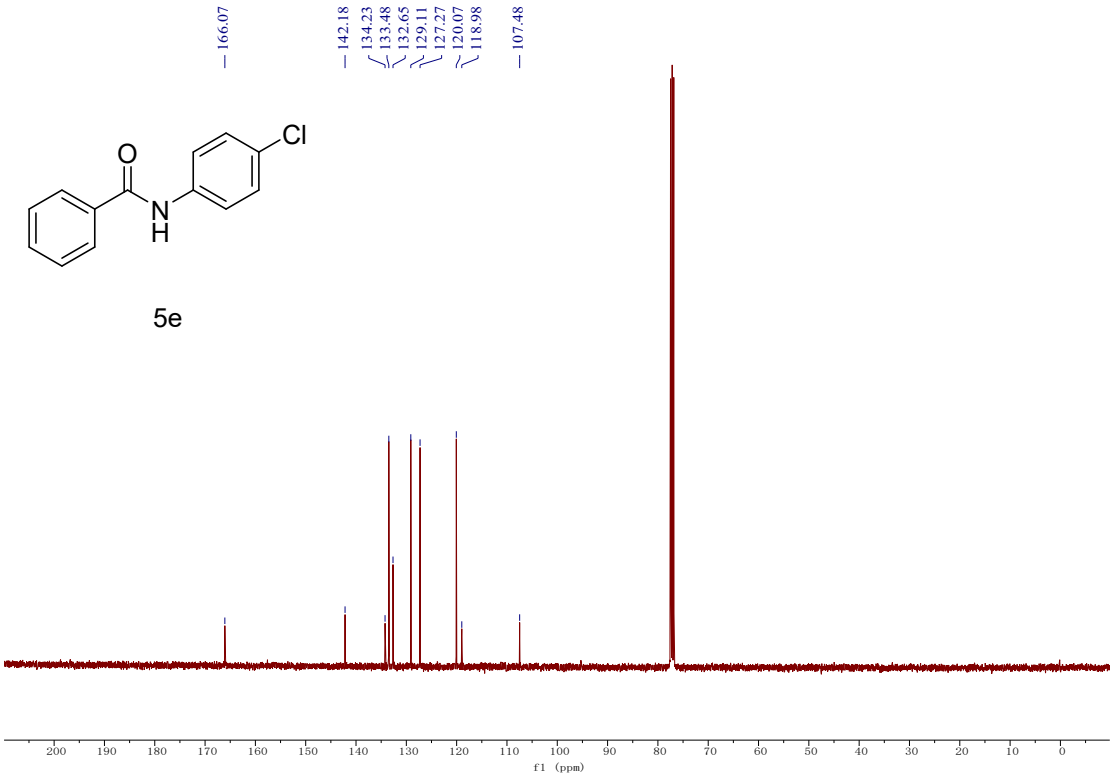

165.41
134.84
134.72
132.33
129.15
129.07
128.01
127.21
124.88
123.16
121.62
Cl
O
N
H
5c
f1 (ppm)

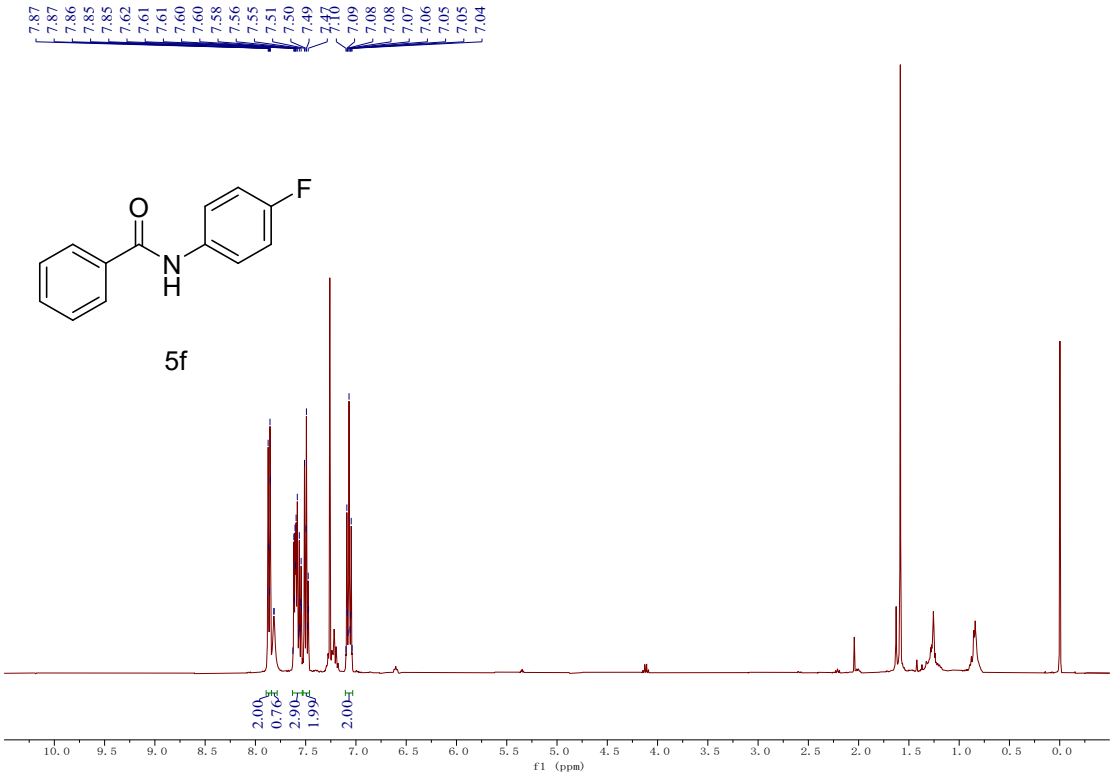

7.99
7.85
7.85
7.84
7.84
7.83
7.83
7.82
7.77
7.76
7.76
7.56
7.56
7.56
7.55
7.54
7.54
7.53
7.52
7.49
7.49
7.49
7.49
7.48
7.47
7.47
7.47
7.47
7.46
7.46
7.45
7.44
7.44
7.44
7.43
7.28
7.26
7.24
7.13
7.12
7.12
7.10
7.10
7.10
7.10

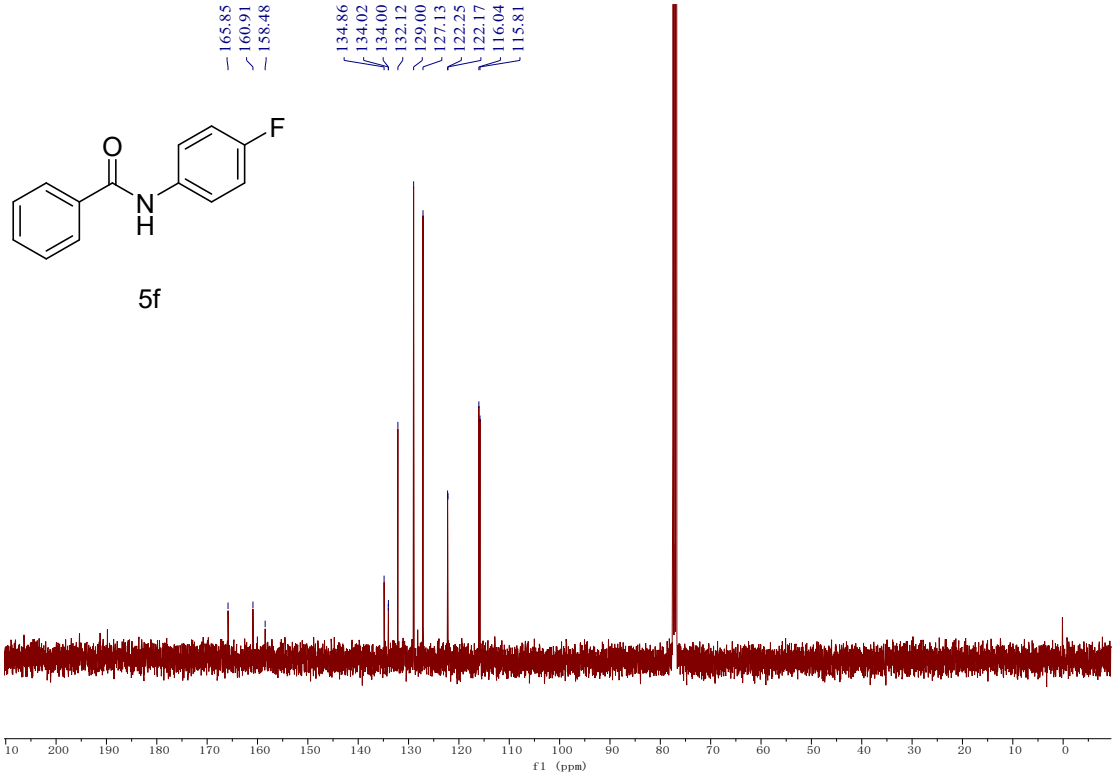

O
N
H
Cl
5d
0.83
2.00
0.96
1.00
3.01
1.21
0.98
f1 (ppm)

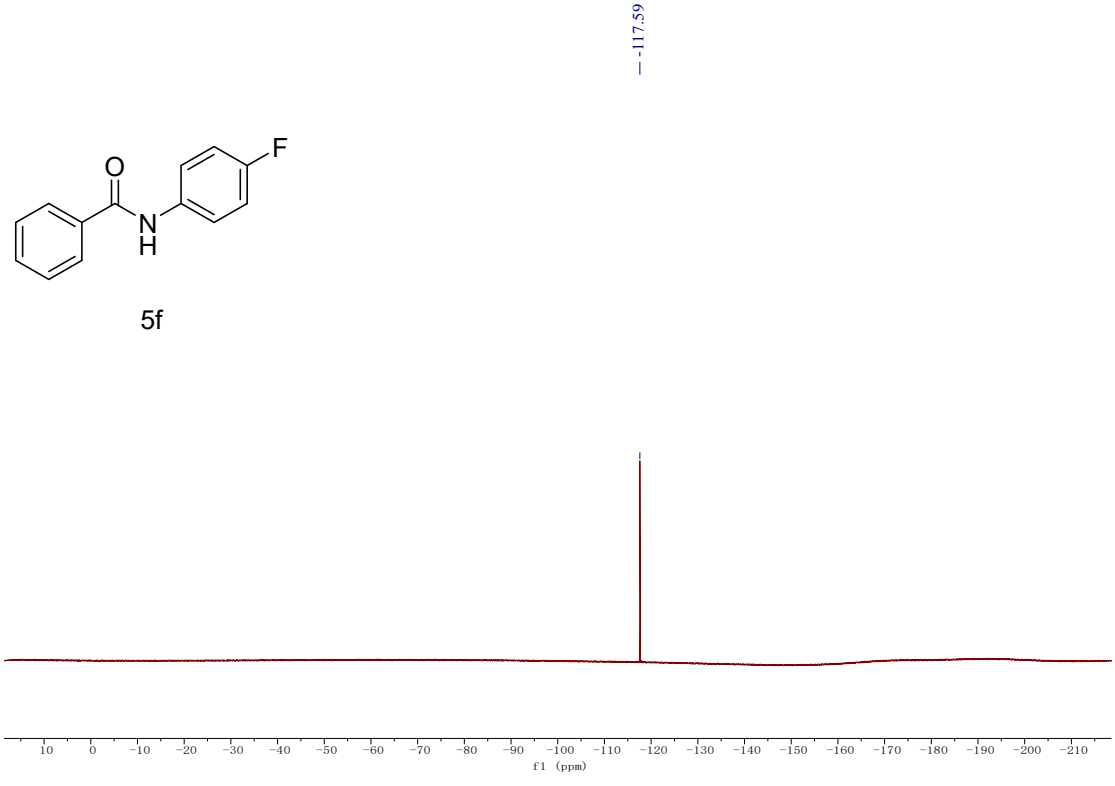

165.99
139.18
134.85
134.63
132.23
130.17
128.97
127.17
124.73
120.46
118.32
O
N
H
Cl
5d
f1 (ppm)

