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Oxidative Coupling of Methylamine with Aminyl Radical: Direct Amidation Catalyzed by I_2/TBHP with HCl

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Supporting Information

Contents:

General Remarks: All reactions were carried out in 10mL flask equipped with a condenser unless otherwise indicated. Reactions were run using Teflon^{TM} -coated

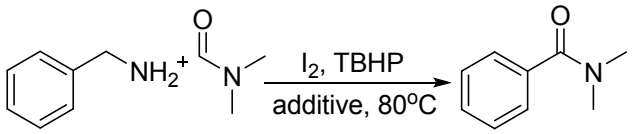
magnetic stir bars. ^{1}H-NMR and ^{13}C-NMR were recorded on a Bruker AC-300 FT (^{1}H: 400 MHz, ^{13}C: 100 MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz respectively. Infrared samples were recorded on a Perkin-Elmer 2000 FTIR spectrometer. HRMS were recorded on the TOF-HRMS–EI at the Instruments' Center for Physical Science, University of Science & Technology of China. All commercially available reagents were used as received.

General Experimental Section

General procedure: TBHP (360 mg, 4.0 mmol, 70 wt.% cyclohexane solution) was added to a mixture of aryl-methylamine (107 mg, 109 μL, 1.0 mmol), I_2 (63.5 mg, 0.25 mmol), and DMF (1.0 mL) in a round-bottom flask equipped with a condenser. And then, 0.1 mL HCl was added into the mixture below the liquid surface. The mixture was heated at 70 ^{o}C for 2h, and allowed to stir at 80 ^{o}C for 18h. After the reaction was complete, the mixture was cooled to room temperature. Then, 3 mL saturated solution of $Na_2S_2O_4$ was added. The mixture was extracted with ethylacetate (3×15 mL). The combined organic phases were dried with Na_2SO_4 and evaporated under vacuum. Purification of the residue by flash column chromatography (petroleum ether/ EtOAc = 2:1-1:1) afforded the desired amide.

Optimization of the reaction condition

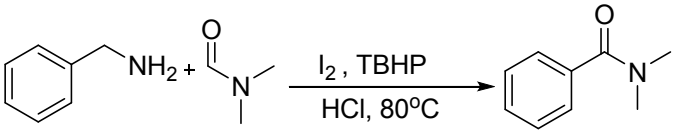
Table S1. Optimization of the reaction conditions [a]



Entry	Catalyst	Oxidant	additive [c]	Yield(%) [a, b]
1	I_2	TBHP	NaOH	<10
2	I_2	TBHP	KOH	<10
3	I_2	TBHP	$LiOH·H_2O$	<10
4	I_2	TBHP	pyridine	complex
5	I_2	TBHP	Et_3N	trace
6	I_2	TBHP	K_2CO_3	trace
7	I_2	TBHP	—	38
8	I_2	TBHP	HCl [c]	54
9	I_2	TBHP [d]	HCl	46
10	I_2	—	HCl	n. d.
11	—	TBHP	HCl	n. d.
12	I_2	TBHP	HCl [e]	82
13	I_2	TBHP	H_2SO_4	23
14	I_2	TBHP	HNO_3	18
15	I_2	$K_2S_2O_8$	HCl	<5
16	I_2	H_2O_2	HCl	trace
17	I_2	DTBP [g]	HCl	trace
18	I_2	m-CPBA [h]	HCl	trace
19	I_2	DDQ [i]	HCl	trace

Reaction conditions: (a) The reactions were carried out with benzylamine (107.1mg, 1 mmol), DMF (1mL), I_2 (63.5mg, 0.25 mmol), TBHP (360mg, 4eq, 70% cyclohexane solution), at 80 °C for 18 h. (b) Yield of isolated product is based on the benzylamine. (c) Base (NaOH, KOH, LiOH, pyridine, Et_3N, K_2CO_3 respectively 0.2 mmol). 0.5mL of concentrated acid (HCl, HNO_3, H_2SO_4). (d) Using aqueous TBHP (70%). (e) 0.1mL concentrated HCl. (g) DTBP = di-tert-butyl peroxide. (h) m-CPBA = *meta*-Chloroperoxybenzoic acid. (i) DDQ = 2,3-dichloro-5,6-dicyano-1,4-benzoquinone.

Table S2. Optimization of the ratio of I_2 /TBHP /HCl



Entry	I_2 (mol%)	TBHP/eq	HCl/mL	yield/%
1	25%	1	0.1	45
2	25%	2	0.1	63
3	25%	4	0.1	80
4	25%	6	0.1	68
5	10%	4	0.1	45
6	50%	4	0.1	53
7	100%	4	0.1	65
8	25%	4	0.01	35
9	25%	4	0.02	48
10	25%	4	0.06	65
11	25%	4	0.1	82
12	25%	4	0.12	70
13	25%	4	0.15	68
14	25%	4	0.2	72

(a) The reactions were carried out with benzylamine (107 mg, 109 μL, 1 mmol), DMF (1mL), I_2, TBHP (70 % cyclohexane solution), at 80 °C for 18 h. (b) Yield of isolated product is based on the benzylamine.

When I_2 (25 mol%) was employed, the reaction gave the highest yield (Table S2, entries 3, 5, 6, 7). If I_2 (100 mol%) was added into the reaction mixture, the reaction became complex and gave low yield amide (Table S2, entry 7). The optimal quantity of TBHP was 4 equivalents of substrate (Table S2, entries 2, 3, 4). Because the great role of HCl in the amidation of benzylamine, detailed ratio of HCl was also researched as shown in Table S1. When 0.01mL HCl was added, the major product is cyanobenzene, only 35% **3aa** was obtained. Increasing the ratio of HCl leads to high yield. When 0.1 mL HCl was added, the reaction gave the highest yield (Table S2,

entries 7 - 13). Higher ratio HCl decreased the yield due to the partial decomposition of amide bond.

Control experiments and the analysis

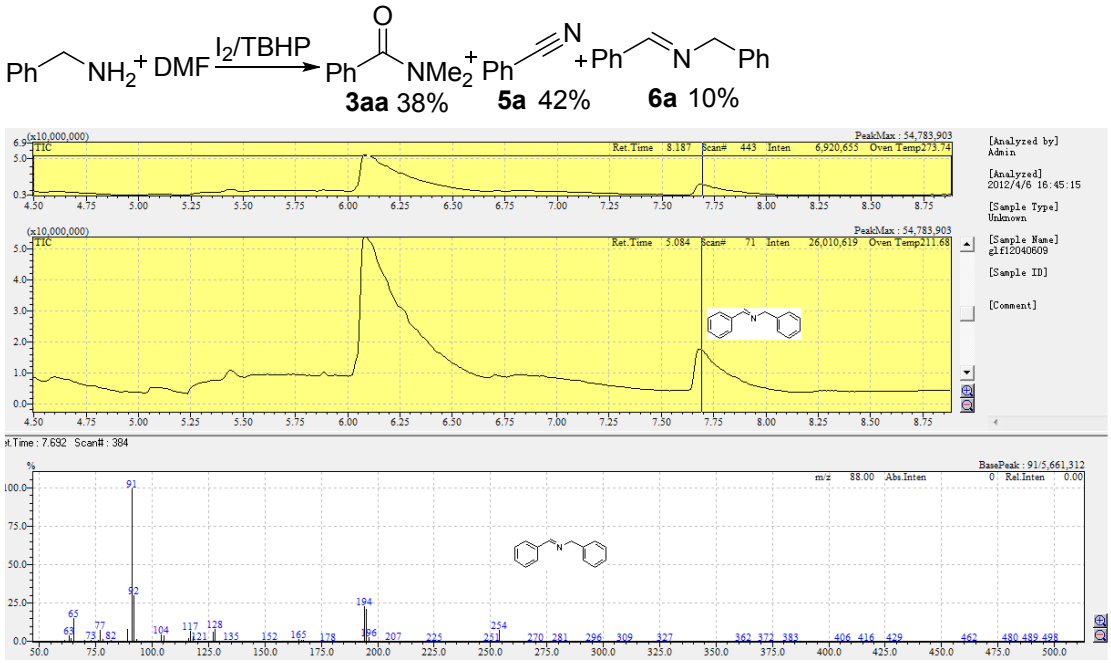
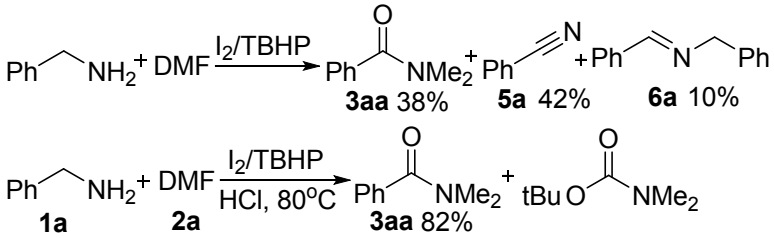


Figure S1. The GC-Ms of the reaction mixture under standard condition, which show that the N-benzyl imine **6a** was detected at the 7.70 min.



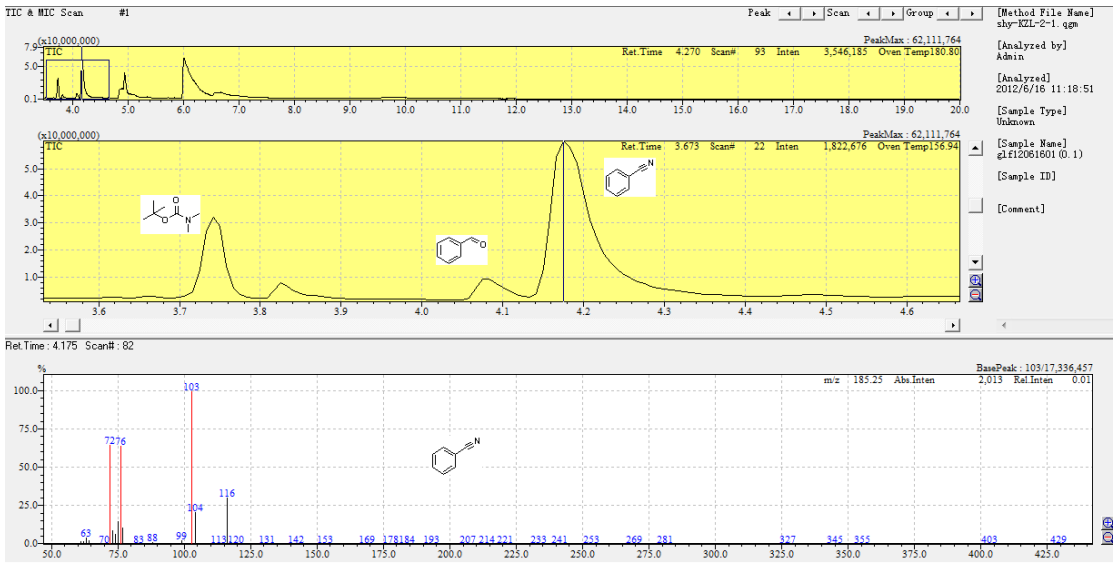
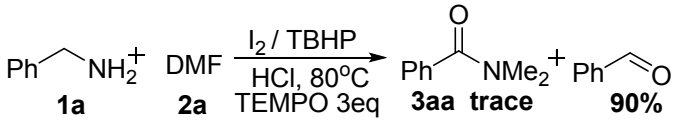


Figure S2. The GC-Ms of the reaction mixture in the presence of base or in the absence of HCl, the imine **4a** major transforms to cyanobenzene **5a** was detected at the 4.1min. And then, a trace amount of benzaldehyde was also detected at 3.9 min.



