

Microwave-Assisted Chemoselective Transamidation of Secondary Amides by Selective N-C(O) bond Cleavage Under Catalyst, Additive and Solvent-Free Conditions

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Supporting information

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Experimental Section

General Information

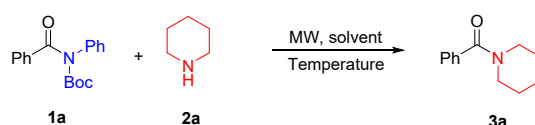
All the chemicals were purchased from Sigma-Aldrich suppliers and used without further purification. Thin layer chromatography (TLC) was conducted with analytical thin layer percolated E. Merck 60 GF254 silica gel plates and spots were visualized using UV light and or iodine vapour and column chromatography was carried out using silica gel of 60-120 mesh size. The melting points were determined in open capillary melting point apparatus and are uncorrected. The microwave irradiation was carried out in CEM's Discover BenchMate single-mode microwave reactor (BenchMate model, USA) system using safe pressure regulation 10-cm³ pressurised vials with "snap-on" caps. ¹H and ¹³C NMR spectra were recorded on Bruker Avance 500 MHz spectrometer in CDCl₃ and DMSO-d₆ using TMS as the internal standard at 500 and 126 MHz frequency respectively. All products synthesised were confirmed by using melting point, ¹H, ¹³C NMR, mass spectra and the reported compounds were compared with the literature data.

Optimization of the reaction conditions

Initially, the unactivated amide, such as *N*-phenyl benzamide, was subjected to the transamidation reaction with piperidine **2a** to obtain the product **3a**. The reaction was conducted under microwave irradiation without catalysts, additives, and promoters at 150 W. However, the desired product **3a** was not obtained under the above-said conditions. Then we performed the reaction with *N*-activated secondary amide, *i.e.*, *N*-Boc, *N*-phenylbenzamide **1a** with piperidine **2a** under the same reaction conditions. To our delight, this reaction gave the product **3a** a good yield. Encouraged by this result, *N*-Boc, *N*-phenylbenzamide **1a**, and piperidine **2a** were chosen as model substrates, and different reaction conditions were examined.

The reaction was carried out in different polar solvents like water, ethanol, methanol, DMF, 1,4- dioxane, dichloromethane, and acetonitrile under controlled microwave irradiation at 150 W in catalyst-free condition for 1 h at 60 °C, which offered only 30 to 55% of the desired product **3a** (Table S1, entry 1-7). Hence, the reaction was carried out in solvent-free conditions at 150 W at 60 °C. Surprisingly the reaction gave the product **3a** in 62% yield within 15 min (Table S1, entry 8). Next, we investigated the effect of microwave power on the yield of the product formation by varying microwave power from 150 W to 350 W in solvent-free conditions at 60 °C. A better yield of 80% was obtained at 300 W in 10 min (Table S1, entry 10). To find the optimal temperature, we conducted the experiments at progressively higher temperatures, 70 °C, 80 °C and 100 °C. Interestingly the reaction at 80 °C under solvent-free conditions was driven to completion with the desired product **3a** to a maximum yield of 93% in 8 min (Table S1, entry 13). Further, an increase in temperature above 80 °C did not show any improvement in yield (Table S1, entries 14). To understand the assistance effect of microwave the model reaction was conducted under the same reaction conditions in the conventional heating method (without microwave) it gave only 20 % yield of the product **3a** even after 4 h (Table S1, entries 15). This suggests that *N*-Boc, *N*-phenylbenzamide **1a**, (1.0 mmol), piperidine **2a**, (1.5 mmol) at 80 °C in MW, 300 W under catalyst and solvent-free is the optimal condition for this transamidation reaction.