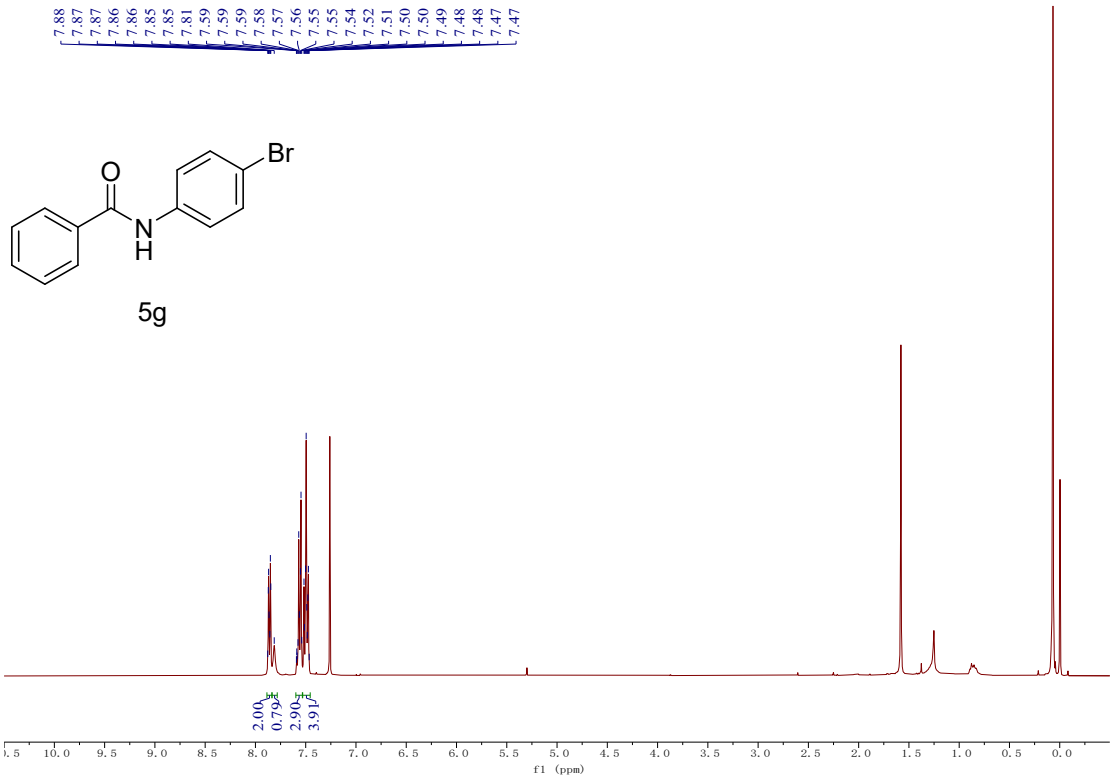

8.13
7.88
7.86
7.86
7.81
7.79
7.65
7.64
7.63
7.61
7.59
7.57
7.52
7.50
7.48
O
N
H
Cl
5e
0.95
2.03
1.98
1.09
2.00
f1 (ppm)

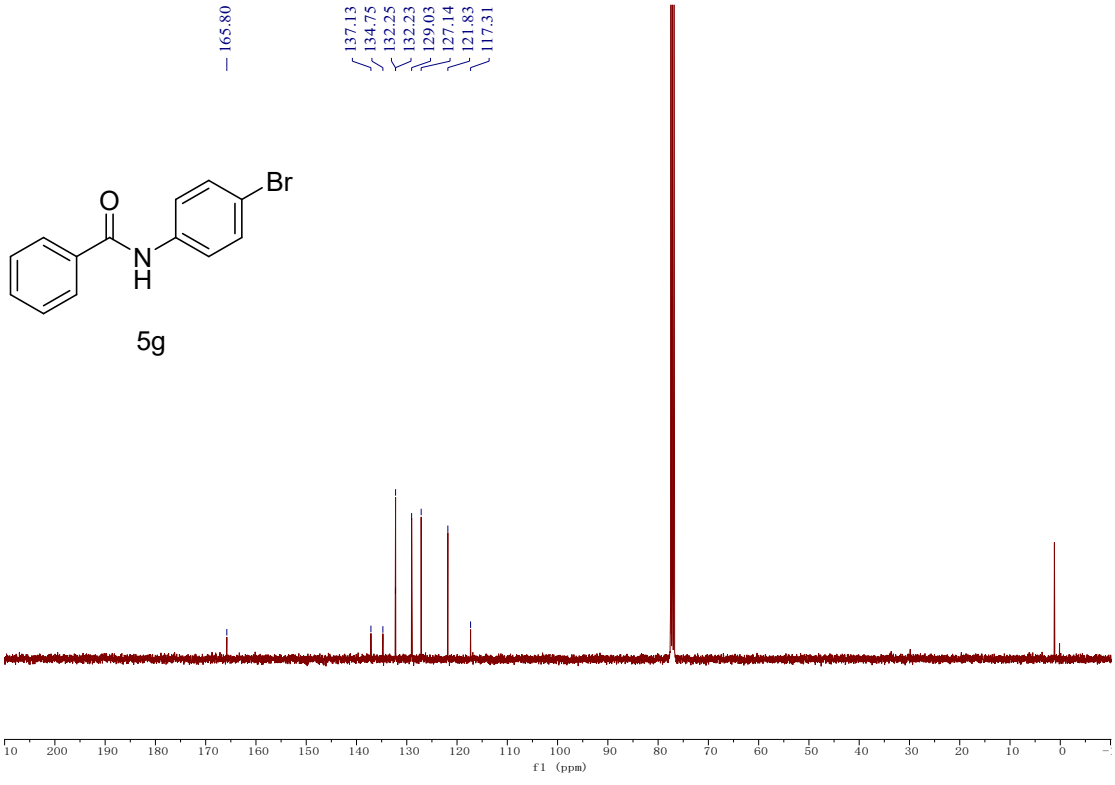

166.07
142.18
134.23
133.48
132.65
129.11
127.27
120.07
118.98
107.48
O
N
H
Cl
5e
f1 (ppm)

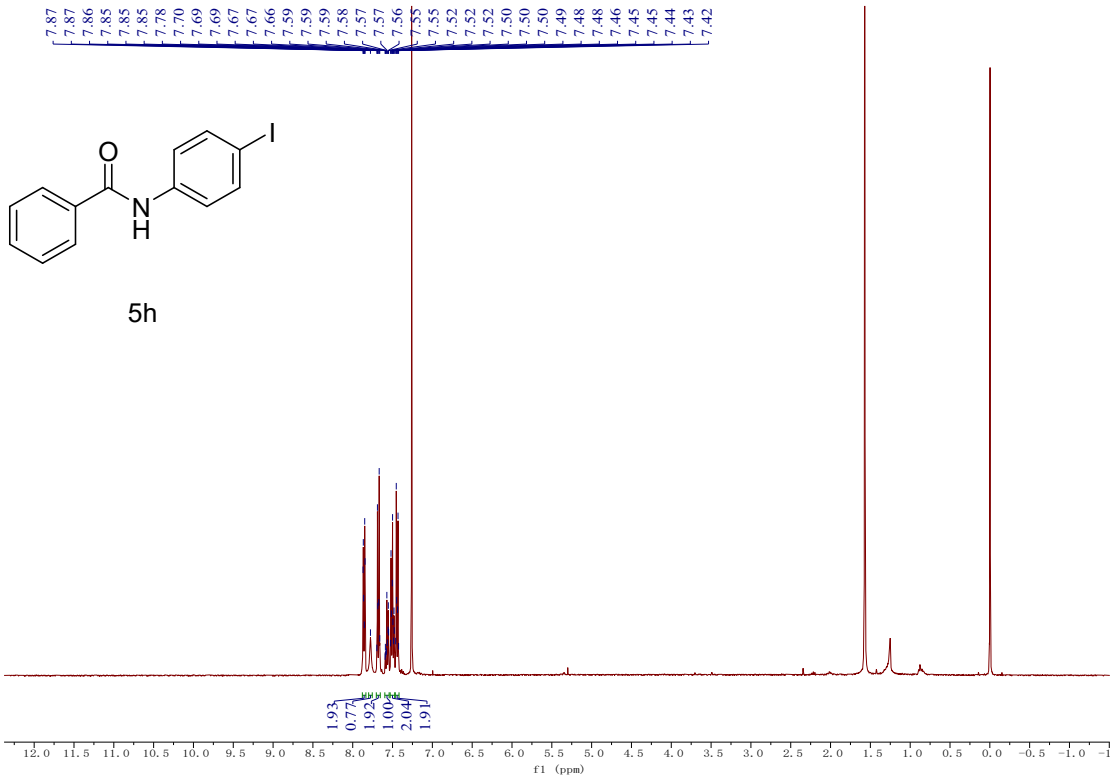

7.87
7.87
7.86
7.85
7.85
7.62
7.61
7.61
7.60
7.60
7.58
7.56
7.55
7.51
7.50
7.49
7.47
7.10
7.09
7.08
7.08
7.07
7.06
7.05
7.05
7.04
O
N
H
F
5f
2.00
0.76
2.90
1.99
2.00
f1 (ppm)

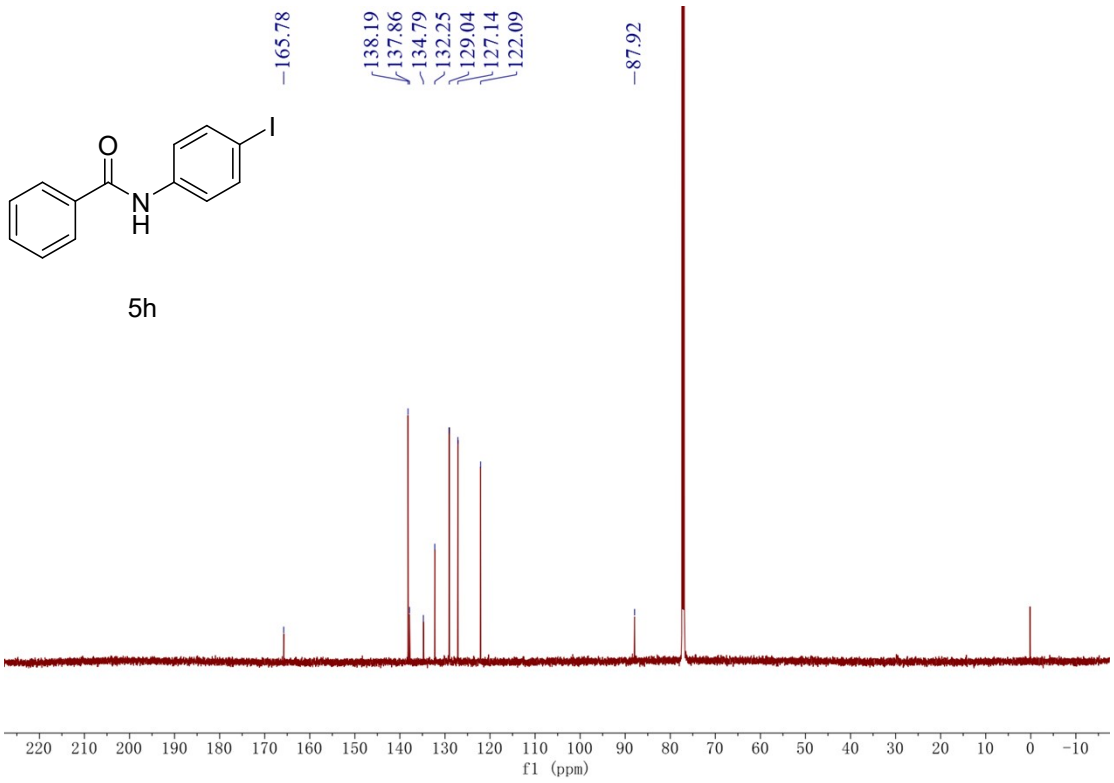

165.85
160.91
158.48
134.86
134.02
134.00
132.12
129.00
127.13
122.25
122.17
116.04
115.81
O
N
H
F
5f
f1 (ppm)

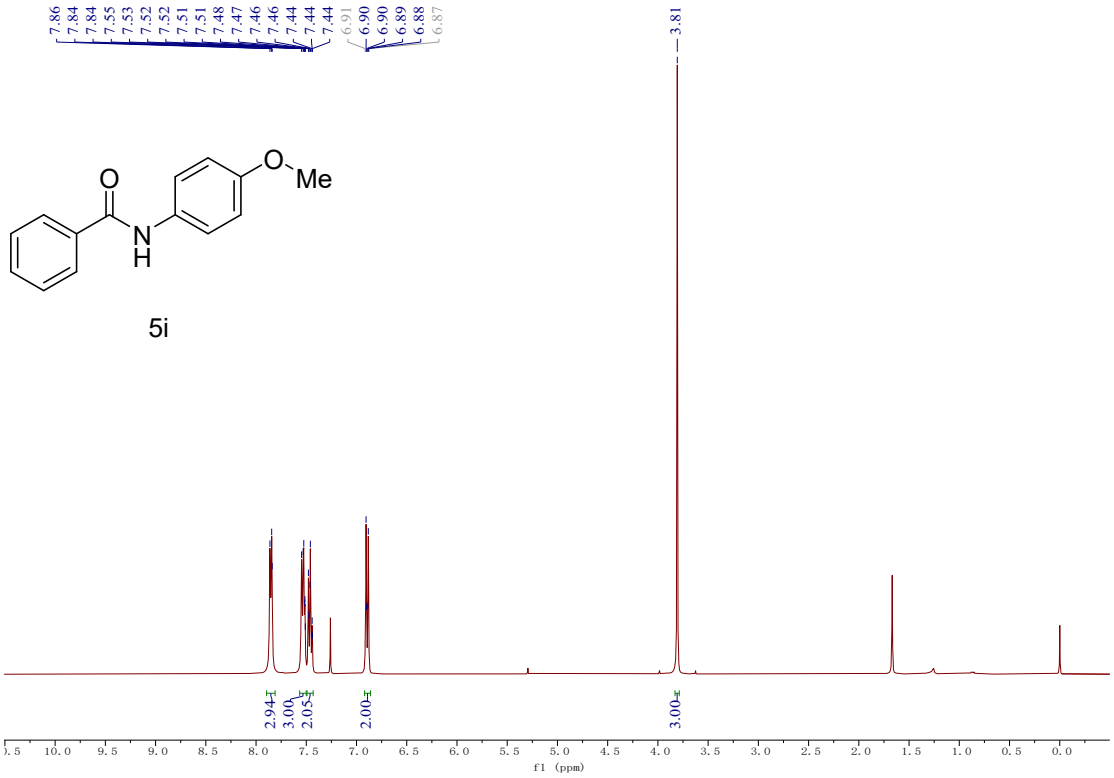

-117.59
O
N
H
F
5f
f1 (ppm)