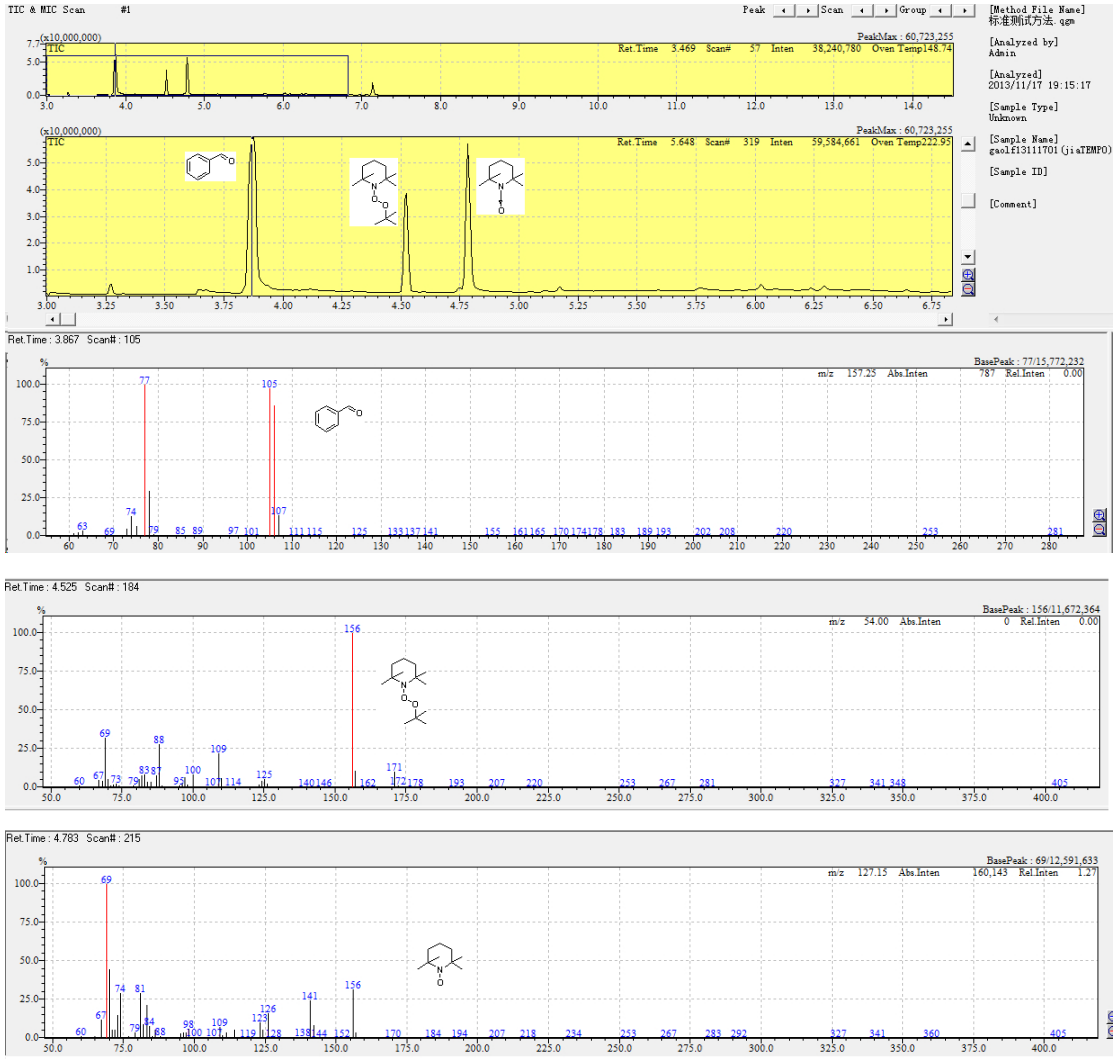


Figure S3. The GC-Ms of the reaction mixture when 3 eq 2,2,6,6-Tetramethylpiperidine-1-oxyl (TEMPO) was added into the reaction, benzaldehyde was obtained in 90% yield, no any amide **3aa** was detected. And 1-(tert-butylperoxy)-2,2,6,6-tetramethylpiperidine of the cross coupling of TBHP with TEMPO was detected at the 4,49 min. There was no any amide **3aa** was detected.

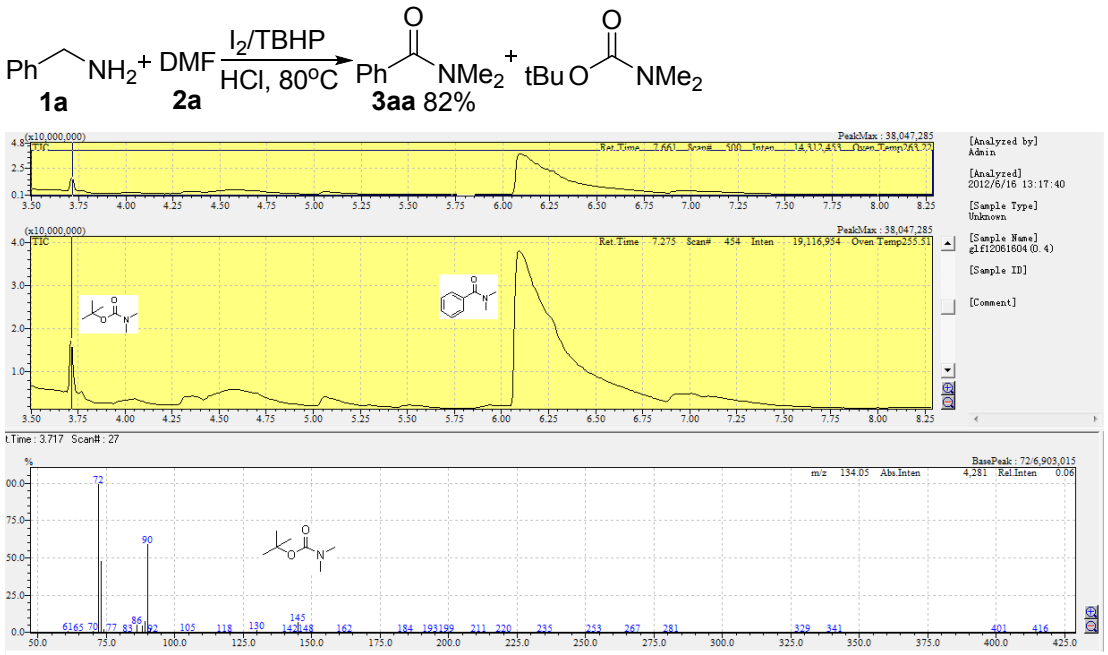
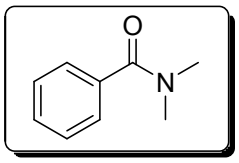


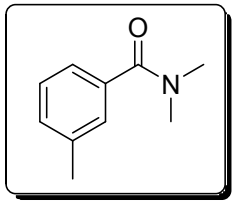
Figure S4. The GC-Ms of the reaction mixture under standard condition, t-Butyl N,N-dimethylcarbamate was detected via GC-Ms at the 3.75min.

Characterization data of all products:



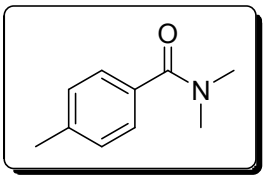
N,N-dimethylbenzamide **3aa**

Compound **3aa** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.10 (*d,* 6H), 7.40 (*s*, 5H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 35.2, 39.5, 126.9, 128.2, 129.4, 136.3, 171.5. IR (liquid film, cm^{-1}): *v* = 3057, 2998, 2934, 2854, 1631, 1431, 1271, 708. HRMS: calcd for $C_9H_{11}NO$: 149.0841, found 149.0847.



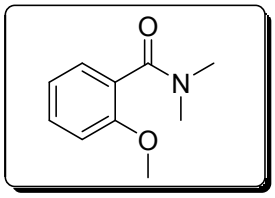
N,N,3-trimethylbenzamide **3ba**

Compound **3ba** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc = 2:1-1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 2.36 (s, 3H), 3.10 (*d,* 6H), 7.21(m, 3H), 7.28 (m, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) = 21.5, 35.5, 39.7, 124.1, 127.8, 128.3, 130.3, 136.5, 138.3, 172.0. HRMS: calcd for $C_{10}H_{13}NO$: 163.0997, found 163.1002.



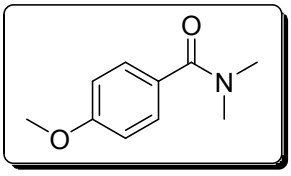
N,N,4-trimethylbenzamide **3ca**

Compound **3ca** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 2.37 (s, 3H), 3.04 (s, 6H), 7.19 (d, *J* =8 Hz, 2H), 7.31(d, *J* =8 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 21.5, 36.1, 38.8, 127.3, 129.0, 133.4, 139.7, 171.9. HRMS: calcd for $C_{10}H_{13}NO$: 163.0997, found 163.1001.



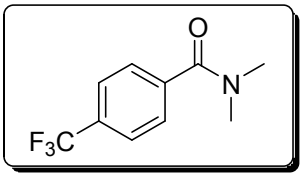
2-methoxy-N,N-dimethylbenzamide **3da**

Compound **3da** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 2.85 (*s,* 3H,), 3.12 (*s,* 3H,), 3.83 (s, 3H,), 6.91 (d, *J* = 8 Hz, 1H). 6.98 (t, *J* = 8 Hz, 1 H), 7.23 (q, *J* = 8 Hz, 1H), 7.34 (m, *J* = 8 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 35.0, 38.5, 55.7, 111.0, 120.9, 126.0, 128.0, 130.5, 155.4, 169.6. IR (liquid film, cm^{-1}): *v* = 3057, 2998, 2934, 2854, 1631, 1431, 1271, 708. HRMS: calcd for $C_{10}H_{13}NO_2$: 179.0946, found 179.0951.



4-methoxy-N,N-dimethylbenzamide **3ea**

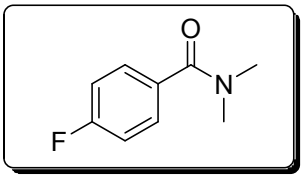
Compound **3ea** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 3.07 (s, 6H), 3.83 (s, 3H), 6.91 (m, 2H), 7.41 (d, *J*= 8Hz, 2H), 7.58 (d, *J* = 4 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 37.2, 38.0, 55.5, 113.7, 128.04, 128.08, 129.4, 160.8, 171.8. HRMS: calcd for $C_{10}H_{13}NO_2$: 179.0946, found 179.0951.



N,N-dimethyl-4-(trifluoromethyl)benzamide **3fa**

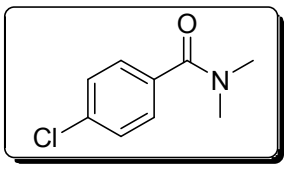
Compound **3fa** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 2.96 (s, 3H), 3.13 (s, 3H), 7.53 (d, *J* = 8Hz, 2H), 7.67 (d, *J* = 8Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 35.4, 39.5, 60.5, 122.5, 125.2, 125.51, 125.55, 125.58, 125.62,

127.50, 131.08, 131.40, 131.7, 132.1, 140.0, 170.2. IR (liquid film, cm^{-1}): ν = 3059, 2960, 2935, 2869, 1628, 1405, 1131, 863. HRMS: calcd for $C_{10}H_{10}F_3NO$: 217.0714, found 217.0719.



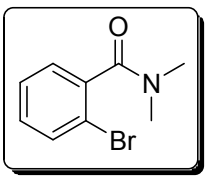
4-fluoro-N,N-dimethylbenzamide **3ga**

Compound **3ga** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.05 (s, 6H), 7.09 (m, 2H), 7.43 (m, 2H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) = 38.1, 115.35, 115.37, 115.59, 129.4, 129.48, 132.21, 132.24, 162.13, 163.41, 164.41, 164.61, 165.90, 167.35, 170.81. IR (liquid film, cm^{-1}): ν = 3288, 3035, 2959, 2925, 2855, 1642, 1449, 1093. HRMS: calcd for $C_9H_{10}FNO$: 167.0746, found 167.0752.



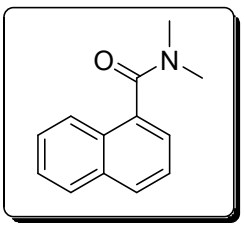
4-chloro-N,N-dimethylbenzamide **3ha**

Compound **3ha** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.04 (s, 6H), 7.37 (s, 4H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 35.4, 39.5, 128.59, 128.62, 134.7, 135.5, 170.5. HRMS: calcd for $C_9H_{10}ClNO$: 183.0451, found 183.0454.



2-bromo-N,N-dimethylbenzamide **3ia**

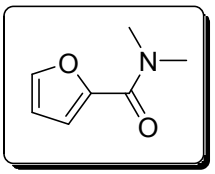
Compound **3ia** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 2.86 (s, 3H), 3.14 (s, 3H), 7.24 (m, 2H), 7.36 (m, 1H), 7.58 (d, J = 8 Hz, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) = 34.8, 38.3, 119.2, 127.8, 130.3, 132.8, 138.6, 169.4. IR (liquid film, cm^{-1}): ν = 3380, 2958, 2925, 2854, 1698, 1606, 1449, 1260, 1028, 799. HRMS: calcd for $C_9H_{10}BrNO$: 226.9946, found: 226.9944.



N,N-dimethyl-1-naphthamide **3ja**

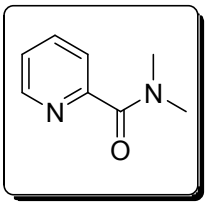
Compound **3ja** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 2.81 (s, 3H), 3.26

(s, 3H), 7.41 (d, *J* =8 Hz 1H), 7.50 (m, 3H), 7.78 (d, *J* =8 Hz 1H), 7.87 (d, *J* =8 Hz 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 35.0, 39.0, 124.0, 125.0, 125.3, 126.5, 127.1, 128.5, 129.1, 133.6, 134.9, 171.0. HRMS: calcd for $C_{13}H_{13}NO$: 199.0997, found 199.1001.



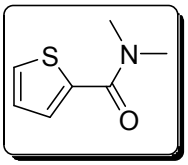
N,N-dimethylfuran-2-carboxamide **3ka**

Compound **3ka** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc = 2:1 - 1:1) to give a white solid. The title compound was white solid. 1H-NMR (300 MHz, $CDCl_3$): δ = 3.19 (s, 6H), 6.47 (q, *J* = 6Hz, 1H), 6.98 (q, *J* = 6 Hz, 1H), 7.49 (q, *J* = 6 Hz, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) = 36.6, 38.3, 111.2, 116.1, 143.8, 148.3, 160.4. HRMS: calcd for $C_7H_9NO_2$: 139.0633, found 139.0635.



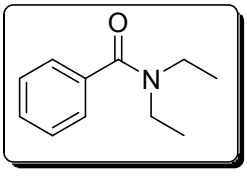
N,N-dimethylpicolinamide **3la**

Compound **3la** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.08(s, 3H), 3.15(s, 3H), 7.37 (t, *J* = 4 Hz, 1H), 7.64 (d, *J* = 8 Hz, 1H), 7.83 (t, *J* = 4 Hz, 1H), 8.60 (d, *J* = 4 Hz, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) = 35.8, 39.1, 123.7, 124.5, 137.4, 148.1, 154.3, 168.8. IR (liquid film, cm^{-1}): *v* = 3284, 3035, 2960, 2854, 2726, 1638, 1450, 1096. HRMS: calcd for $C_8H_{10}N_2O$: 150.0793, found 150.0795.