Table S1. Optimization of reaction conditions for transamidation of *N*-Boc, *N*-phenylbenzamide under microwave irradiation^[a]



Entry	Solvent	microwave power (W)	Temp. (°C)	Time (min.)	Yields (%) ^[b]
1	Water	150	60	60	30
2	Ethanol	150	60	60	35
3	Methanol	150	60	60	38
4	1,4 Dioxane	150	60	60	32
5	DMF	150	60	60	55
6	DMSO	150	60	60	45
7	Acetonitrile	150	60	60	40
8	-	150	60	15	62
9	-	200	60	12	75
10	-	300	60	10	80
11	-	350	60	10	80
12	-	300	70	10	85
13	-	300	80	8	93
14	-	300	100	8	93
15 ^[c]	-	-	80	4 h	20

^[a]Reaction conditions: *tert*-butylbenzoyl(phenyl)carbamate **1a** (1.0 mmol), piperidine **2a** (1.5 mmol), in solvent (1ml) under Microwave-irradiation. ^[b]Isolated yield. ^[c]Conventional heating at 80 °C for 4 h.

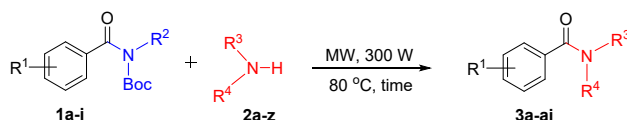
General procedure for the synthesis of amides

All amides used in the study were synthesized by previously reported methods.¹

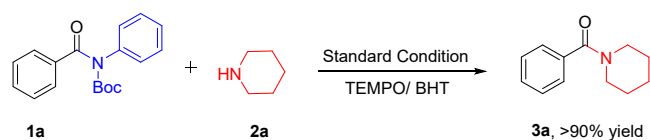
General procedure for Synthesis of *N*-Boc activated secondary amides

N-Boc-activated amides were synthesized according to the reported method.² To an oven-dried round bottom flask, amide (1.0 equiv.), DMAP (0.1 equiv.) and dichloromethane were added, the reaction temperature was maintained at 0 °C to this Boc anhydride (1.5 equiv.) was added dropwise. After the addition of Boc anhydride, the reaction mixture was stirred for 14-24 h at room temperature. The progress of the reaction was monitored with TLC, after the completion of the reaction, mixture was concentrated under reduced pressure and purified by column chromatography and the product was obtained in excellent yield.

General procedure for metal-free transamidation of *N*-Boc activated secondary amides with amines



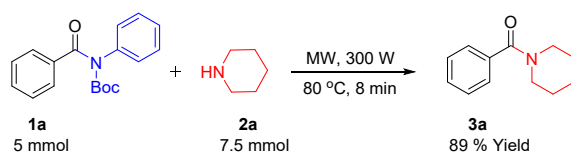
A mixture of appropriate *N*-Boc activated amide (1.0 mmol) and amine (1.5 mmol) was placed in a 10-mL pressurised vials with “snap-on” cap and irradiated in the microwave using 300 W power at 80 °C for 8-10 min. The completion of the reaction was monitored with TLC, after completion the reaction mixture was cooled to room temperature and diluted with ethyl acetate, and washed water. The organic layer was dried over Na₂SO₄ and concentrated under vacuum and the crude product was purified by column chromatography on silica gel using *n*-hexane-ethyl acetate as eluent to give the desired products.



Scheme S1. Control experiments with radical scavengers.

Gram-scale synthesis of 3a

Tested the practical applicability of this protocol in multigram scale synthesis (Scheme S2). The reaction of *N*-Boc, *N*-phenylbenzamide **1a** (1.56 g, 5.0 mmol), and piperidine **2a** (0.64 ml, 7.5 mmol) gave the desired transamidated product **3a** in (1.95g) 89% yield under the optimized reaction conditions.



Scheme S2. Gram-scale procedure for transamidation of *N*-Boc amide with amine.