7.88
7.87
7.87
7.86
7.86
7.85
7.85
7.81
7.59
7.59
7.59
7.58
7.57
7.56
7.55
7.55
7.54
7.52
7.51
7.50
7.50
7.49
7.48
7.48
7.47
7.47
Br
O
N
H
5g
2.00
0.79
2.90
3.91
f1 (ppm)

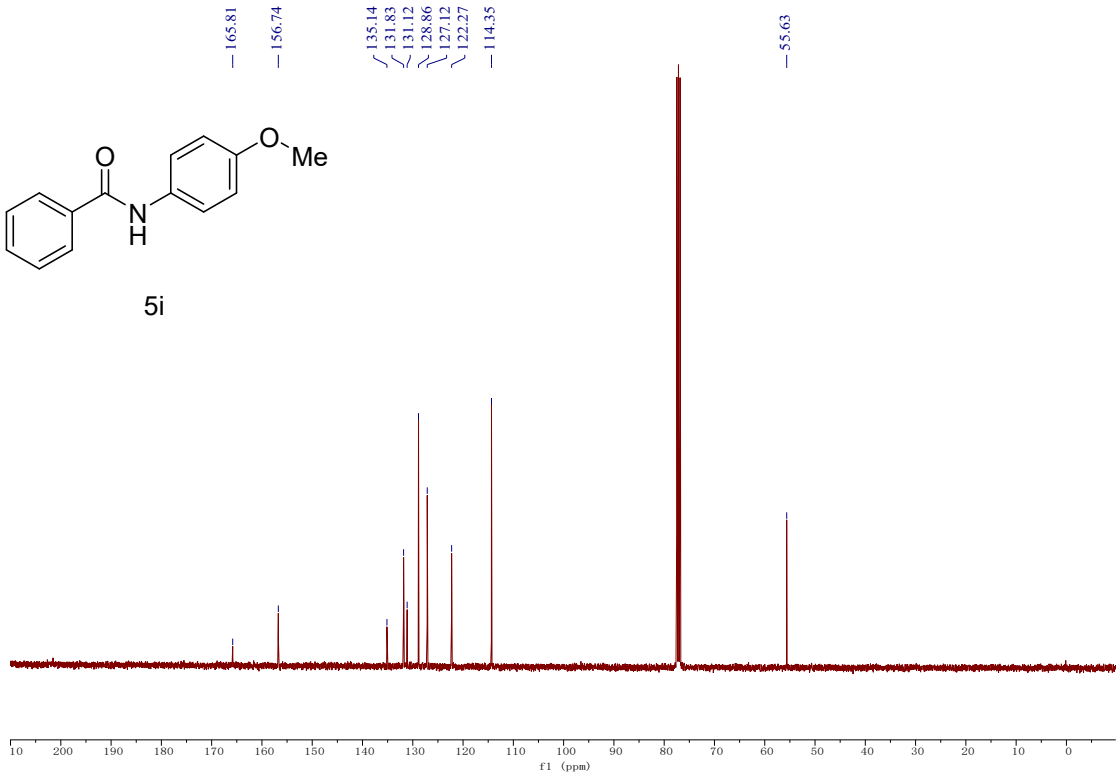

165.80
137.13
134.75
132.25
132.23
129.03
127.14
121.83
117.31
Br
O
N
H
5g
f1 (ppm)


7.87
7.87
7.86
7.85
7.85
7.85
7.78
7.70
7.69
7.69
7.67
7.67
7.66
7.59
7.59
7.59
7.58
7.57
7.57
7.56
7.55
7.55
7.52
7.52
7.52
7.50
7.50
7.50
7.49
7.48
7.48
7.46
7.45
7.45
7.44
7.43
7.42
O
N
H
I
5h
1.93
0.77
1.92
1.00
2.04
1.91
f1 (ppm)

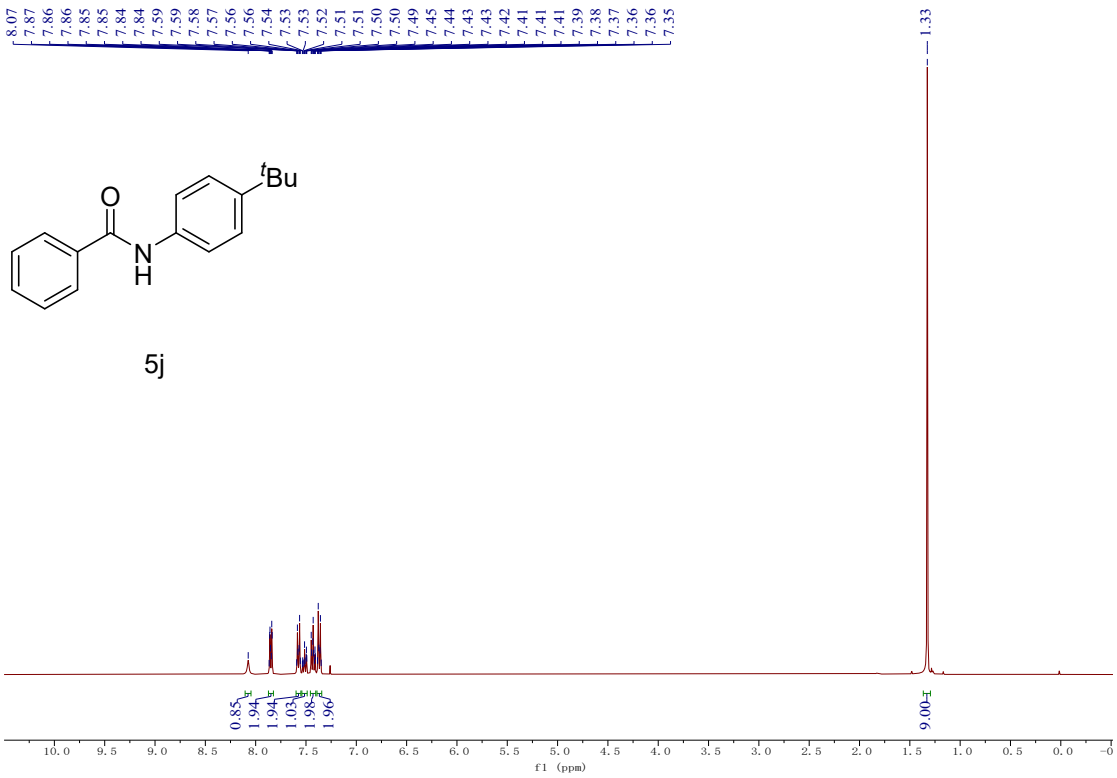

165.78
138.19
137.86
134.79
132.25
129.04
127.14
122.09
87.92
O
N
H
I
5h
f1 (ppm)


7.86
7.84
7.84
7.55
7.53
7.52
7.52
7.51
7.51
7.48
7.47
7.46
7.46
7.44
7.44
7.44
6.91
6.90
6.90
6.89
6.88
6.87
3.81
O
Me
N
H
2.94
3.00
2.05
2.00
3.00
f1 (ppm)

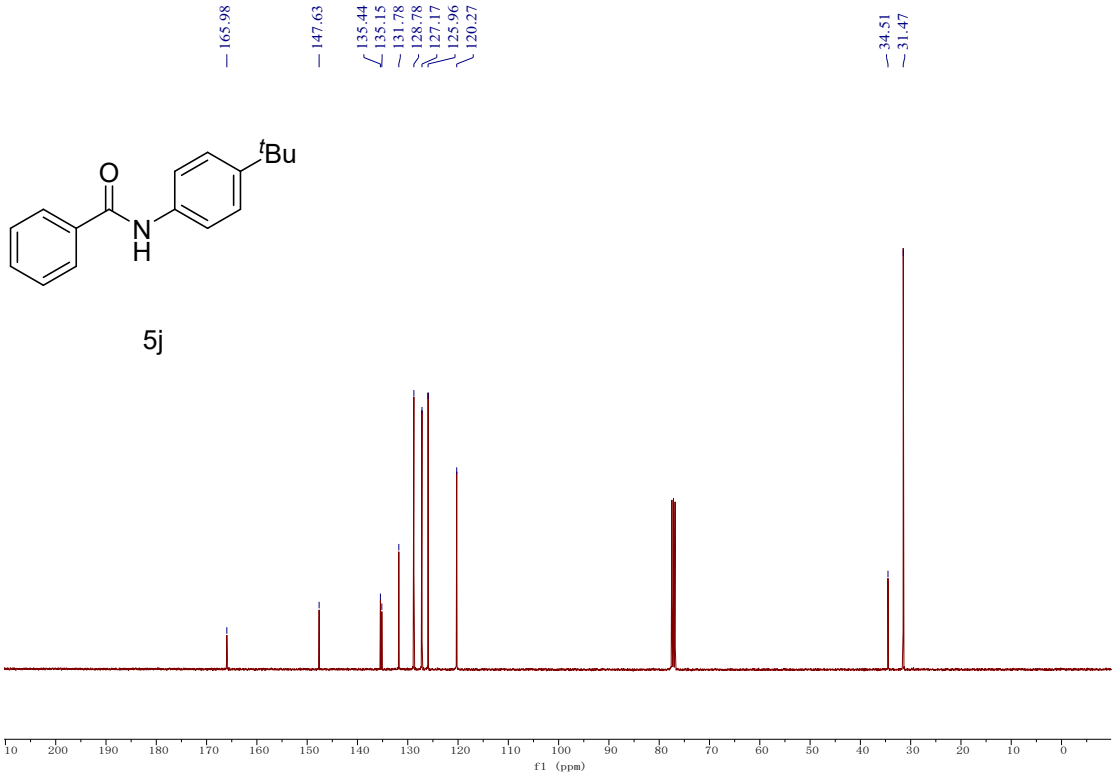
5i


165.81
156.74
135.14
131.83
131.12
128.86
127.12
122.27
114.35
55.63
O
Me
N
H
f1 (ppm)