N,N-dimethylthiophene-2-carboxamide **3ma**

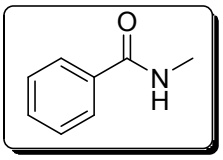
Compound **3ma** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether : EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.18 (s, 6H), 7.04 (q, *J* = 4 Hz, 1H), 7.35 (q, *J* = 4 Hz, 1H), 7.43 (q, *J* = 4 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 37.0, 39.1, 126.7, 128.8, 129.2, 138.0, 164.5. HRMS: calcd for C_7H_9NOS: 155.0405, found 155.0410.



N,N-diethylbenzamide **3an**

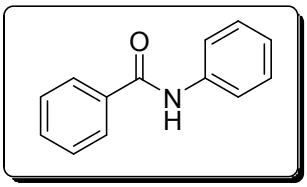
Compound **3an** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 1.18 (m, 6 H), 3.26 (*s,* 6H), 3.54 (*d,*

4H), 7.37 (s, 5H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 12.9, 14.1, 39.4, 43.3, 126.2, 128.3, 129.1, 137.0, 171.4. IR (liquid film, cm^{-1}): *v* = 3057, 2998, 2934, 2854, 1631, 1431, 1271, 706. HRMS: calcd for $C_{11}H_{15}NO$: 177.1154, found 177.1158.



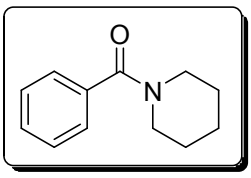
N-methylbenzamide **3ao**

Compound **3ao** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc = 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.02 (d, *J* = 4 Hz, 3H), 6.16 (s, 1H), 7.43 (m, 2H), 7.50 (q, *J* = 4 Hz, 1H), 7.76 (d, *J* = 4 Hz, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) = 27.0, 126.9, 128.7, 131.5, 134.8, 168.4. HRMS: calcd for C_8H_9NO: 135.0684, found 135.0686.



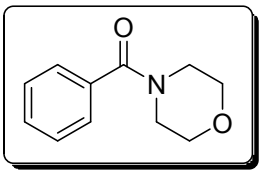
N-phenylbenzamide **3ap**

Compound **3ap** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.09 (m, 2H), 7.15 (m, 1H), 7.32 (m, 3H), 7.53 (d, *J* = 8 Hz, 1H), 7.62 (q, *J* = 8 Hz, 1H), 7.86 (s, 1H), 8.35 (d, *J* = 4 Hz, 1H), 8.69 (d, *J* = 4 Hz, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) = 120.4, 124.7, 127.2, 128.8, 129.2, 131.9, 135.1, 138.1, 166.0. IR (liquid film, cm^{-1}): *v* = 3455, 3344, 3051, 2960, 2926, 2853, 1656, 1439, 1074, 750. HRMS: calcd for $C_{13}H_{11}NO$: 197.0841, found 197.0847.



phenyl(piperidin-1-yl)methanone **3aq**

Compound **3aq** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title compound was white solid. 1H-NMR (300 MHz, CDCl3): δ = 1.59 (s, 3H), 1.67 (d, 6H), 3.53 (s, 4H), 7.39 (s, 5H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) = 24.7, 26.2, 44.5, 48.1, 126.9, 128.5, 129.5, 136.4, 170.4. IR (liquid film, cm^{-1}): *v* = 3254, 3056, 2934, 2855, 1632, 1432, 1277, 1108, 706. HRMS: calcd for $C_{12}H_{15}NO$: 189.1154, found 189.1156.



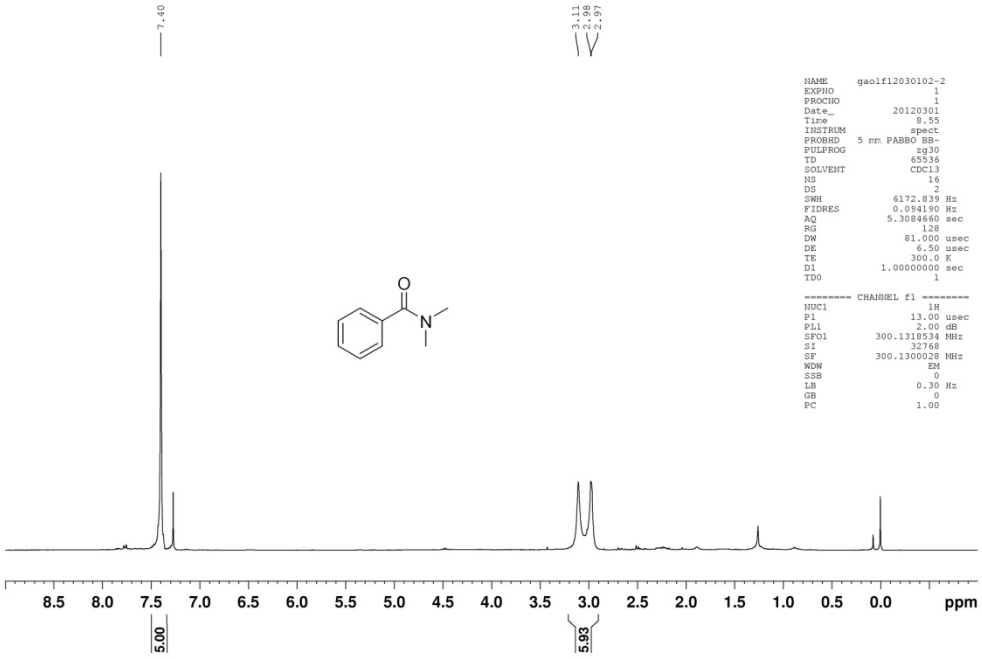
morpholino(phenyl)methanone **3ar**

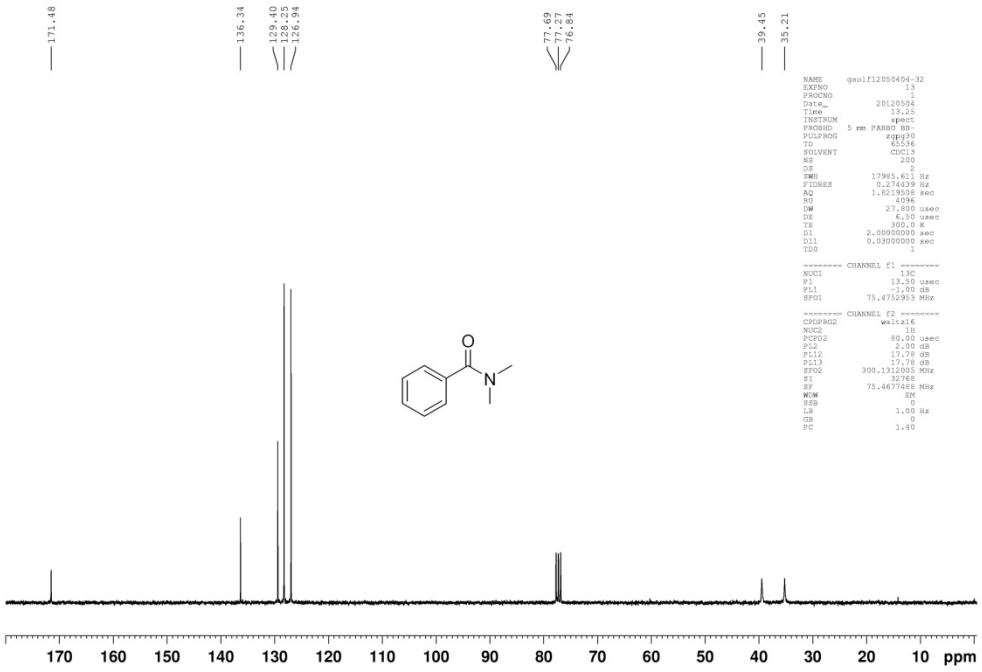
Compound **3ar** was prepared according to the general procedure and purified by column chromatography (Petroleum Ether: EtOAc= 2:1 - 1:1) to give a white solid. The title

compound was white solid. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 3.47, 3.69 (8H), 7.41 (m, 5H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) = 42.7, 48.2, 66.9, 127.1, 128.5, 129.9, 135.3, 170.4. IR (liquid film, cm^{-1}): *v* = 3361, 2959, 2922, 2853, 1634, 1428, 1113. HRMS: calcd for $C_{11}H_{13}NO_2$: 191.0946, found 191.0949.

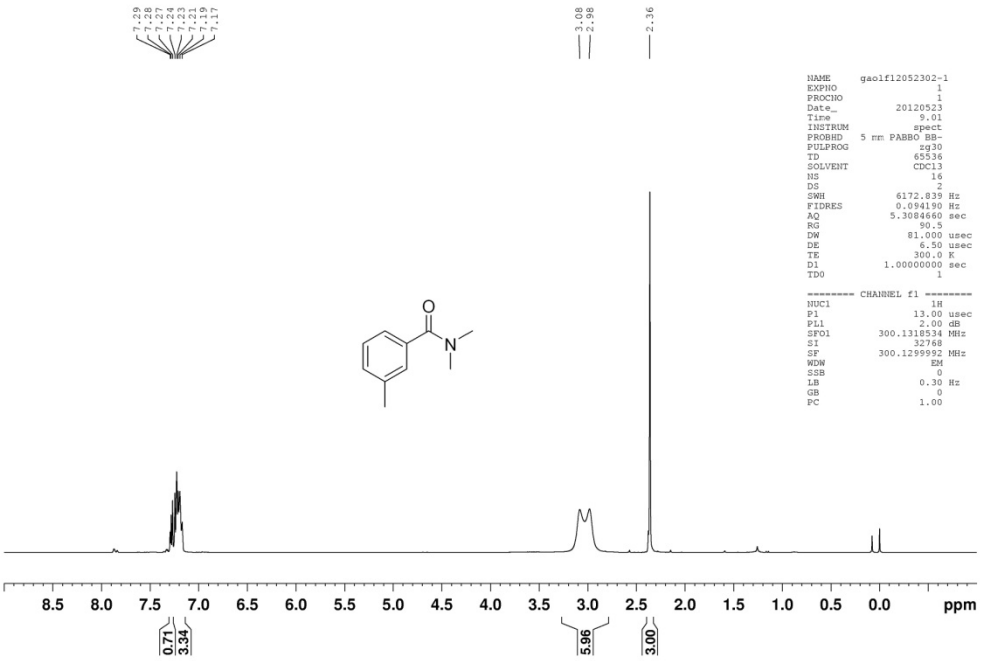
NMR Spectra of all compounds

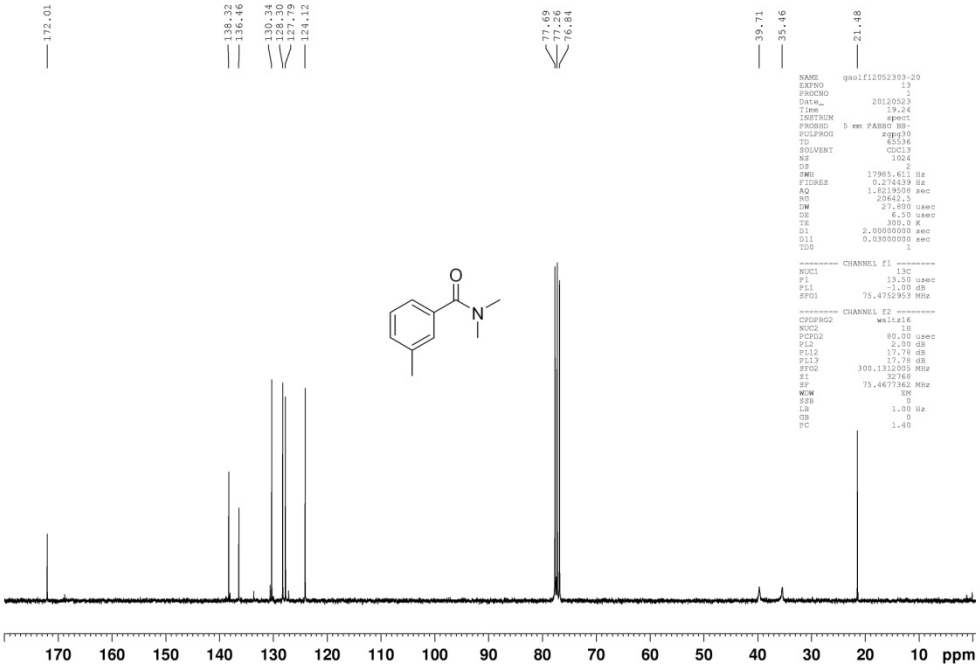
^{1}H NMR, ^{13}C NMR of 3aa:



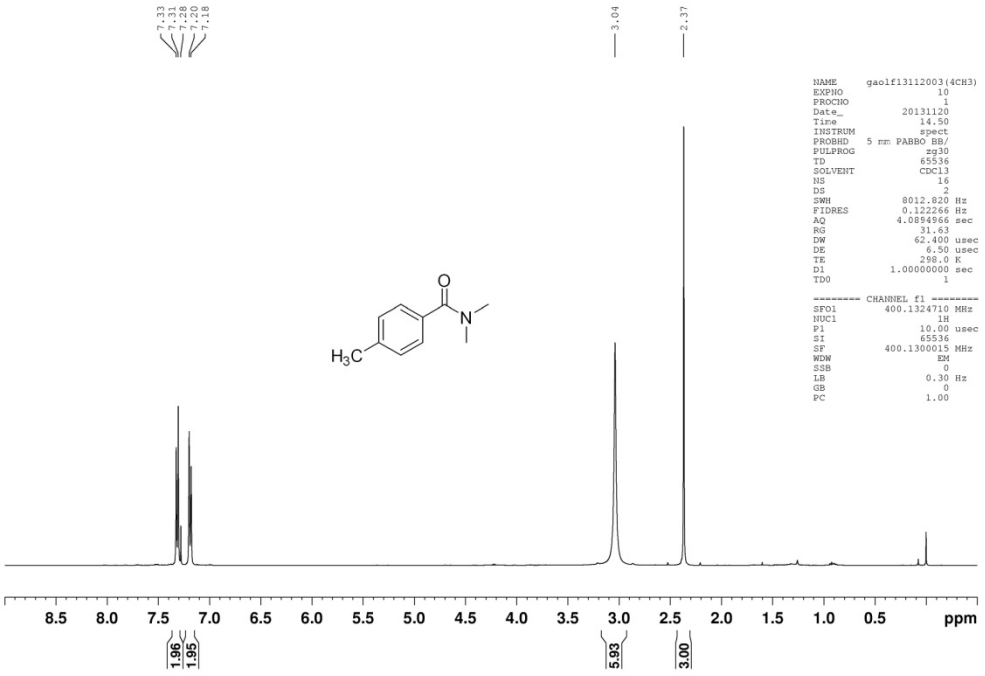


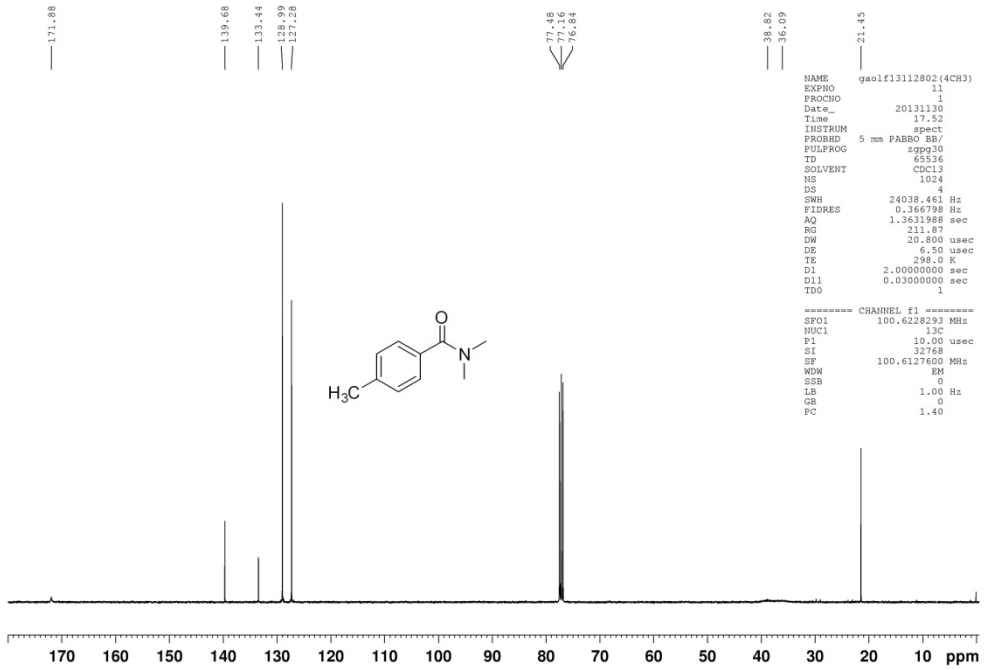
^{1}H NMR, ^{13}C NMR of 3ba:



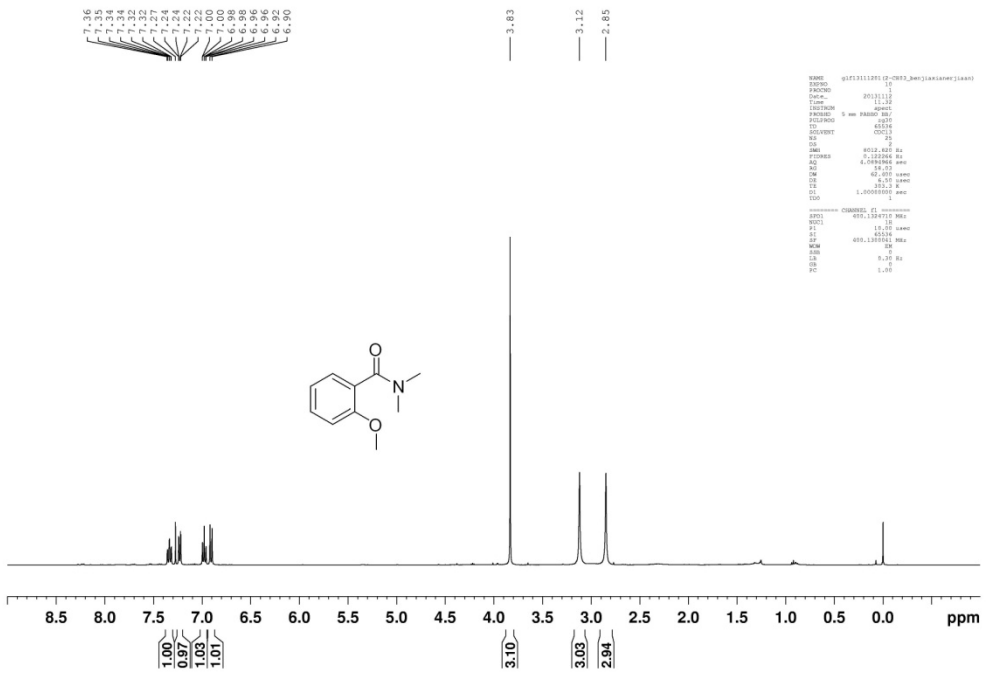


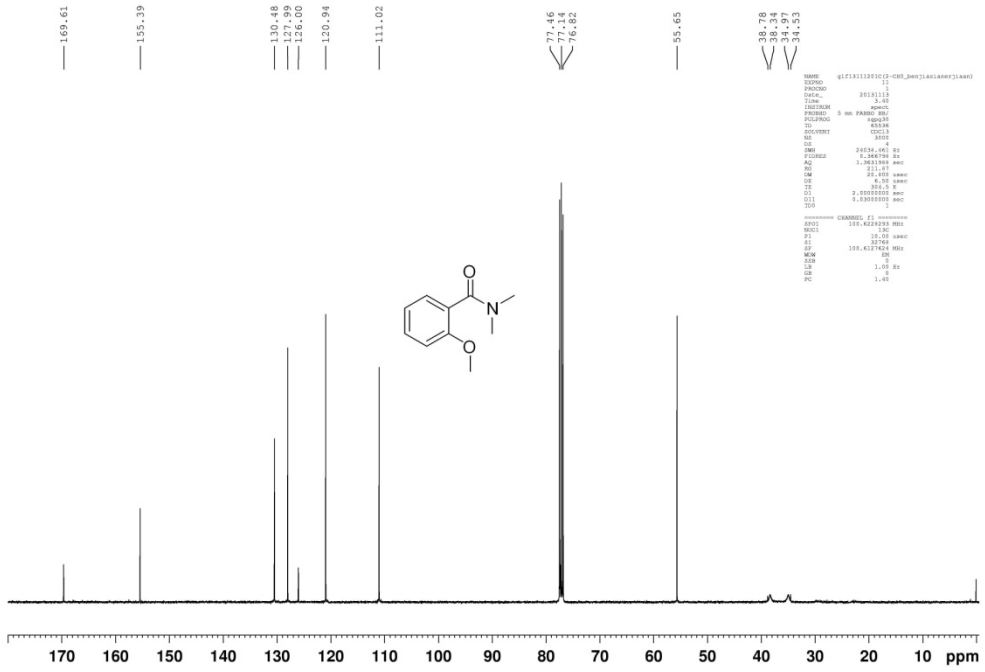
^{1}H NMR, ^{13}C NMR of 3ca:



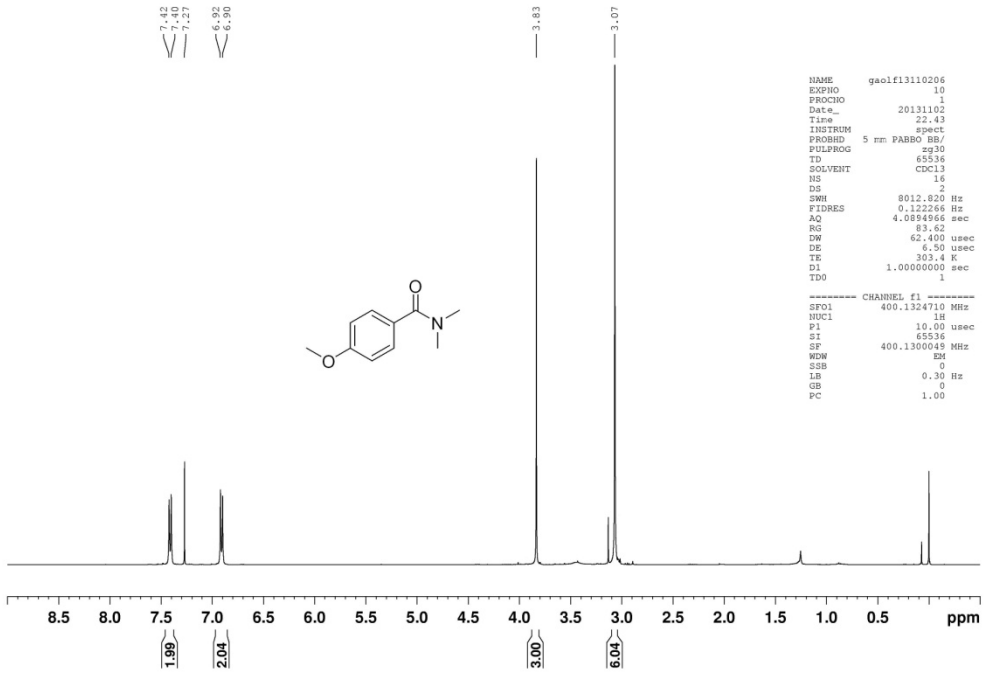


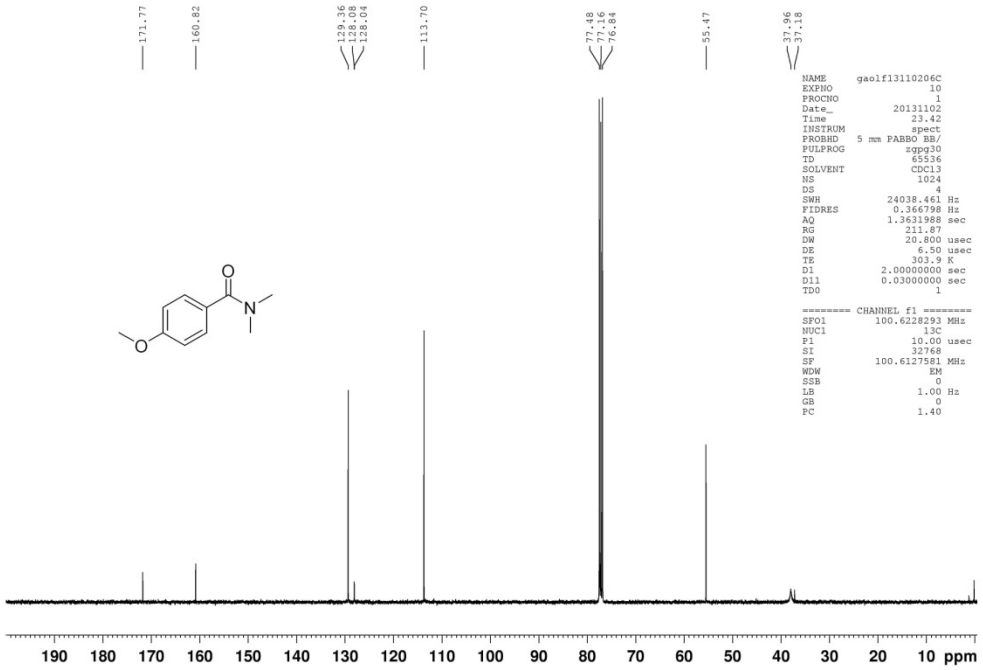
^{1}H NMR, ^{13}C NMR of 3da:



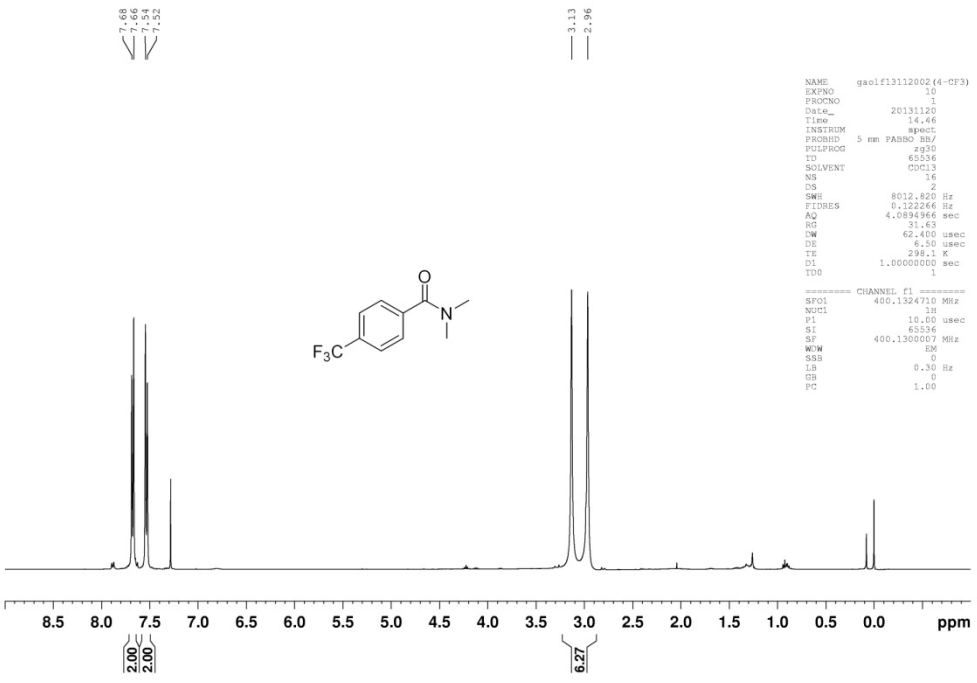


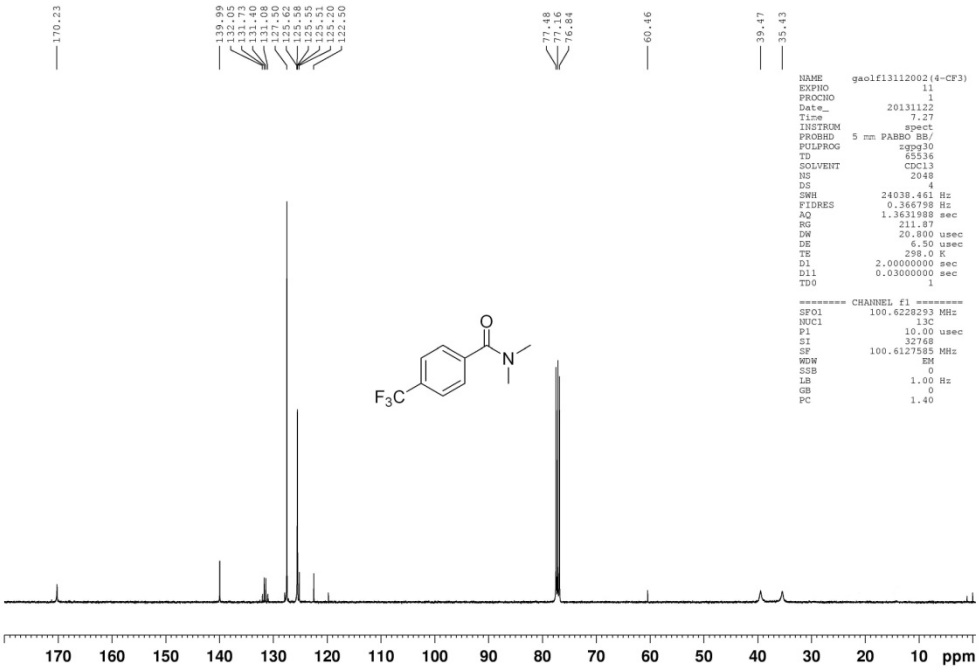
^{1}H NMR, ^{13}C NMR of 3ea:



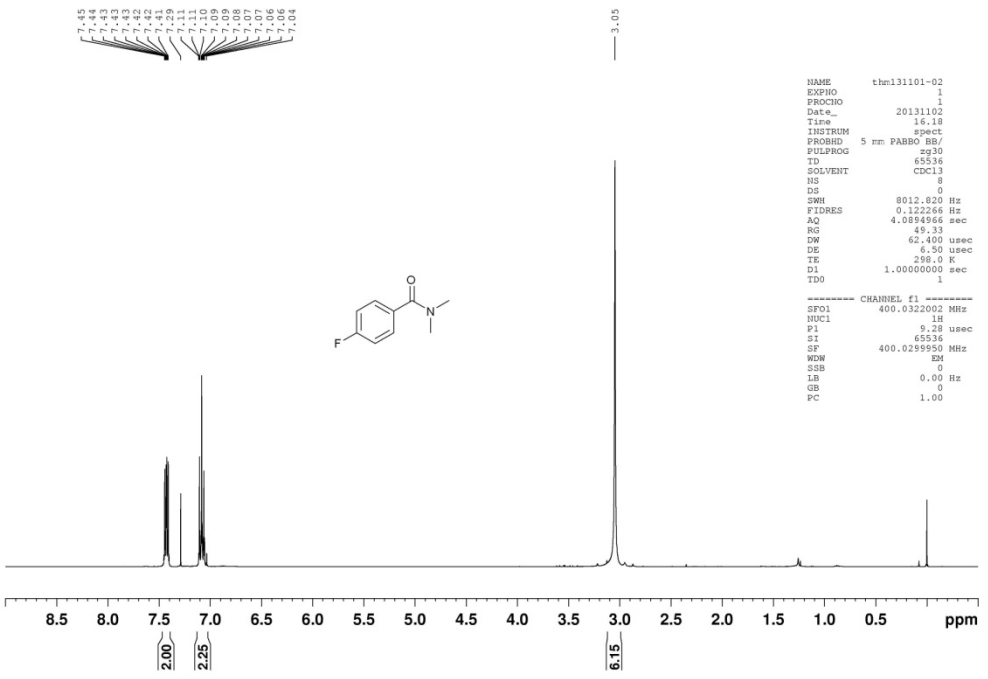


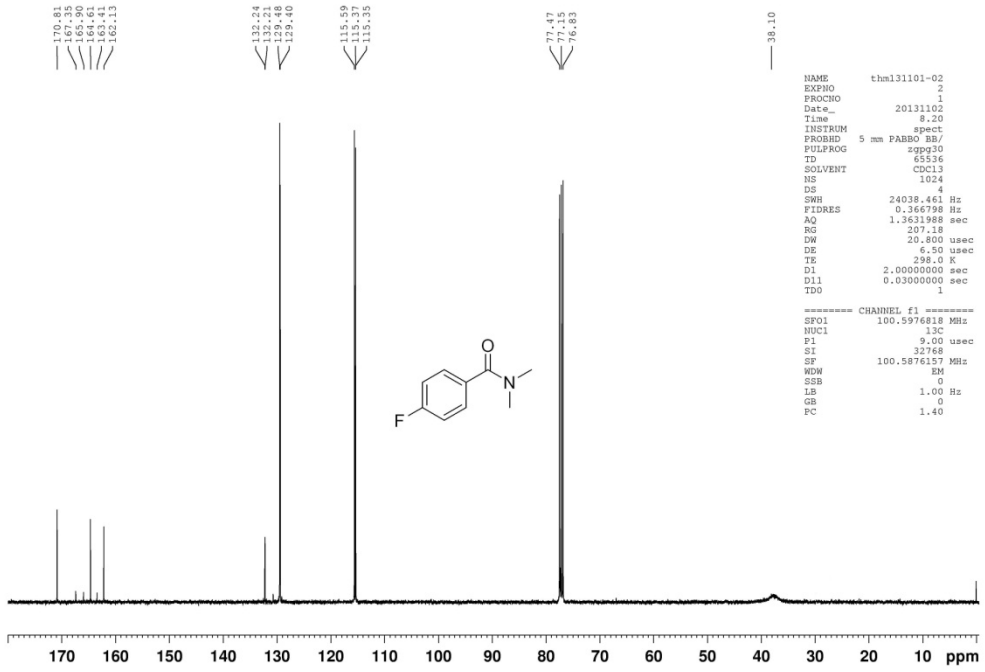
^{1}H NMR, ^{13}C NMR of 3fa:



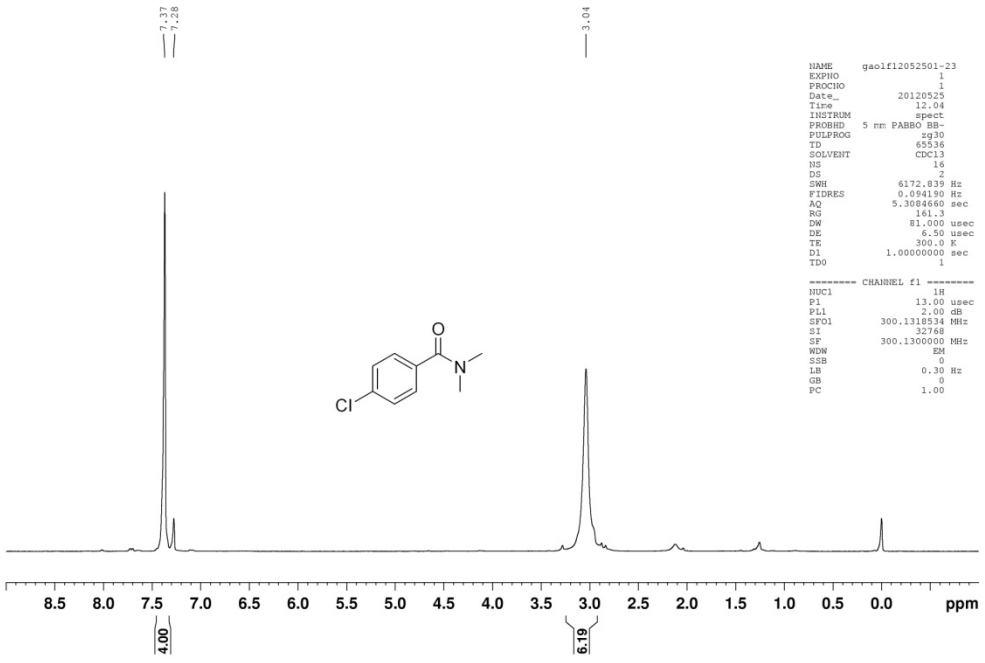


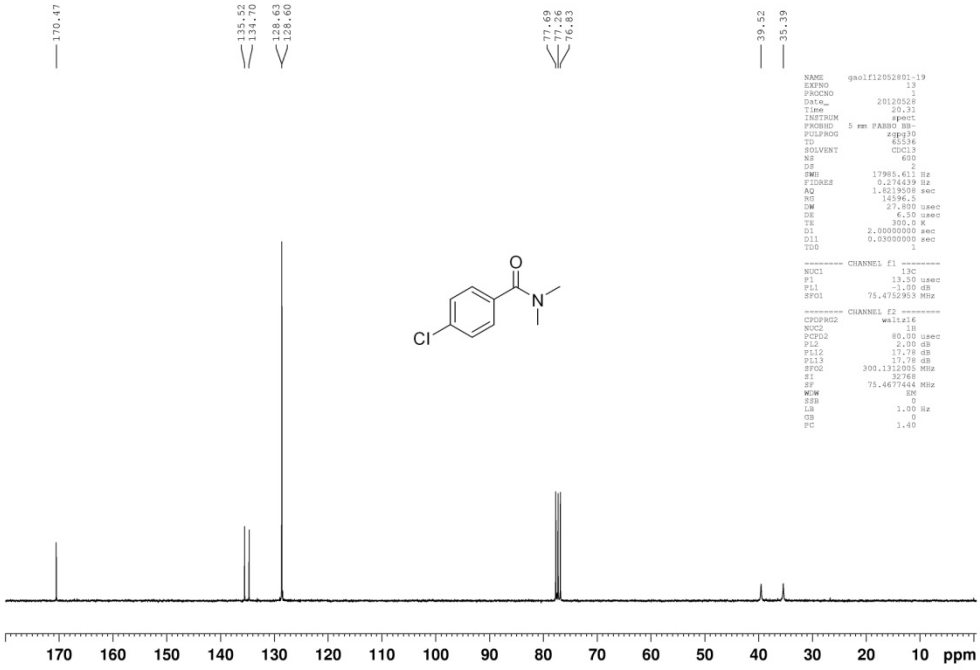
^{1}H NMR, ^{13}C NMR of 3ga:



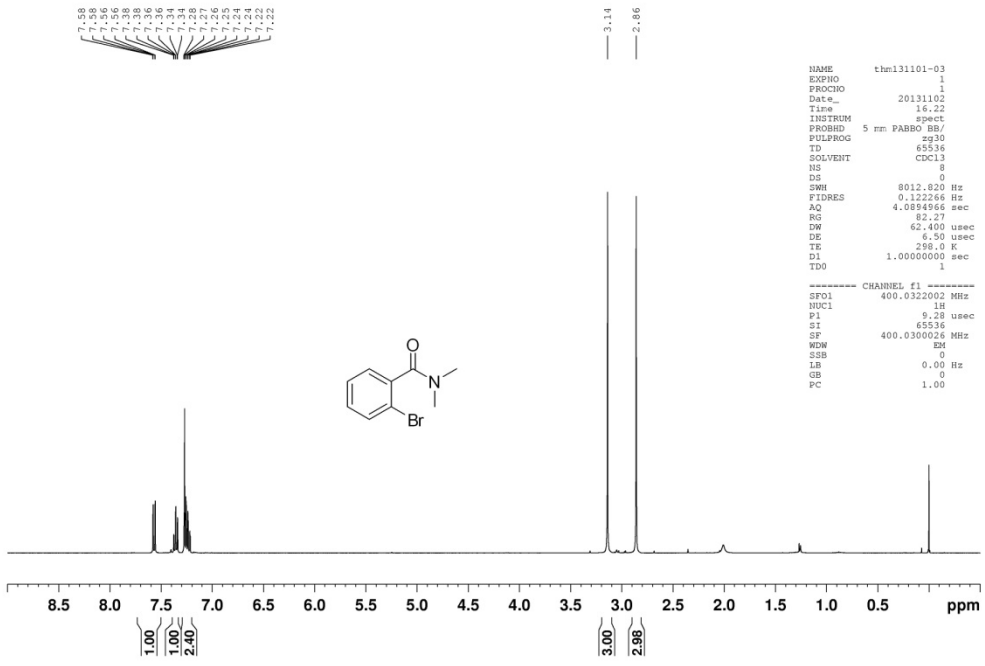


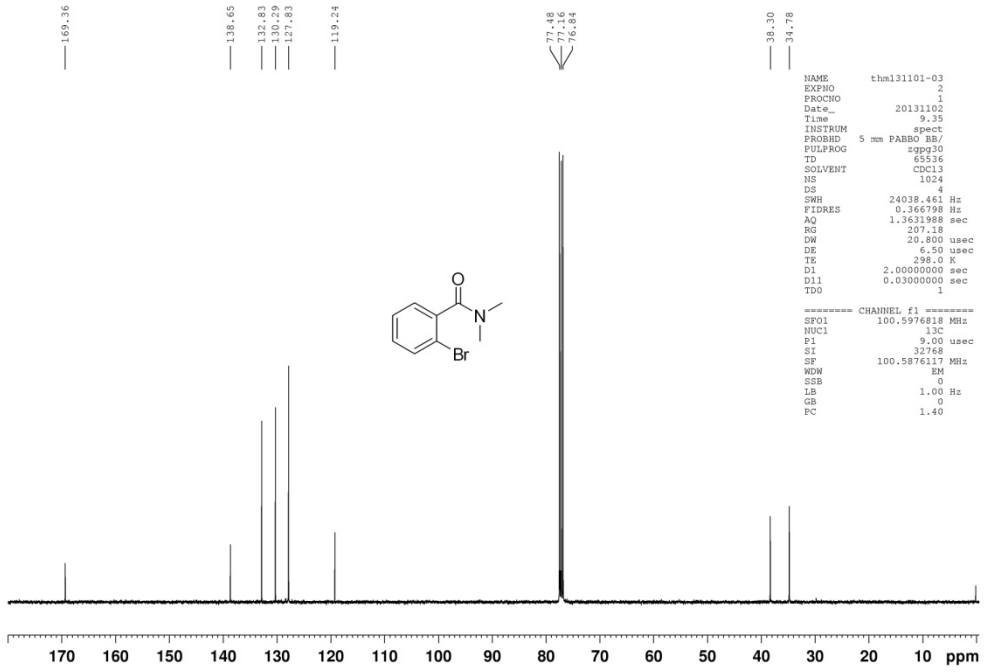
^{1}H NMR, ^{13}C NMR of 3ha:



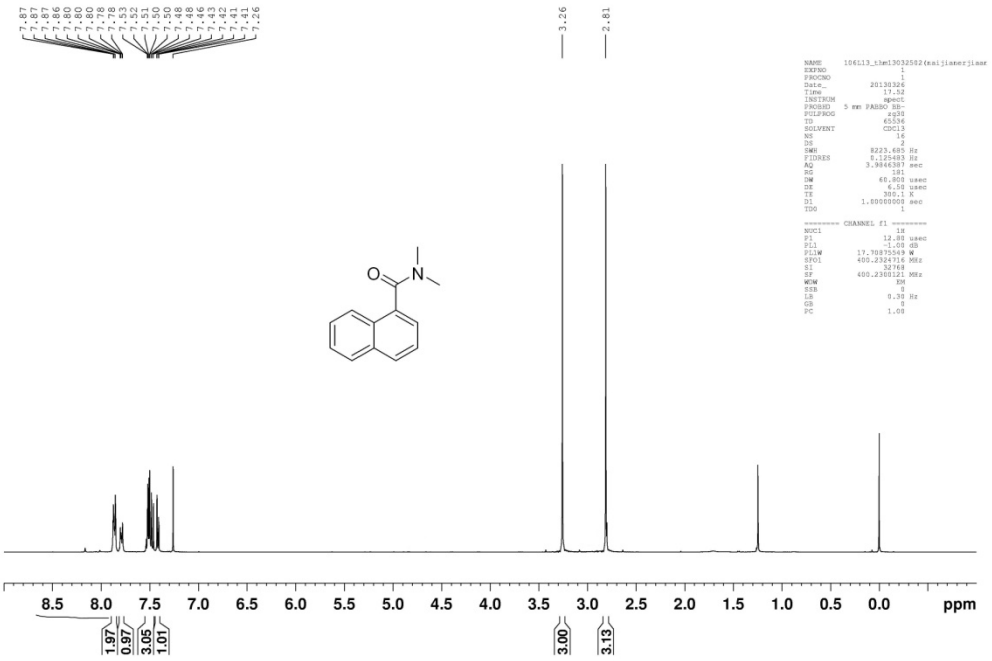


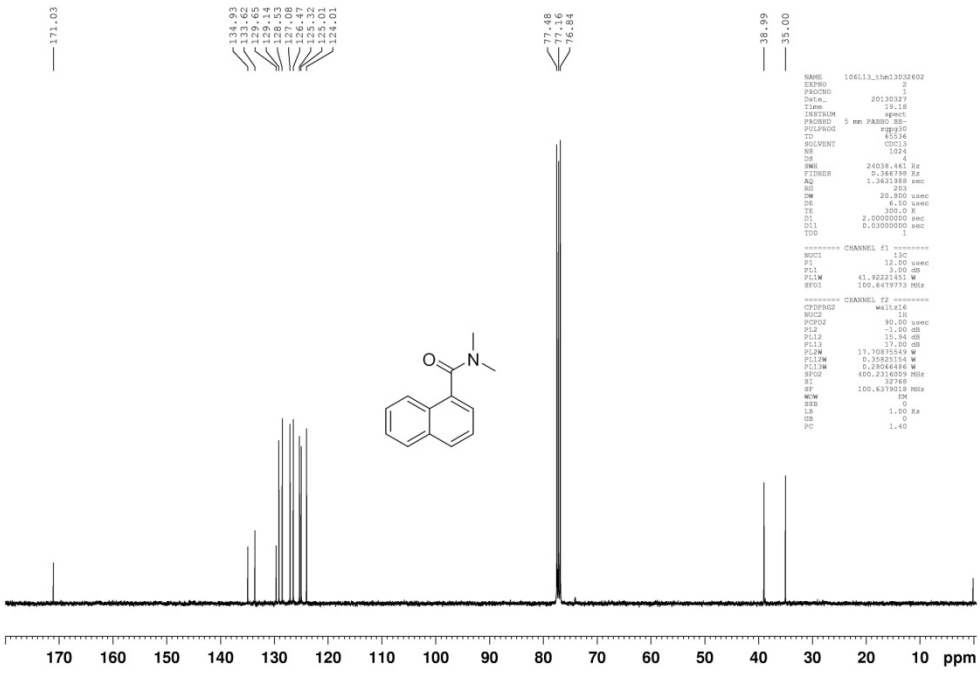
^{1}H NMR, ^{13}C NMR of 3ia:



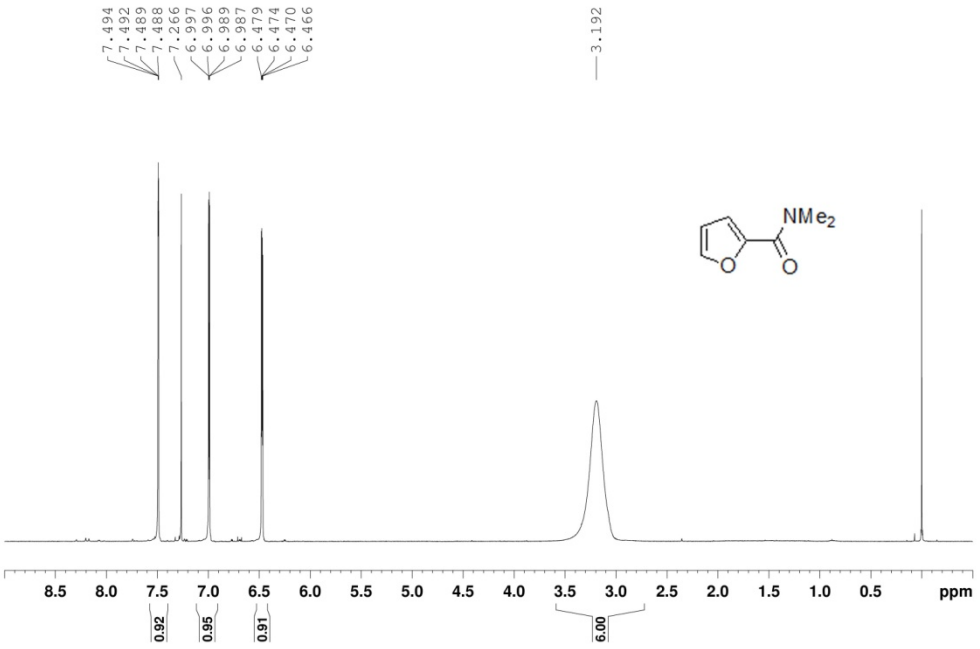


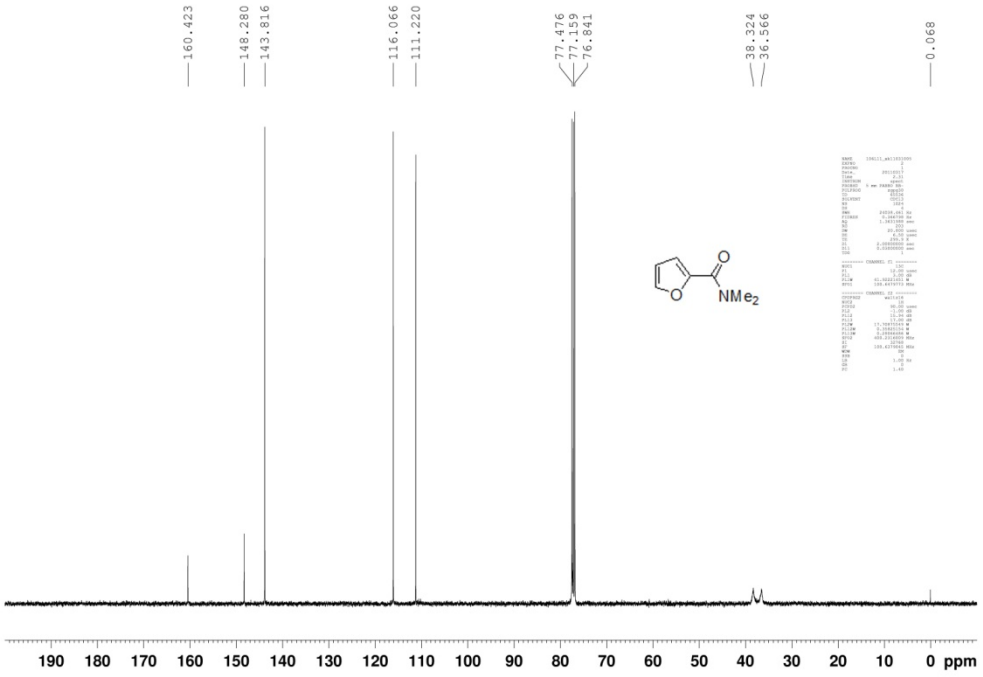
^{1}H NMR, ^{13}C NMR of 3ja:



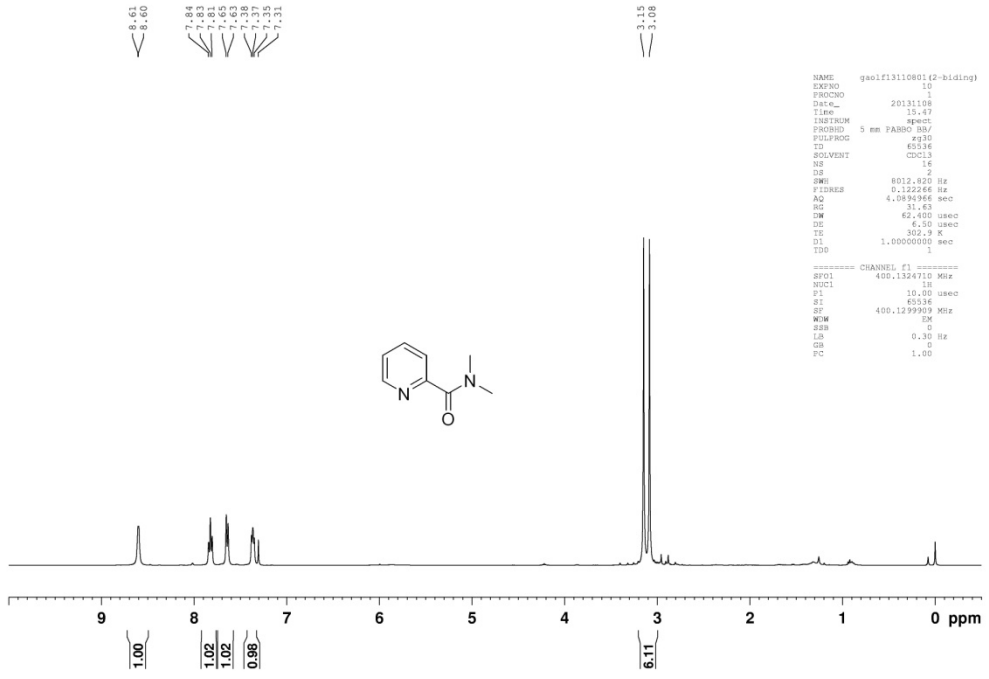


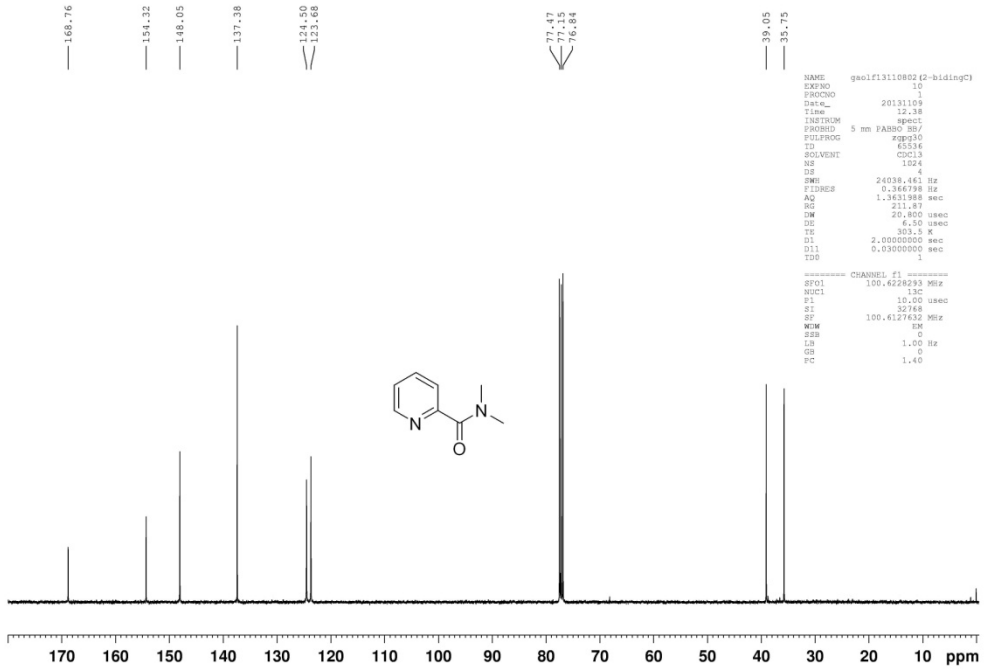
^{1}H NMR, ^{13}C NMR of 3ka:



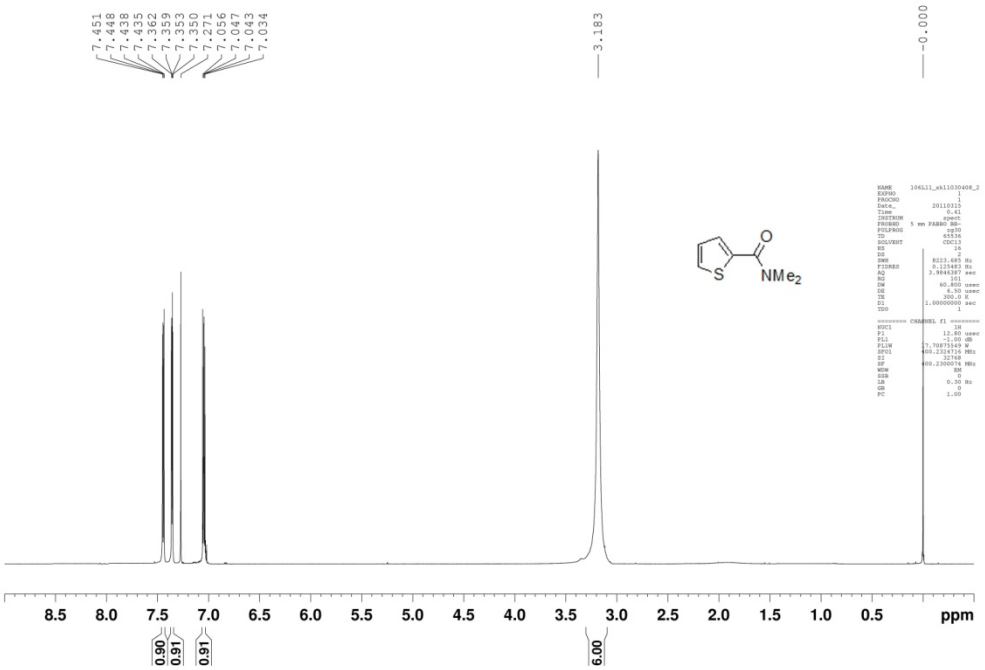


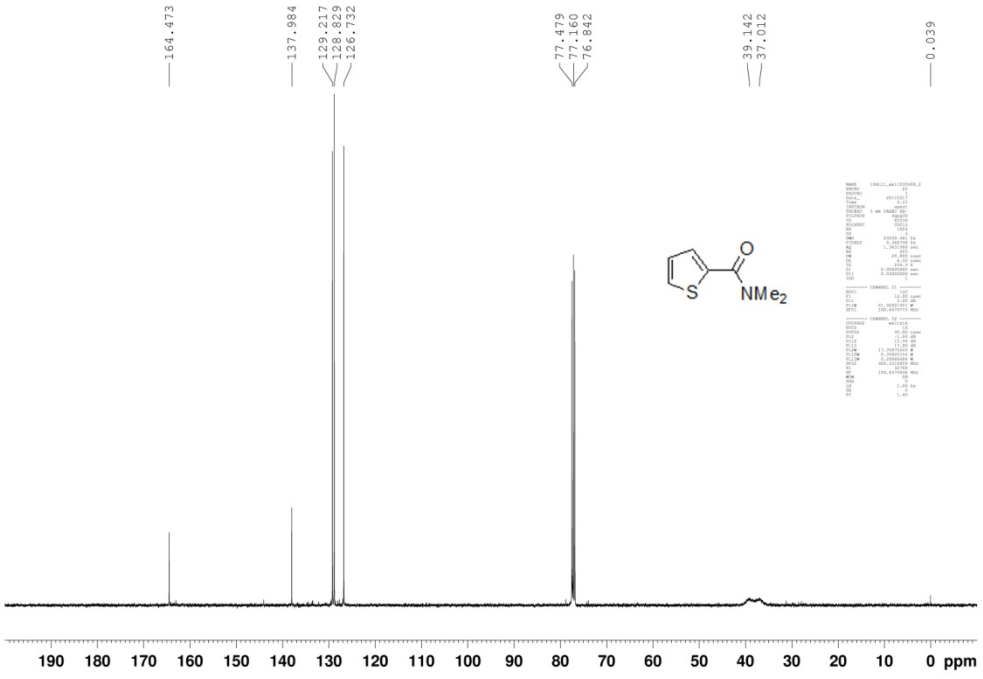
^{1}H NMR, ^{13}C NMR of 3la:



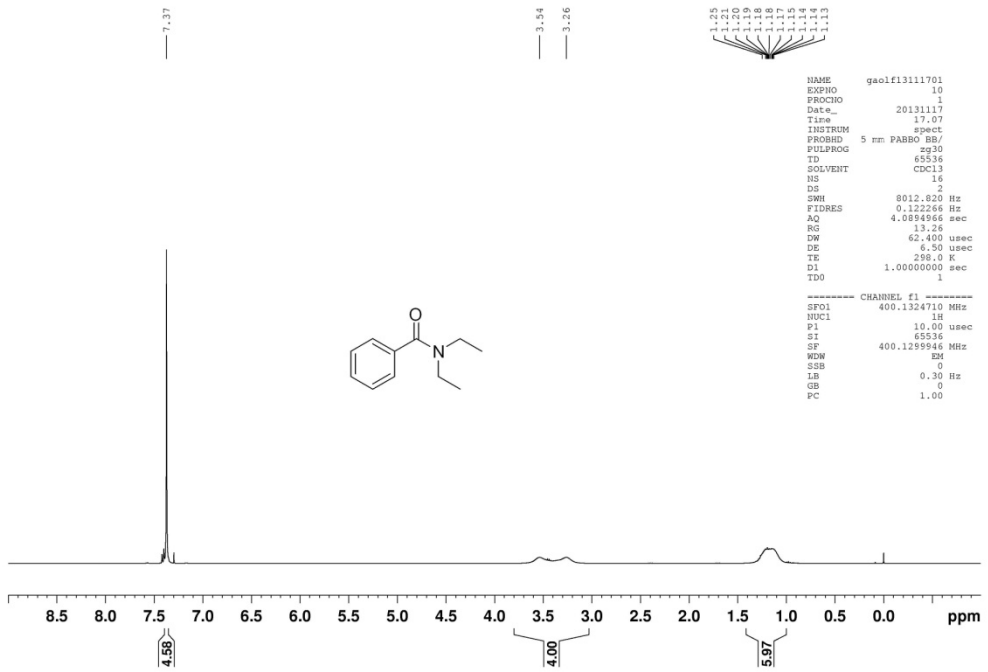


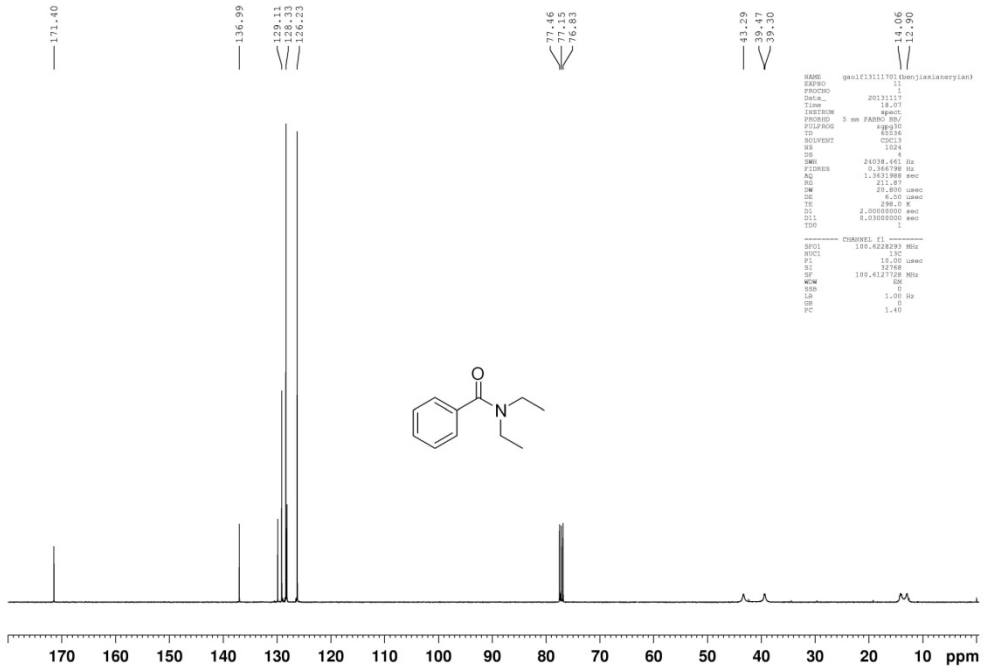
^{1}H NMR, ^{13}C NMR of 3ma:



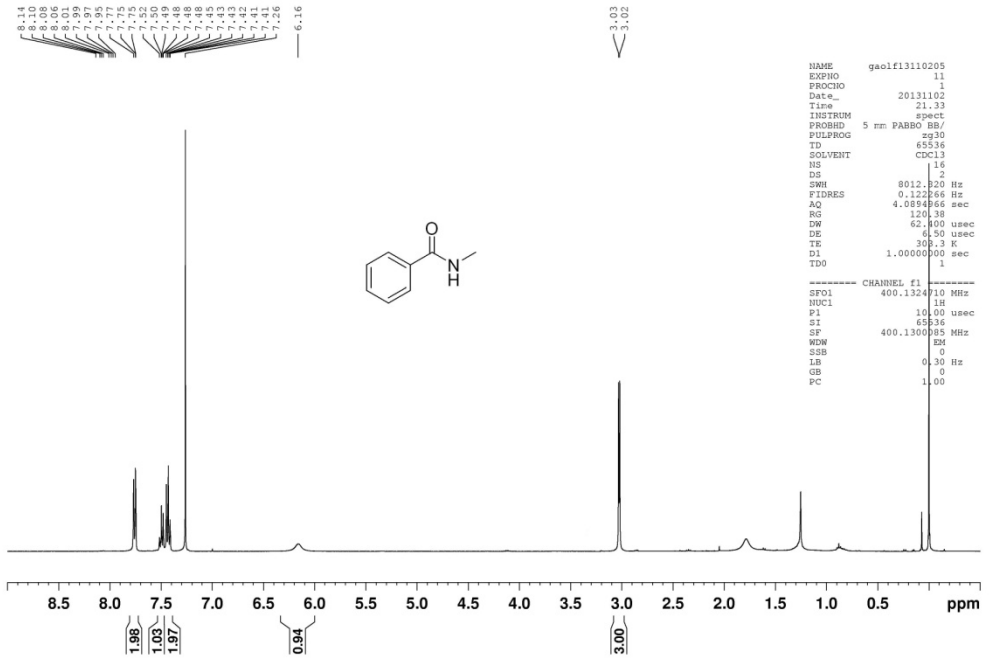


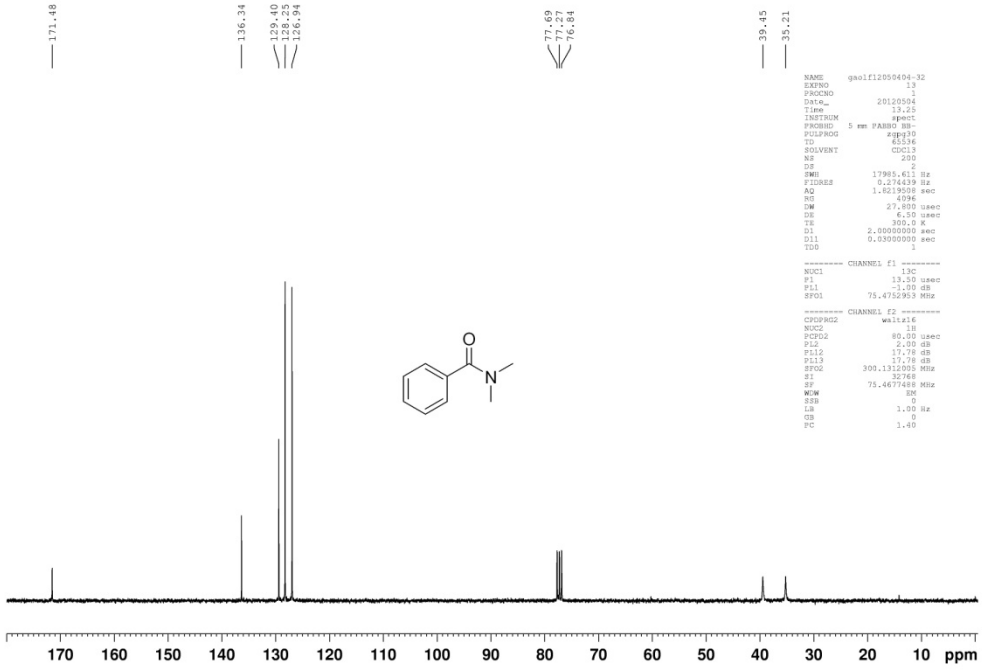
^{1}H NMR, ^{13}C NMR of 3an:



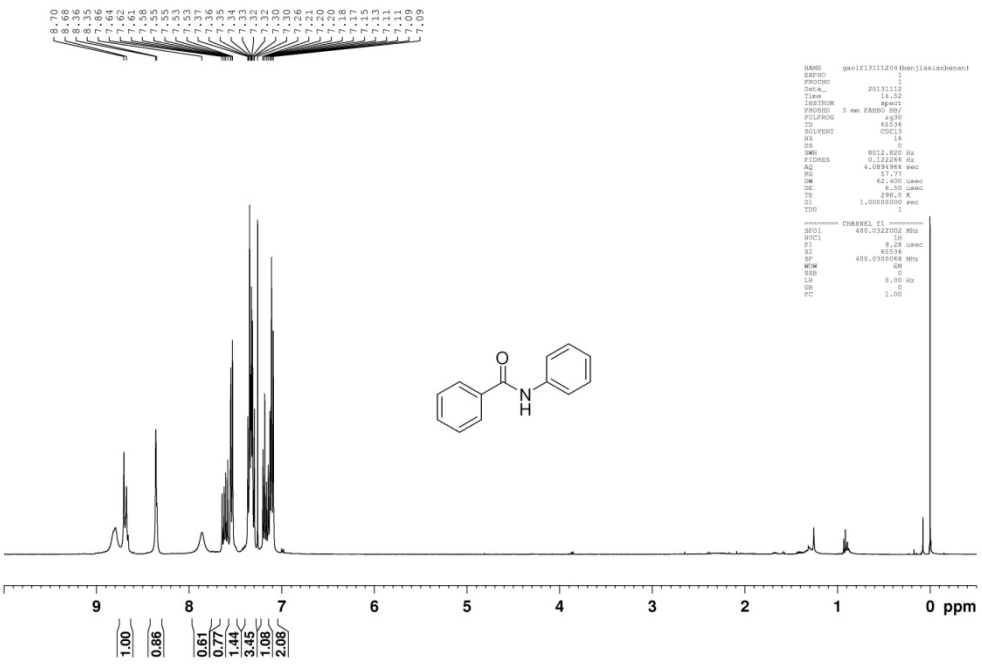


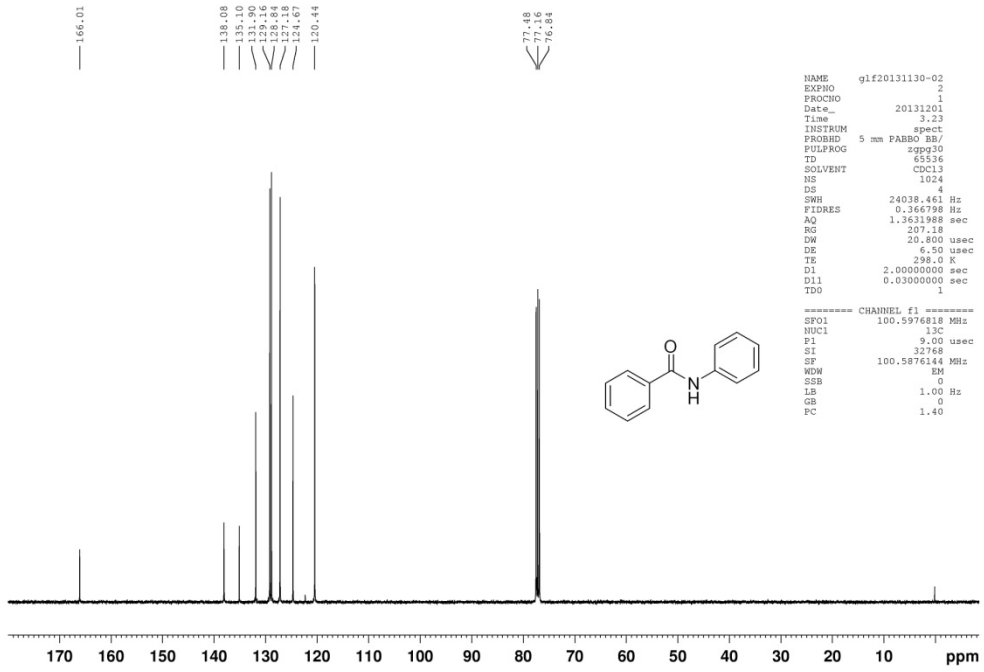
^{1}H NMR, ^{13}C NMR of 3ao:



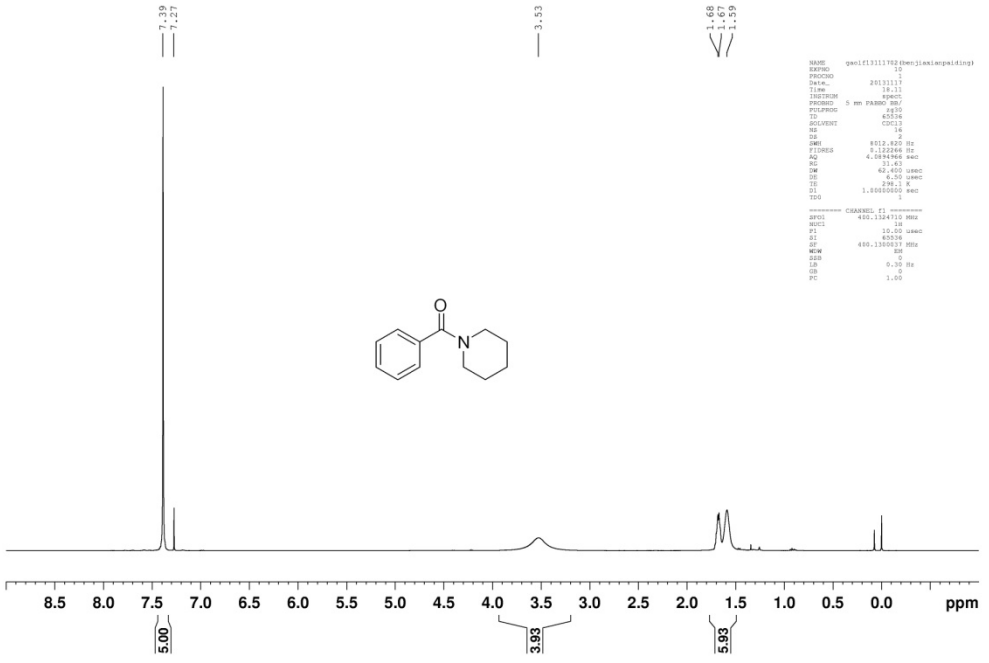


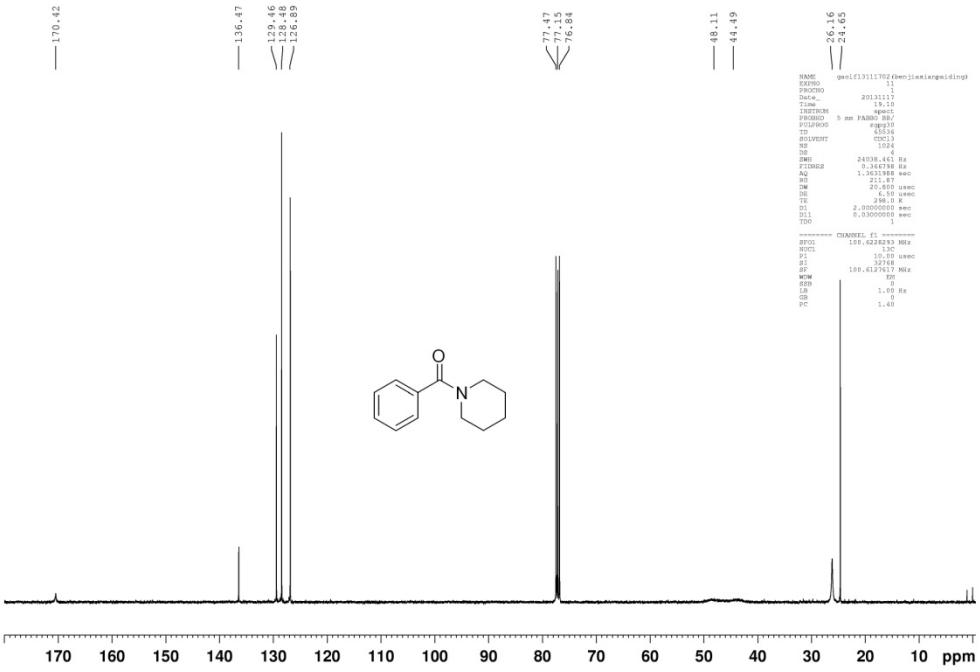
^{1}H NMR, ^{13}C NMR of 3ap:



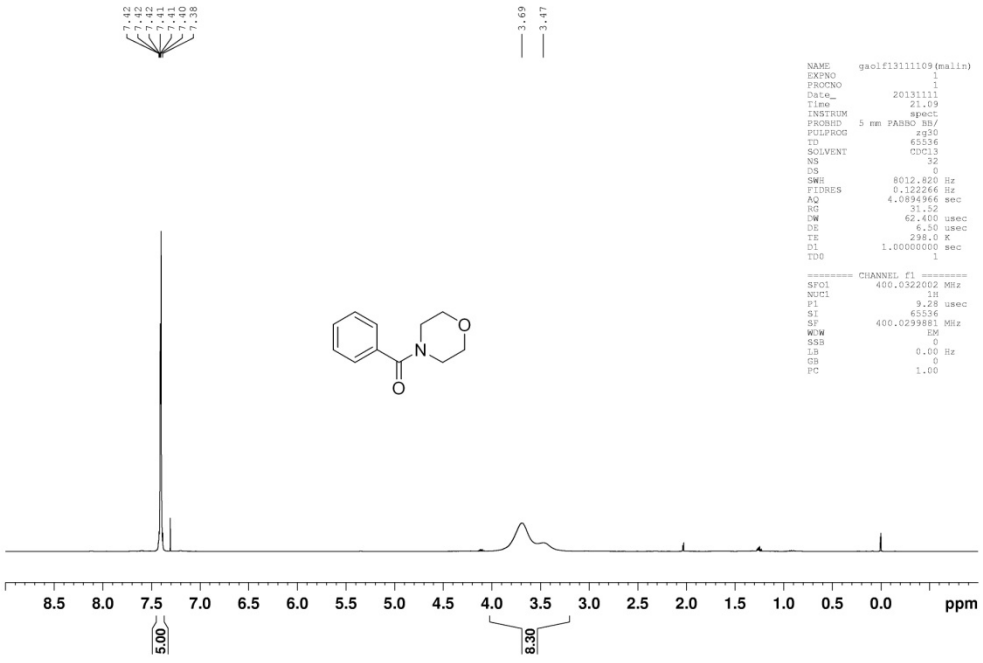


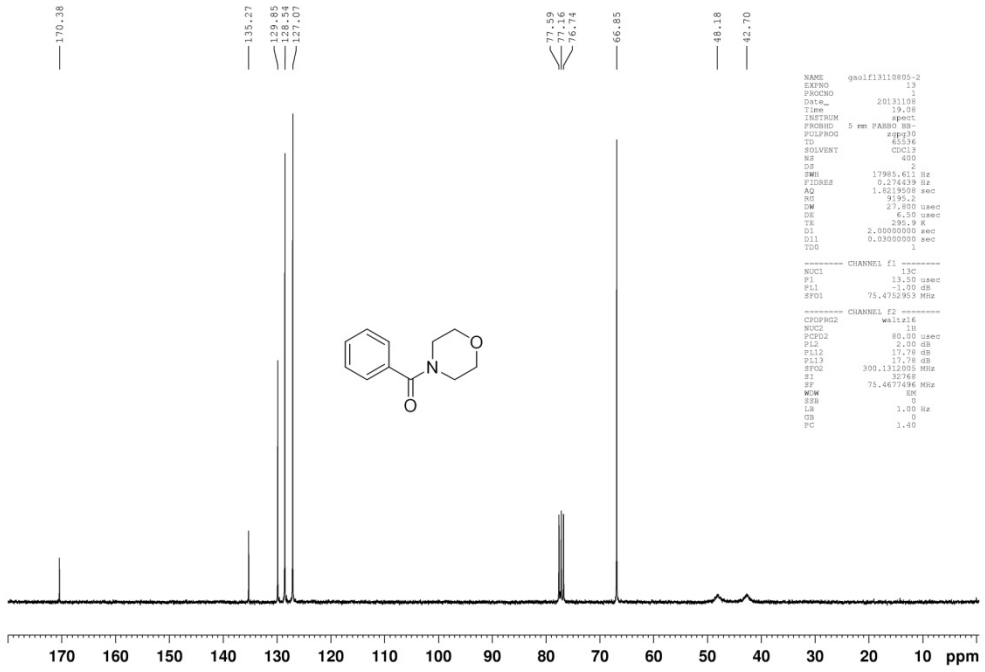
^{1}H NMR, ^{13}C NMR of 3aq:





^{1}H NMR, ^{13}C NMR of 3ar:

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