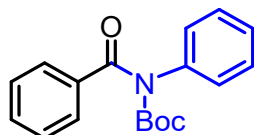
Author Contributions

Vishal Singh: Methodology, investigation, data curation, formal analysis and writing draft; K. Rajput: Investigation, data curation and analysis; A. Mishra: Data curation; S. Singh: Analysis and review; Vandana Srivastava: Supervision, conceptualization, resources, review, and final editing.

Compounds Characterization

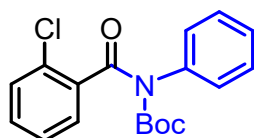
Characterization Data of Starting Materials

tert-butyl benzoyl(phenyl)carbamate (**1a**)²



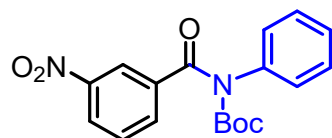
Yield 95%; white solid; m.p. 97 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.76 (d, 2H), 7.54 – 7.53 (m, 1H), 7.47 – 7.43 (m, 4H), 7.37 – 7.36 (m, 1H), 7.30 (d, 2H), 1.25 (s, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 172.9, 153.5, 139.3, 137.1, 131.9, 129.4, 128.4, 128.3, 128.1, 127.9, 83.6, 27.6; HRMS (ESI) for C₁₈H₁₉NO₃ (m/z) [M + H]⁺ calcd: 298.1365, found: 298.1382.

tert-butyl (2-chlorobenzoyl)(phenyl)carbamate (**1b**)³



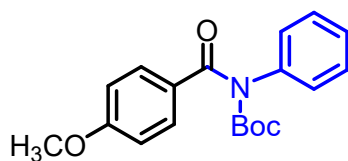
Yield 94%; white solid; m.p. 235–238 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.52 – 7.40 (m, 3H), 7.39 – 7.28 (m, 6H), 1.22 (s, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 170.1, 152.6, 138.8, 138.2, 131.2, 130.6, 129.9, 129.7, 129.2, 128.8, 128.8, 127.5, 84.4, 27.9; HRMS (ESI) for C₁₈H₁₈ClNO₃ (m/z) [M + H]⁺ calcd: 332.0975, found: 332.0963.

tert-butyl (3-nitrobenzoyl)(phenyl)carbamate (**1c**)^{3,4}



Yield 95%; white solid; m.p. 223–225 °C; ¹H NMR (500 MHz, CDCl₃): δ 8.45 (s, 1H), 8.30 (d, 1H), 7.96 – 7.94 (d, 1H), 7.58 – 7.55 (t, 1H), 7.40 – 7.36 (t, 2H), 7.32 – 7.29 (t, 1H), 7.29 – 7.18 (m, 2H), 1.20 (s, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 170.3, 152.9, 148.0, 138.5, 133.8, 129.6, 129.5, 128.4, 128.1, 126.0, 123.0, 84.5, 28.5, 27.7; HRMS (ESI) for C₁₈H₁₈N₂O₅ (m/z) [M + H]⁺ calcd: 343.1216, found: 343.1243.

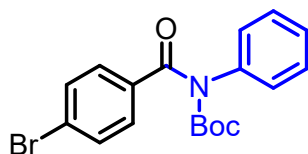
tert-butyl (4-methoxybenzoyl)(phenyl)carbamate (**1d**)⁵



Yield 88%; white solid; m.p. 143–145 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.45 (d, 2H), 7.33 (t, 2H), 7.23 (d, 2H), 7.16 (t, 1H), 6.79 (d, 2H), 3.71 (s, 3H), 1.08 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 172.7, 162.3, 153.8, 138.2, 130.0, 129.6, 128.5, 128.1,

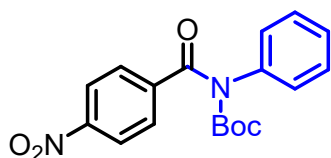
127.4, 113.4, 82.8, 55.4, 27.5; HRMS (ESI) for $C_{19}H_{21}NO_4$ (m/z) [M + H]⁺ calcd: 328.1471, found: 328.1467.