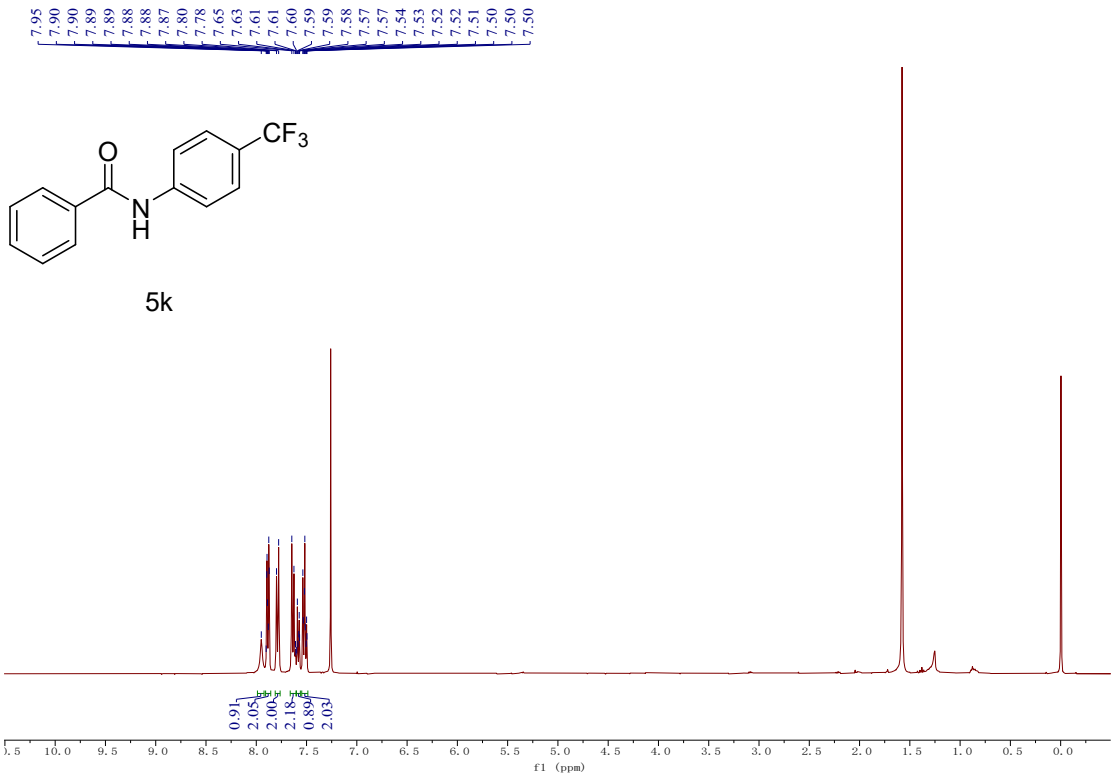
5i

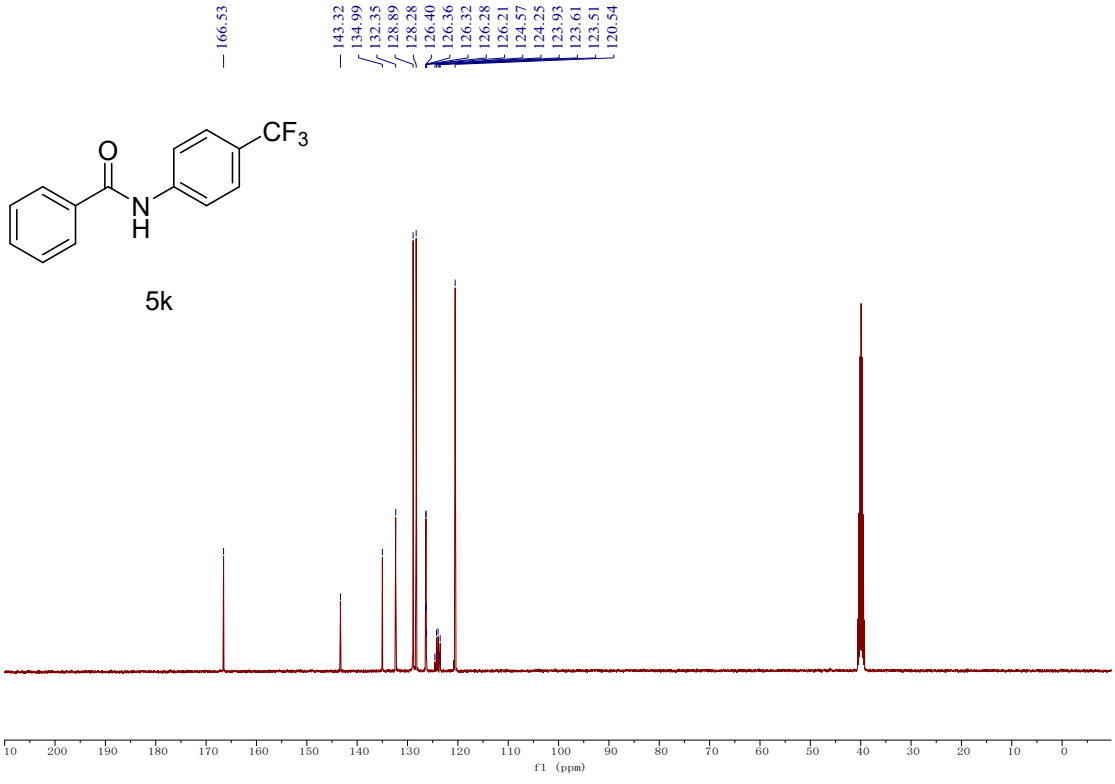

8.07
7.87
7.86
7.86
7.85
7.85
7.84
7.84
7.59
7.59
7.58
7.57
7.56
7.56
7.54
7.53
7.53
7.52
7.51
7.51
7.50
7.50
7.49
7.45
7.44
7.43
7.43
7.42
7.41
7.41
7.41
7.39
7.38
7.37
7.36
7.36
7.35
1.33
tBu
O
N
H
0.85
1.94
1.94
1.03
1.98
1.96
9.00
f1 (ppm)

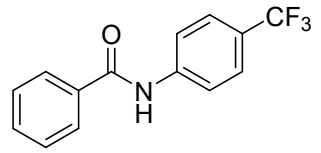
5j


165.98
147.63
135.44
135.15
131.78
128.78
127.17
125.96
120.27
34.51
31.47
tBu
O
N
H
f1 (ppm)

5j

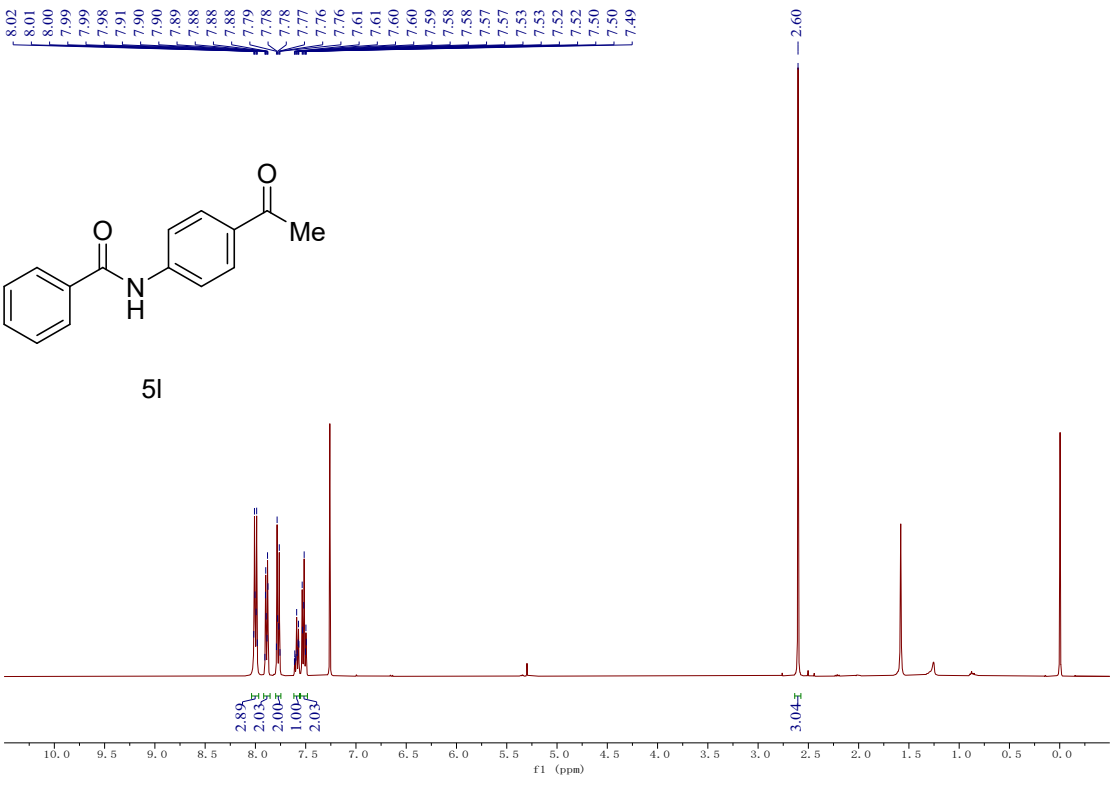

7.95
7.90
7.90
7.89
7.89
7.88
7.88
7.87
7.80
7.78
7.65
7.63
7.61
7.61
7.60
7.59
7.59
7.58
7.57
7.57
7.54
7.53
7.52
7.52
7.51
7.50
7.50
7.50
O
N
H
CF3
5k
0.91
2.05
2.00
2.18
0.89
2.03
f1 (ppm)


166.53
143.32
134.99
132.35
128.89
128.28
126.40
126.36
126.32
126.28
126.21
124.57
124.25
123.93
123.61
123.51
120.54
O
N
H
CF3
5k
f1 (ppm)


O
N
H
CF3

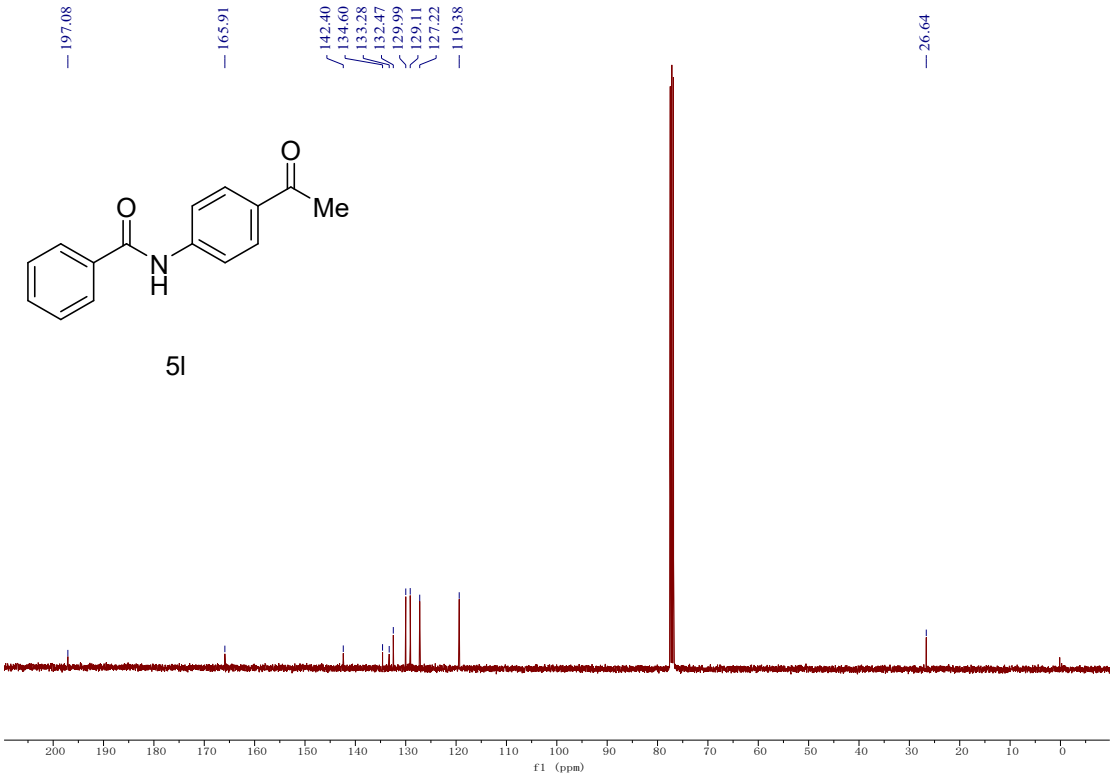
5k

-62.11
f1 (ppm)

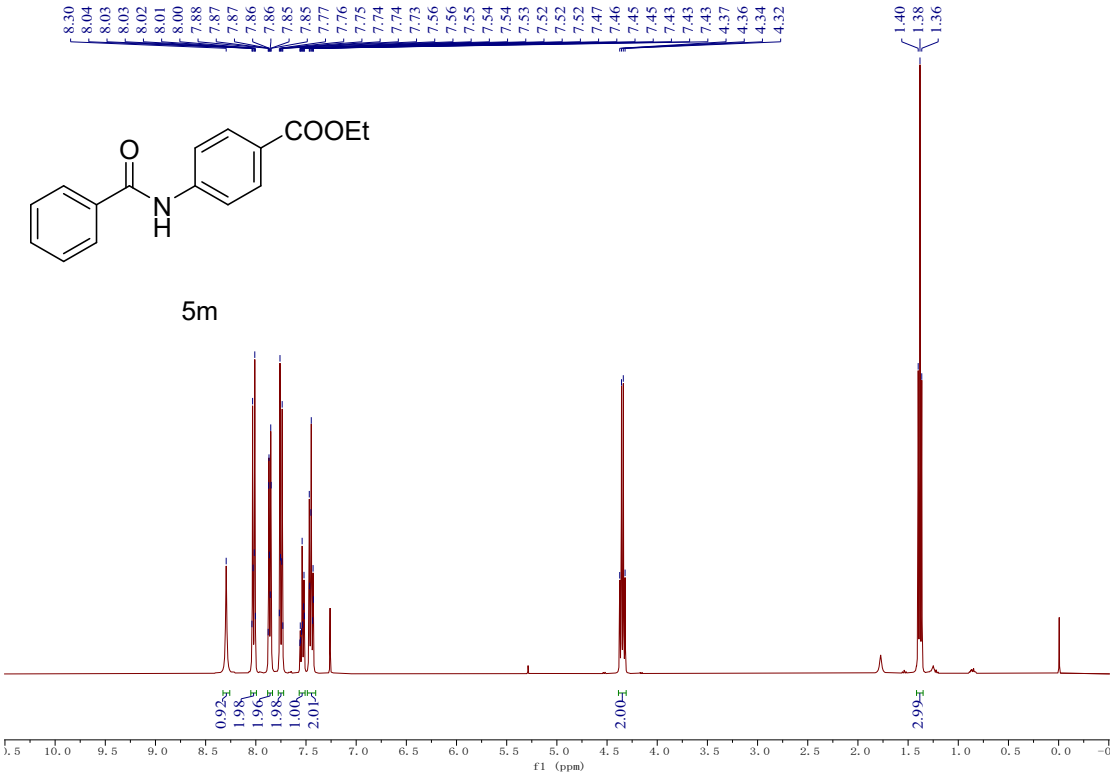

8.02
8.01
8.00
7.99
7.99
7.98
7.91
7.90
7.90
7.90
7.89
7.88
7.88
7.88
7.79
7.78
7.78
7.77
7.76
7.76
7.76
7.61
7.61
7.60
7.60
7.60
7.59
7.58
7.58
7.57
7.57
7.57
7.53
7.53
7.52
7.52
7.50
7.50
7.49
2.60
2.89
2.03
2.00
1.00
2.03
3.04
f1 (ppm)

O
O
N
H
Me

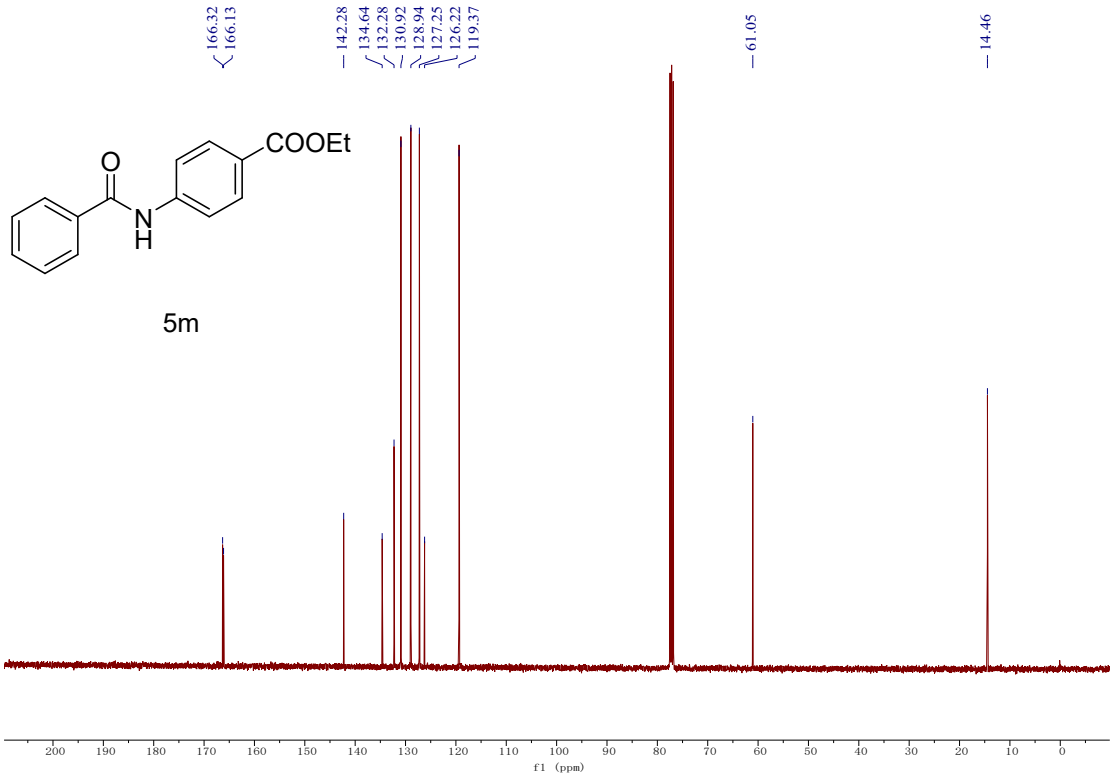
5l

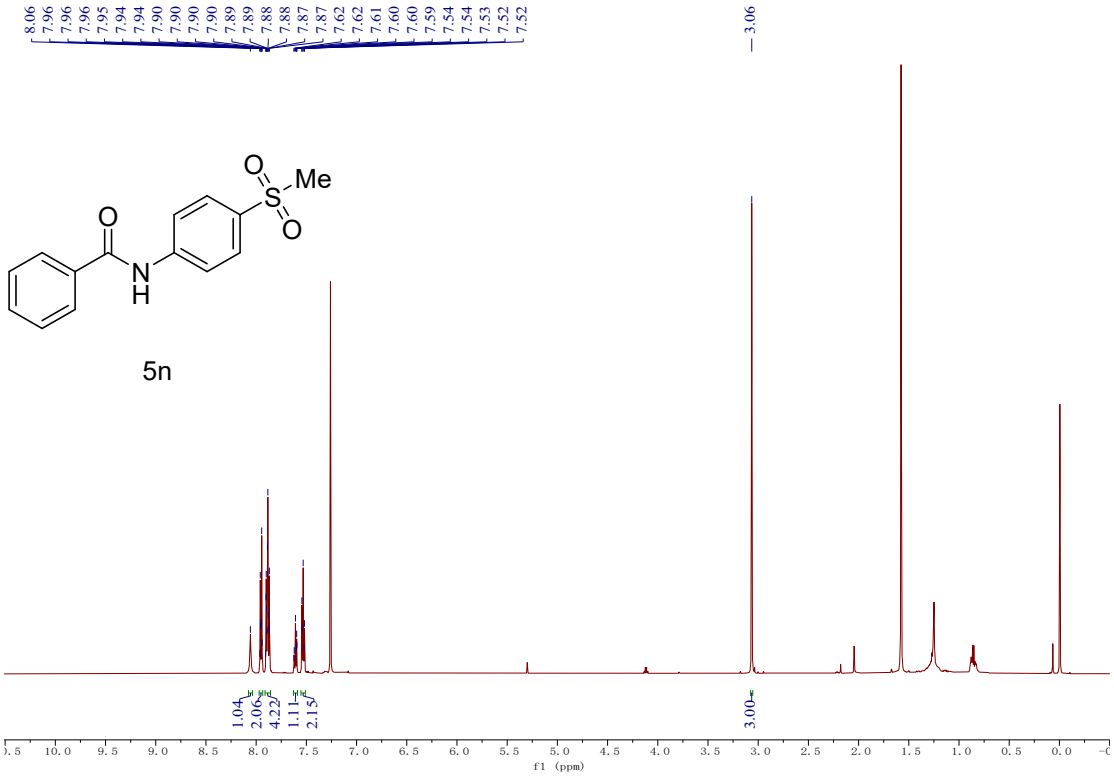

197.08
165.91
142.40
134.60
133.28
132.47
129.99
129.11
127.22
119.38
26.64
O
Me
O
N
H
f1 (ppm)

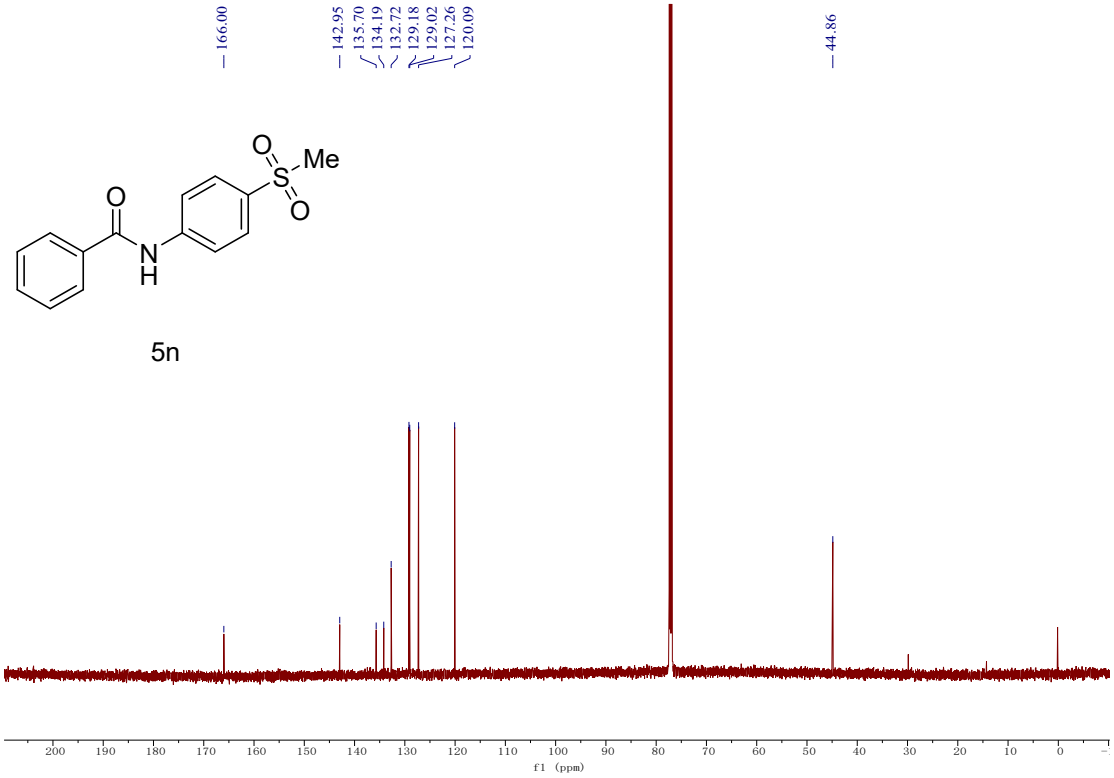
5l

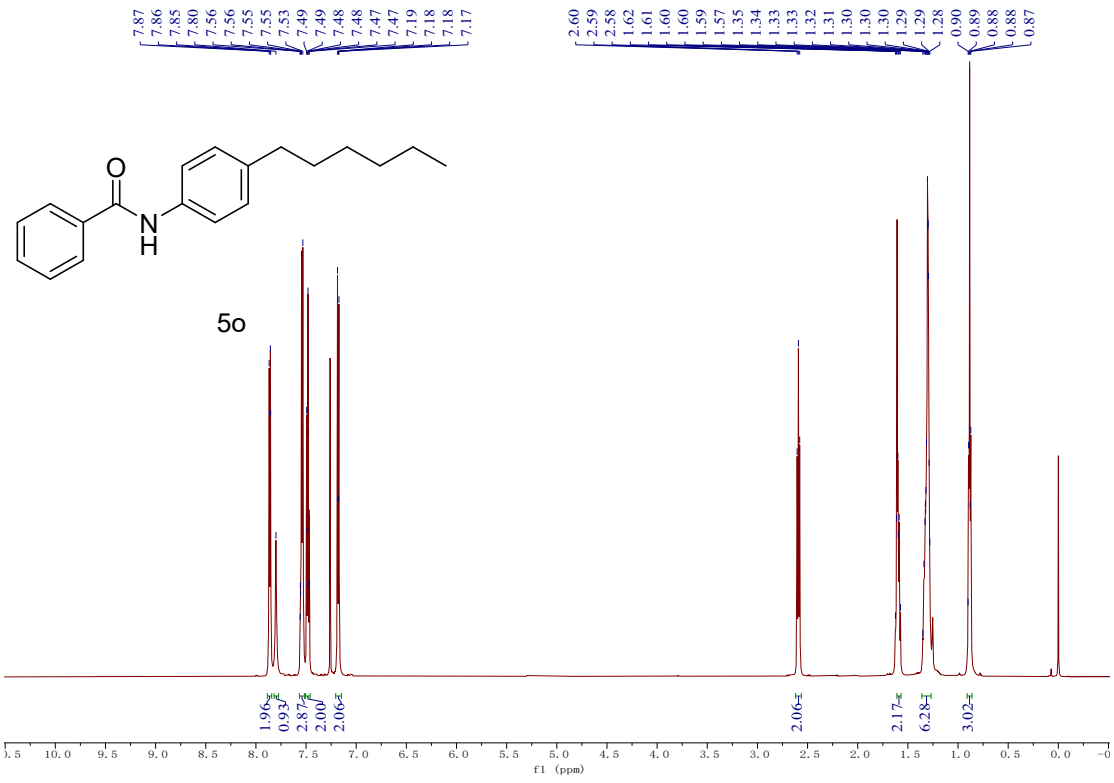

8.30
8.04
8.03
8.03
8.02
8.01
8.00
7.88
7.87
7.87
7.86
7.86
7.85
7.85
7.77
7.76
7.75
7.74
7.74
7.73
7.56
7.56
7.55
7.54
7.54
7.53
7.52
7.52
7.52
7.47
7.46
7.45
7.45
7.43
7.43
7.43
4.37
4.36
4.34
4.32
1.40
1.38
1.36
COOEt
O
N
H
0.92
1.98
1.96
1.98
1.00
2.01
2.00
2.99
f1 (ppm)