tert-butyl (4-bromobenzoyl)(phenyl)carbamate (1e)⁵



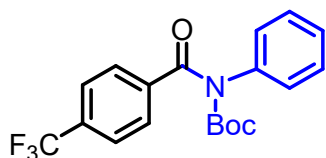
Yield 88%; white solid; m.p. 83–85°C; ¹H NMR (500 MHz, CDCl₃): δ 7.71 (d, 2H), 7.62 (d, 2H), 7.39 – 7.27 (m, 4H), 7.13 (t, 1H), 1.21 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 172.8, 162.4, 153.4, 138.2, 130.2, 129.8, 128.6, 128.2, 127.5, 113.5, 79.3, 27.7; HRMS (ESI) for $C_{18}H_{18}BrNO_3$ (m/z) [M + H]⁺ calcd: 376.0470, found: 376.0483.

tert-butyl (4-nitrobenzoyl)(phenyl)carbamate (1f)⁵



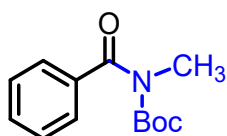
Yield 88%; white solid; (m.p. 103 °C; ¹H NMR (500 MHz, CDCl₃): δ 8.33 (d, 2H), 7.92 (d, 2H), 7.39 – 7.30 (m, 4H), 7.29 – 7.11 (m, 1H), 1.21 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 172.4, 162.0, 153.5, 137.9, 129.8, 129.3, 128.2, 127.8, 127.0, 113.0, 28.6, 27.2; HRMS (ESI) for $C_{18}H_{18}N_2O_5$ (m/z) [M + H]⁺ calcd: 343.1216, found: 343.1204.

tert-butyl b(4-trifluoromethyl)(benzoyl)(phenyl)carbamate (1g)⁵



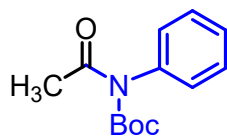
Yield 88%; white solid; m.p. 125–127°C; ¹H NMR (500 MHz, CDCl₃): δ 7.61 (d, 2H), 7.55 (d, 2H), 7.38 – 7.36 (t, 2H), 7.31 (d, 2H), 7.24 (t, 1H), 1.09 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 172.2, 153.4, 141.6, 137.9, 132.7 (q, *J* = 32.8 Hz, 1C), 129.0, 128.6, 128.0, 127.8, 125.6 (q, *J* = 3.8 Hz, 1C), 125.2 (q, *J* = 272.1 Hz, 1C), 84.3, 27.8; ¹⁹F NMR (471 MHz, CDCl₃): δ -63.2; HRMS (ESI) for $C_{19}H_{18}F_3NO_3$ (m/z) [M + H]⁺ calcd: 366.1239, found: 366.1254.

tert-butyl benzoyl(methyl)carbamate (1h)²



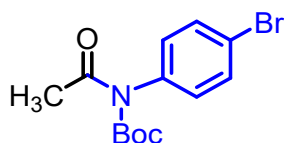
Yield 87%; yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.52 (m, 2H), 7.47 – 7.45 (m, 1H), 7.41 – 7.38 (m, 2H), 3.32 (s, 3H), 1.16 (s, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 173.7, 153.6, 137.9, 130.9, 128.1, 127.5, 83.0, 71.8, 32.6, 27.4; HRMS (ESI) for $C_{13}H_{17}NO_3$ (m/z) [M + H]⁺ calcd: 236.1208, found: 236.1219.

tert-butyl acetyl(phenyl)carbamate (1i)⁶



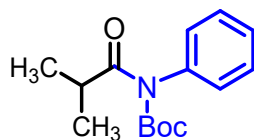
Yield 84%; white solid; (m.p. 58 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.43 – 7.36 (m, 2H), 7.35 – 7.28 (m, 1H), 7.11 – 7.10 (m, 2H), 2.59 (s, 3H), 1.40 (s, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 172.9, 152.8, 138.9, 128.9, 128.2, 127.8, 83.2, 27.8, 26.3; HRMS (ESI) for C₁₃H₁₇NO₃ (m/z) [M + H]⁺ calcd: 236.1208, found: 236.1251.