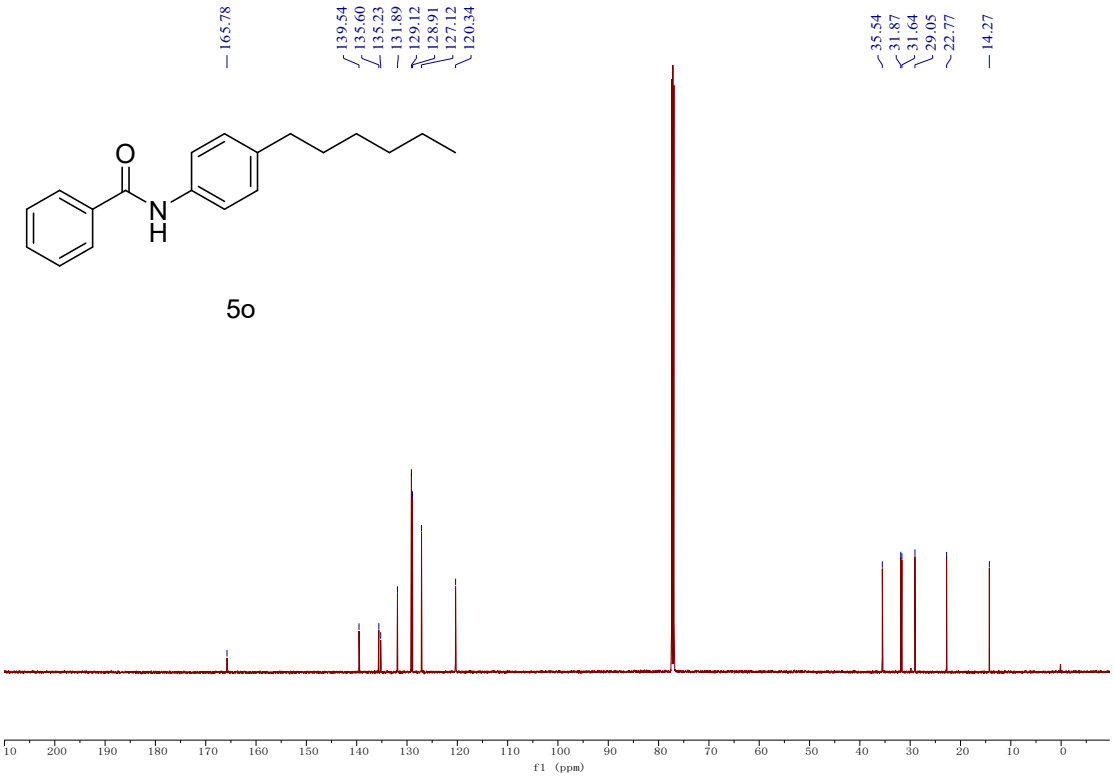
5m

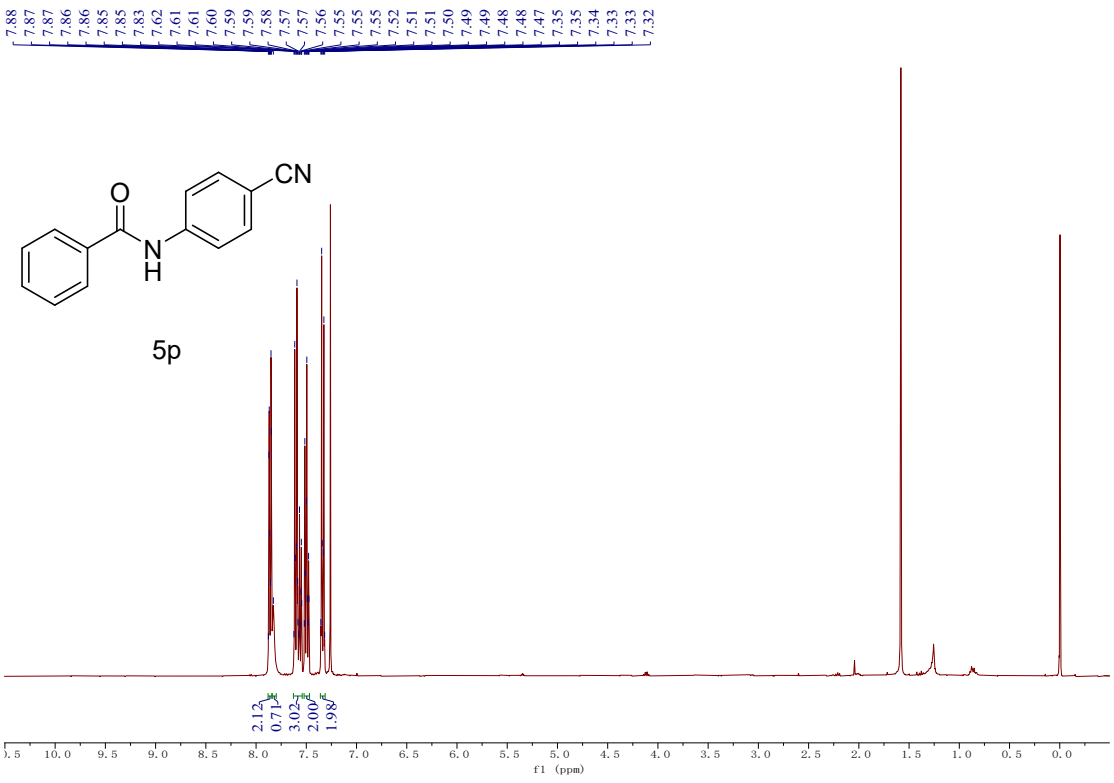

166.32
166.13
142.28
134.64
132.28
130.92
128.94
127.25
126.22
119.37
61.05
14.46
COOEt
O
N
H
5m
f1 (ppm)

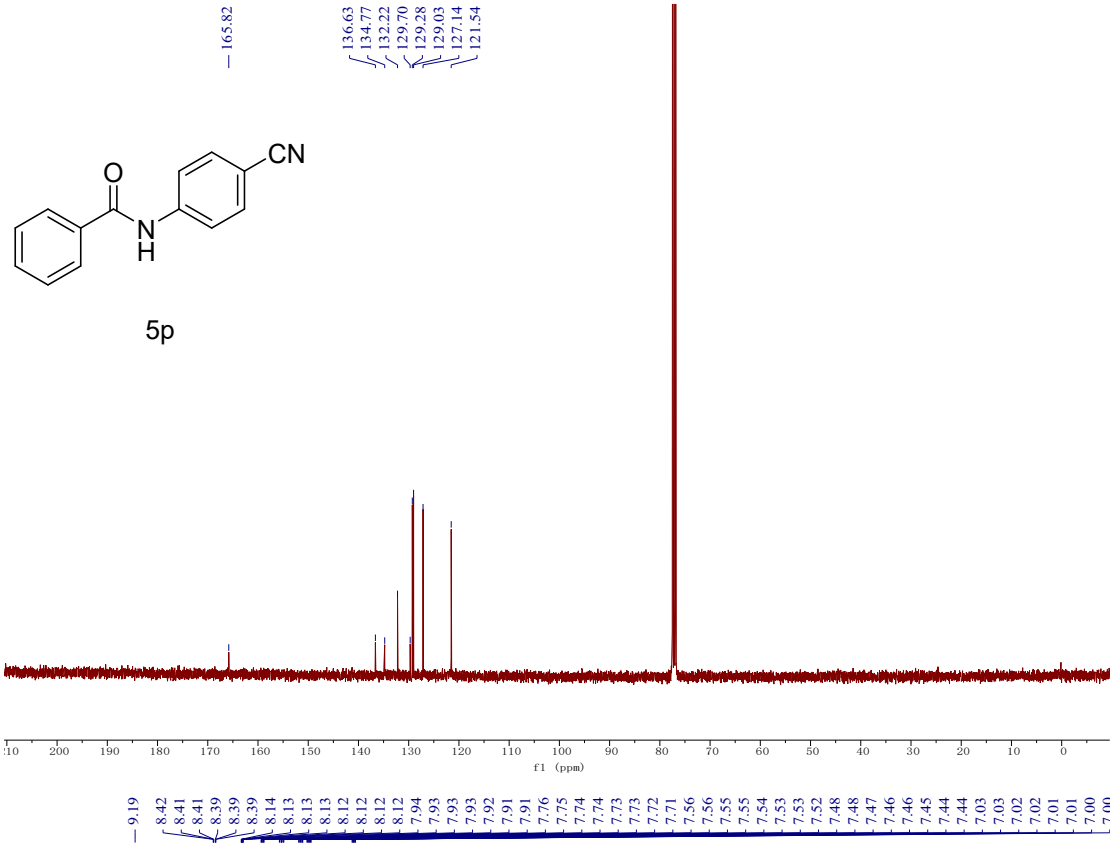

8.06
7.96
7.96
7.96
7.95
7.94
7.94
7.90
7.90
7.90
7.90
7.89
7.89
7.88
7.88
7.87
7.87
7.62
7.62
7.61
7.60
7.60
7.59
7.54
7.54
7.53
7.52
7.52
3.06
O
Me
S
O
O
N
H
5n
1.04
2.06
4.22
1.11
2.15
3.00
f1 (ppm)


166.00
142.95
135.70
134.19
132.72
129.18
129.02
127.26
120.09
44.86
O
S
Me
O
O
N
H
5n
f1 (ppm)


7.87
7.86
7.85
7.80
7.56
7.56
7.55
7.55
7.53
7.49
7.49
7.48
7.48
7.47
7.47
7.19
7.18
7.18
7.17
2.60
2.59
2.58
1.62
1.61
1.60
1.60
1.59
1.57
1.35
1.34
1.33
1.33
1.32
1.31
1.30
1.30
1.30
1.29
1.29
1.28
0.90
0.89
0.88
0.88
0.87
O
N
H
5o
1.96
0.93
2.87
2.00
2.06
2.06
2.17
6.28
3.02
f1 (ppm)

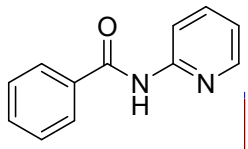

165.78
139.54
135.60
135.23
131.89
129.12
128.91
127.12
120.34
35.54
31.87
31.64
29.05
22.77
14.27
O
N
H
5o
f1 (ppm)


7.88
7.87
7.87
7.86
7.86
7.85
7.85
7.83
7.62
7.61
7.61
7.60
7.59
7.59
7.58
7.57
7.57
7.56
7.55
7.55
7.55
7.52
7.51
7.51
7.50
7.49
7.49
7.48
7.48
7.47
7.35
7.35
7.34
7.33
7.33
7.32
CN
O
N
H
5p
2.12
0.71
3.02
2.00
1.98
f1 (ppm)


165.82
136.63
134.77
132.22
129.70
129.28
129.03
127.14
121.54
O
N
H
CN
f1 (ppm)

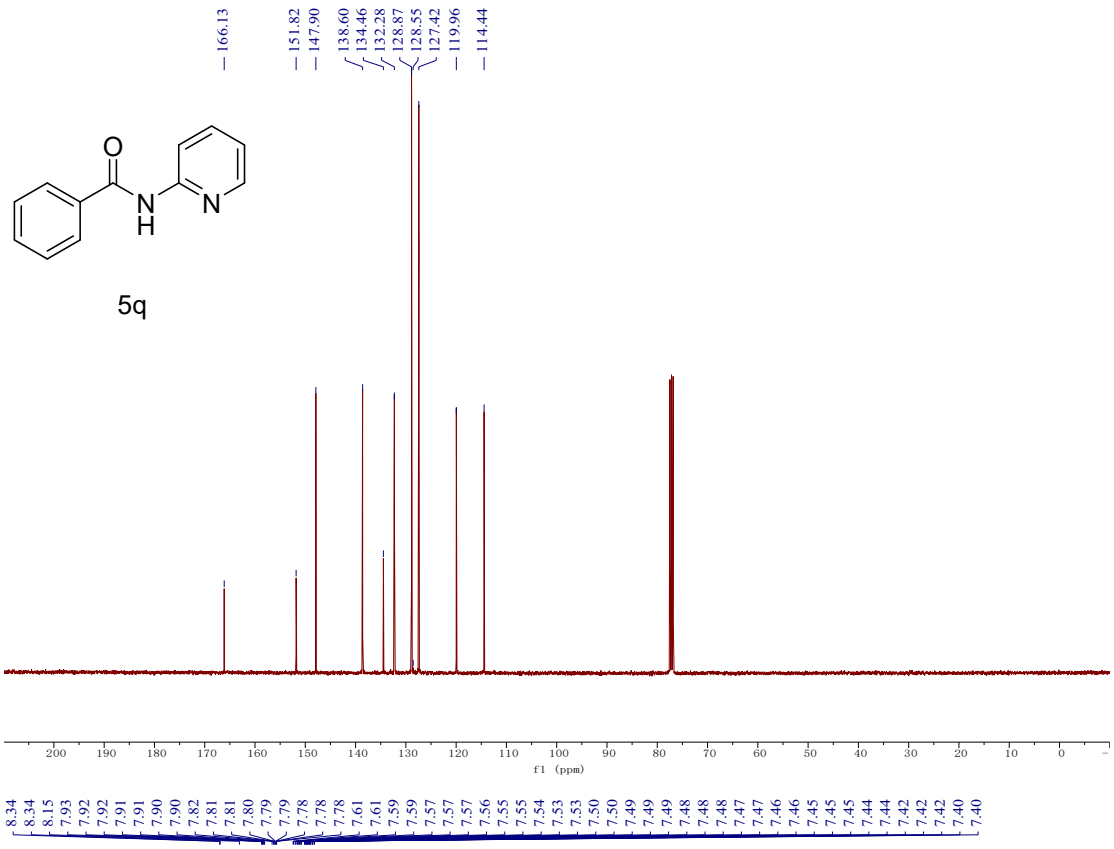
5p

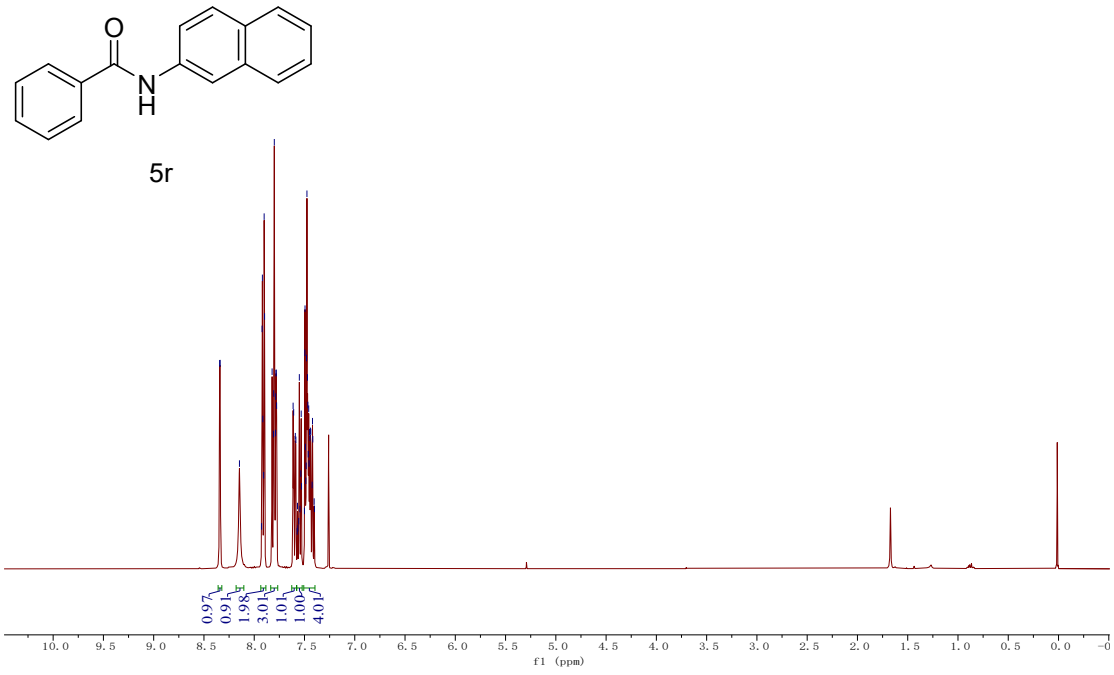
9.19
8.42
8.41
8.41
8.39
8.39
8.39
8.14
8.13
8.13
8.12
8.12
8.12
8.12
7.94
7.93
7.93
7.93
7.92
7.91
7.91
7.76
7.75
7.74
7.74
7.73
7.73
7.72
7.71
7.56
7.56
7.55
7.55
7.54
7.53
7.53
7.52
7.48
7.48
7.47
7.46
7.46
7.45
7.44
7.44
7.03
7.03
7.02
7.02
7.01
7.01
7.00
7.00

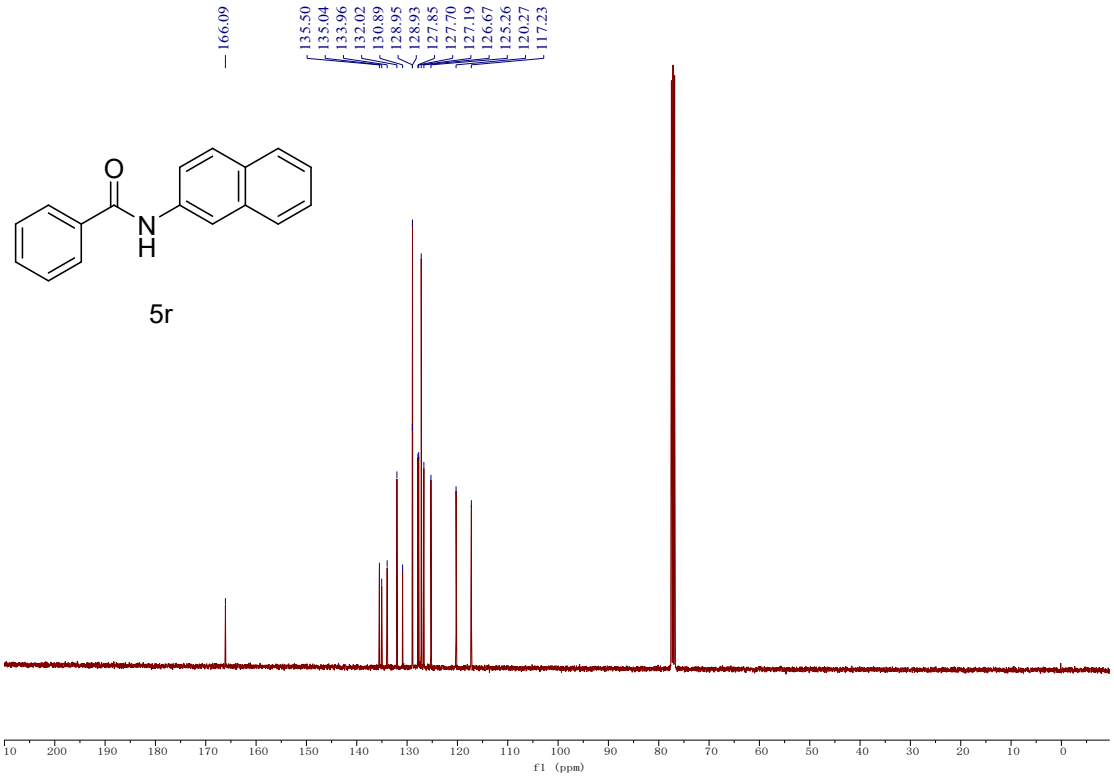

O
N
H
N

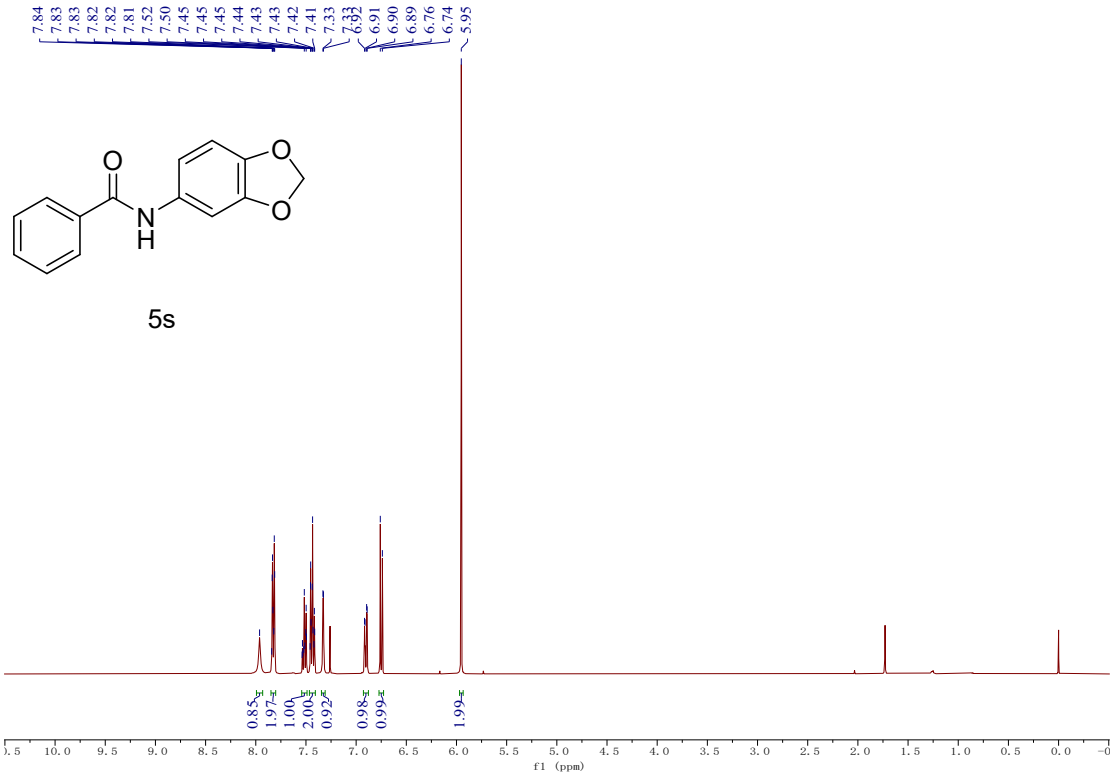
5q

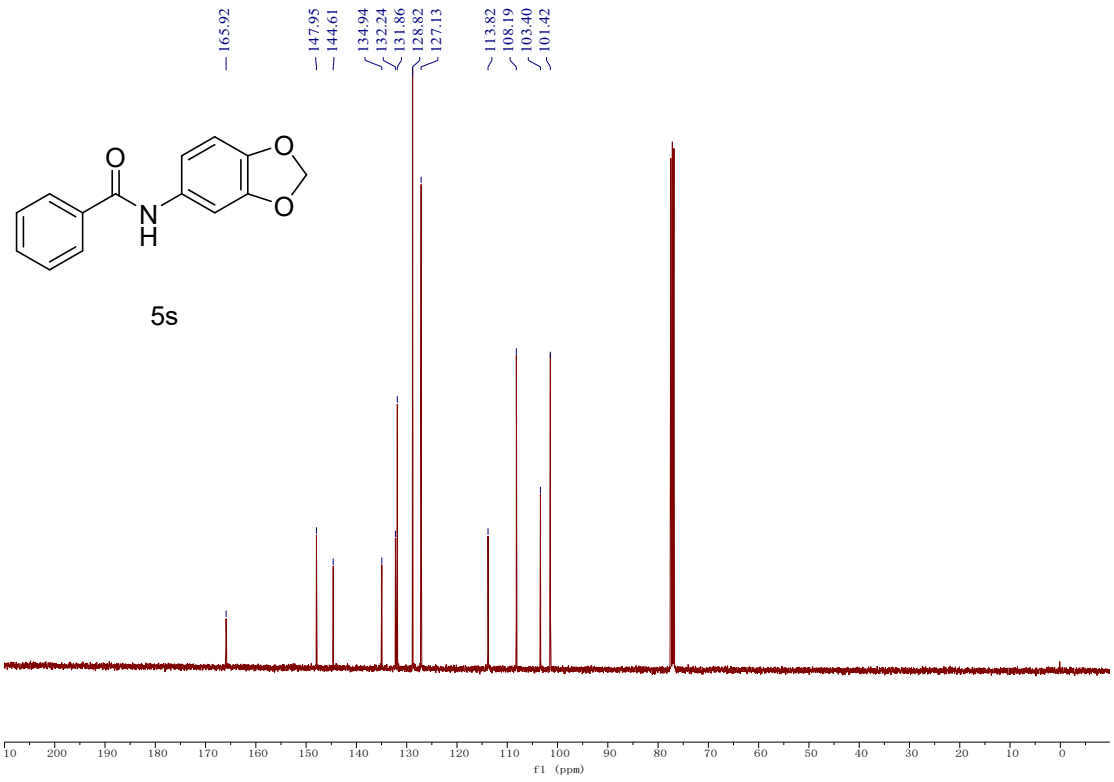
0.87
0.96
0.97
1.98
1.01
1.00
2.00
0.99
f1 (ppm)