tert-butyl acetyl(4-bromophenyl)carbamate (1j)⁷



Yield 84%; white solid; (m.p. 122 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.54 (d, 2H), 6.99 (d, 2H), 2.61 (s, 3H), 1.41 (s, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 172.8, 152.4, 137.9, 132.2, 129.9, 121.7, 83.6, 27.9, 26.5; HRMS (ESI) for C₁₃H₁₆BrNO₃ (m/z) [M + H]⁺ calcd: 314.0314, found: 314.0308.

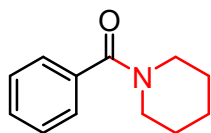
tert-butyl isobutyryl(phenyl)carbamate (1k)⁸



Yield 82%; yellow oil; (¹H NMR (500 MHz, CDCl₃): δ 7.30 – 7.27 (t, 2H), 7.23 – 7.21 (d, 1H) 6.99 – 6.97 (m, 2H), 3.57 – 3.53 (m, 1H), 1.30 (s, 9H), 1.15 (d, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 180.5, 152.8, 139.5, 128.9, 128.2, 128.2, 127.5, 82.9, 34.8, 34.7, 27.9, 19.6; HRMS (ESI) for C₁₅H₂₁NO₃ (m/z) [M + H]⁺ calcd: 264.1521, found: 264.1543.

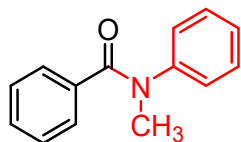
Characterization Data of Transamidation Products

phenyl(piperidin-1-yl)methanone (3a)⁹



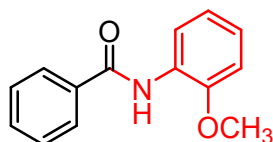
Yield 93%; white solid; m.p. 48 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.43 – 7.29 (m, 5H), 3.74 (s, 2H), 3.36 (s, 2H), 1.77 (s, 2H), 1.70 (s, 2H), 1.54 (s, 2H); ¹³C NMR (126 MHz, CDCl₃): δ 170.5, 135.3, 129.8, 128.6, 127.0, 48.7, 43.0, 24.5; HRMS (ESI) for C₁₂H₁₅NO (m/z) [M + H]⁺ calcd: 190.1189, found: 190.1223.

N-methyl-N-phenylbenzamide (3b)¹⁰



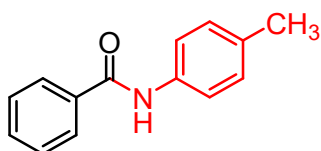
Yield 87%; yellow oil; (¹H NMR (500 MHz, CDCl₃): δ 7.25 (d, 2H), 7.18 – 7.11 (m, 3H), 7.09 – 7.05 (m, 3H), 6.98 – 6.97 (d, 2H), 3.44 (s, 3H); ¹³C NMR (126 MHz, CDCl₃): δ 170.8, 145.0, 126.0, 129.7, 129.3, 128.8, 127.8, 127.0, 123.6, 38.5; HRMS(ESI) for C₁₄H₁₃NO (m/z) [M + H]⁺ calcd: 212.0997, found: 212.1023.