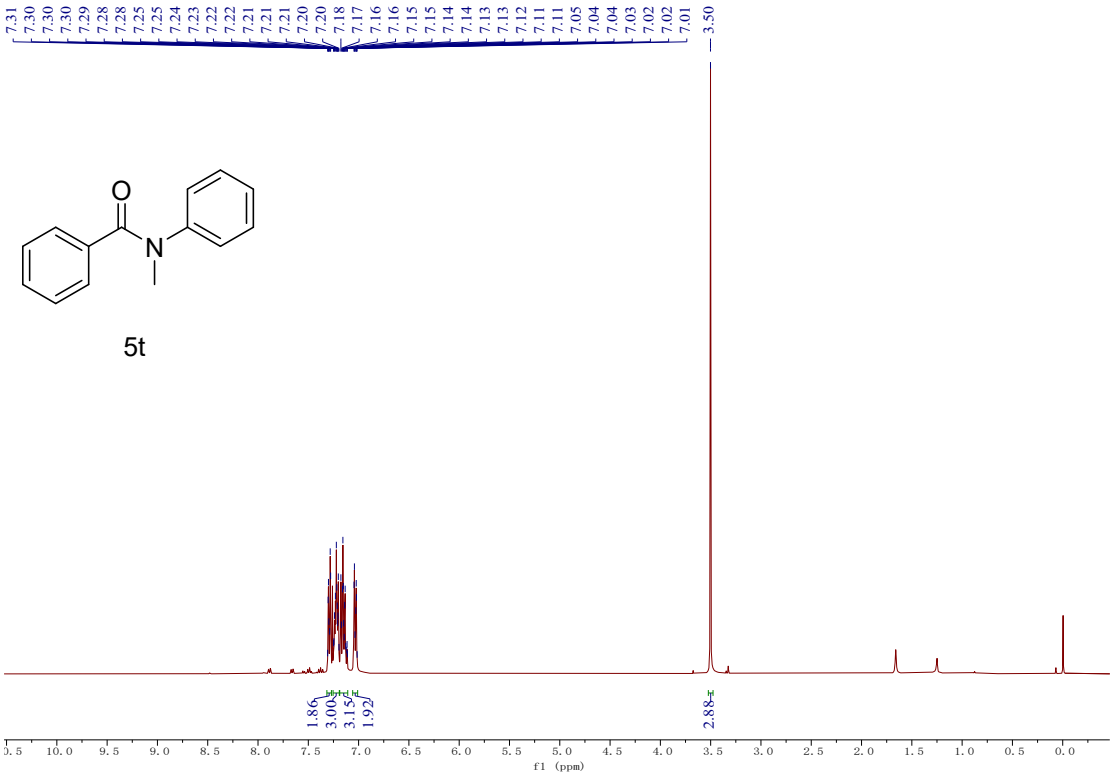

166.13
151.82
147.90
138.60
134.46
132.28
128.87
128.55
127.42
119.96
114.44
O
N
H
N
5q
f1 (ppm)

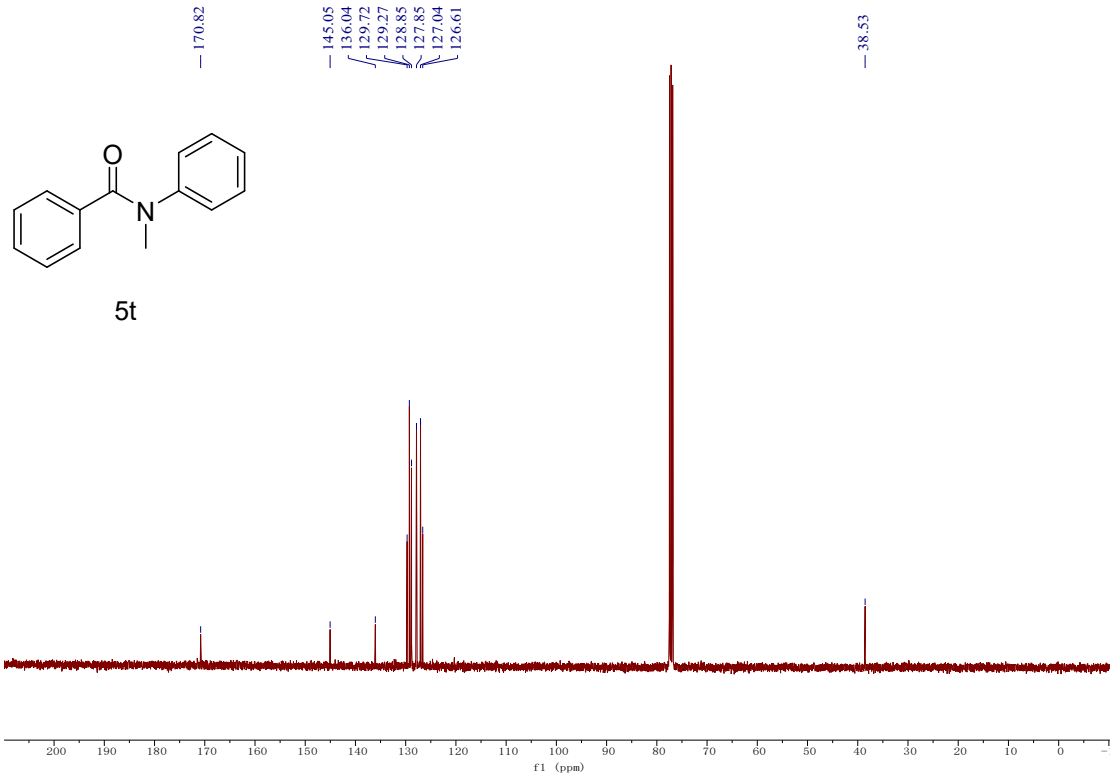

O
N
H
5r
0.97
0.91
1.96
3.01
1.01
1.00
4.01
f1 (ppm)


166.09
135.50
135.04
133.96
132.02
130.89
128.95
128.93
127.85
127.70
127.19
126.67
125.26
120.27
117.23
O
N
H
5r
f1 (ppm)

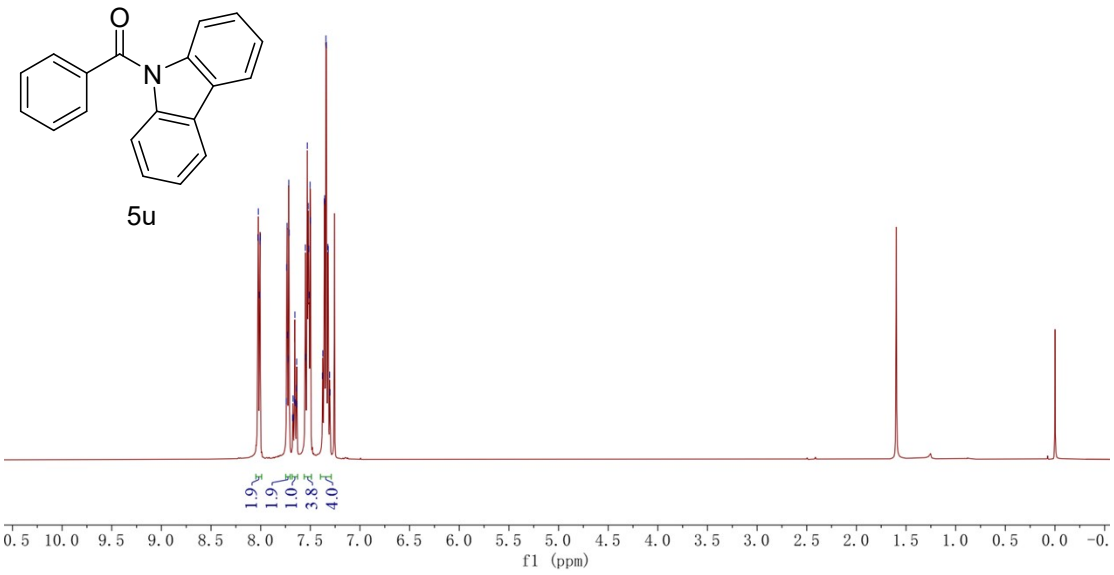

7.84
7.83
7.83
7.82
7.82
7.81
7.52
7.50
7.45
7.45
7.45
7.44
7.43
7.43
7.42
7.41
7.33
7.33
6.92
6.91
6.90
6.89
6.76
6.74
5.95
O
O
O
N
H
5s
0.85
1.97
1.00
2.00
0.92
0.98
0.99
1.99
f1 (ppm)

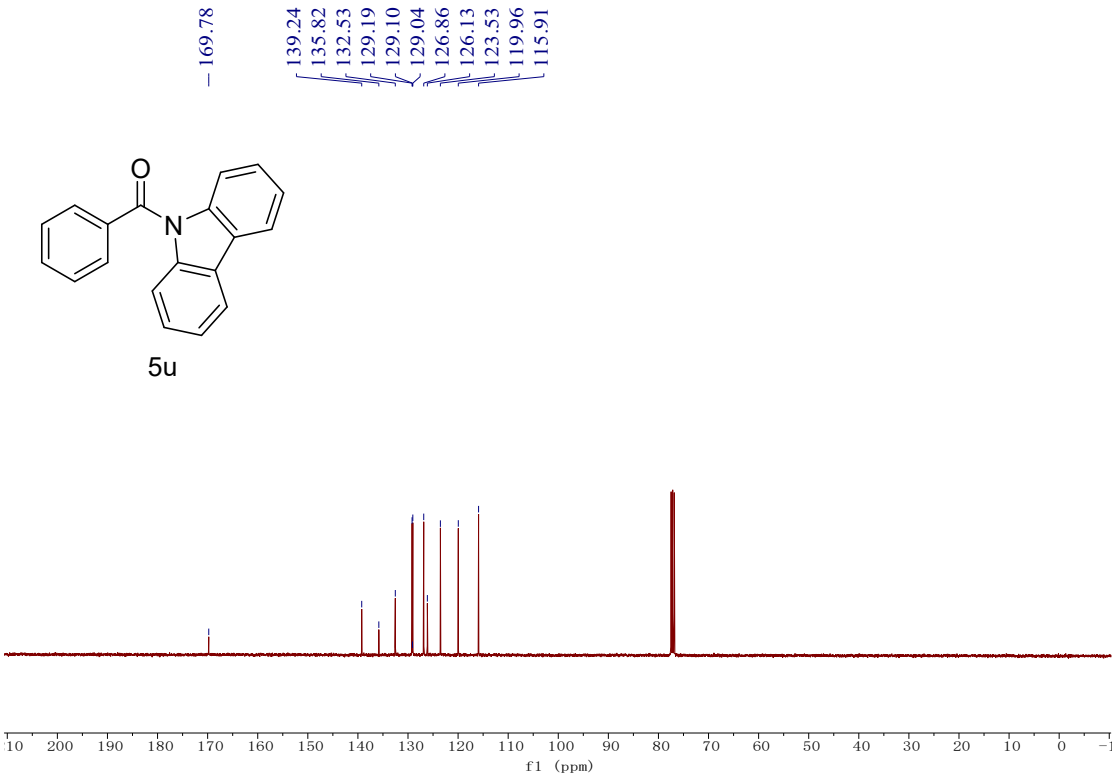

165.92
147.95
144.61
134.94
132.24
131.86
128.82
127.13
113.82
108.19
103.40
101.42
O
N
H
O
O
5s
f1 (ppm)


7.31
7.30
7.30
7.30
7.29
7.28
7.28
7.25
7.25
7.24
7.23
7.22
7.22
7.21
7.21
7.21
7.20
7.20
7.18
7.17
7.16
7.16
7.15
7.15
7.14
7.14
7.13
7.13
7.12
7.11
7.11
7.05
7.04
7.04
7.03
7.02
7.02
7.01
3.50
O
N
5t
1.86
3.00
3.15
1.92
2.88
f1 (ppm)


170.82
145.05
136.04
129.72
129.27
128.85
127.85
127.04
126.61
38.53
O
N
5t
f1 (ppm)


8.03
8.02
8.01
8.01
8.01
7.74
7.74
7.73
7.73
7.72
7.72
7.71
7.68
7.67
7.67
7.66
7.66
7.65
7.64
7.64
7.63
7.55
7.55
7.53
7.52
7.52
7.51
7.50
7.50
7.38
7.37
7.36
7.36
7.34
7.34
7.32
7.32
7.31
7.30


O
N
5u
1.9
1.9
1.0
3.8
4.0
f1 (ppm)


169.78
139.24
135.82
132.53
129.19
129.10
129.04
126.86
126.13
123.53
119.96
115.91
O
N
5u
f1 (ppm)