***N*-(2-methoxyphenyl)benzamide (3c)¹¹**



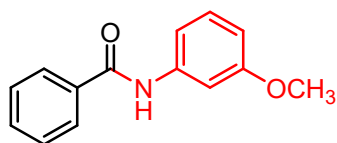
Yield 91%; white solid; m.p. 60 °C; ¹H-NMR (500 MHz, CDCl₃): δ 8.54 (d, 2H), 7.90 (m, 2H), 7.55 – 7.51 (m, 3H), 7.50 (m, 2H), 6.93 (s, 1H), 3.92 (s, 3H); ¹³C-NMR (125 MHz, CDCl₃): δ 165.5, 148.4, 135.6, 131.9, 129.0, 128.1, 127.3, 124.1, 121.5, 121.5, 120.1, 110.2, 56.1; HRMS (ESI) for C₁₄H₁₃NO₂ (m/z) [M + H]⁺ calcd: 228.0946, found: 228.0932.

***N*-(4-methylphenyl)benzamide (3d)¹¹**



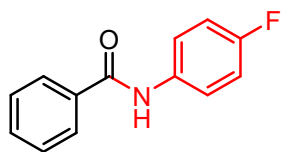
Yield 92%; white solid; m.p. 158 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 10.17 (s, 1H), 7.96 (d, 2H), 7.67 (d, 2H), 7.60 – 7.57 (m, 1H), 7.54 (d, 2H), 7.17 (d, 2H), 2.29 (s, 3H); ¹³C NMR (126 MHz, DMSO-d₆): δ 165.8, 137.1, 135.5, 133.0, 131.9, 129.5, 128.8, 128.1, 120.9, 21.0; HRMS (ESI) for C₁₄H₁₃NO (m/z) [M + H]⁺ calcd: 212.0997, found: 212.0930.

***N*-(3-methoxyphenyl)benzamide (3e)¹¹**



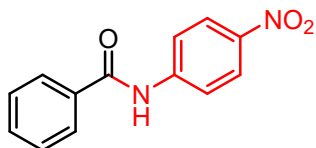
Yield 91%; white solid; m.p. 108 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 7.68 (d, 2H), 7.39 (s, 1H), 7.67 (d, 2H), 7.36 – 7.31 (m, 3H), 7.23 – 7.16 (m, 1H), 7.15 (m, 1H), 6.93 (d, 1H), 6.74 – 6.72 (m, 1H), 3.81 (s, 3H); ¹³C NMR (126 MHz, DMSO-d₆): δ 165.8, 156.7, 138.9, 135.2, 131.3, 129.3, 128.9, 127.1, 112.2, 110.2, 105.3, 55.6; HRMS (ESI) for C₁₄H₁₃NO₂ (m/z) [M + H]⁺ calcd: 228.0946, found: 228.0957.

***N*-(4-fluorophenyl)benzamide (3f)¹²**



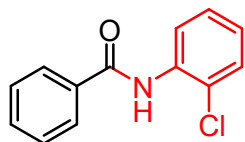
Yield 88%; white solid m.p. 185–187°C; ¹H NMR (500 MHz, DMSO-d₆): δ 10.36 (s, 1H), 8.00 – 7.85 (d, 2H), 7.83 (d, 2H), 7.65 (m, 1H), 7.60 (d, 2H), 7.59 – 7.23 (m, 2H); ¹³C NMR (126 MHz, DMSO-d₆): δ 165.8, 159.6 (d, *J* = 240.6 Hz, 1C), 135.7, 135.0 (d, *J* = 6.3 Hz, 1C), 131.9, 128.7, 127.9, 122.5 (d, *J* = 7.6 Hz, 1C), 115.5 (d, *J* = 22.7 Hz, 1C); ¹⁹F NMR (471 MHz, CDCl₃): δ - 117.8; HRMS (ESI) for C₁₃H₁₀FNO (m/z) [M + H]⁺ calcd: 216.0746, found: 216.0732.

***N*-(4-nitrophenyl)benzamide (3g)¹¹**



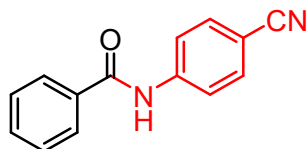
Yield 82%; white solid; m.p. 196 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 10.84 (s, 1H), 8.29 (d, 2H), 8.08 (d, 2H), 7.67 – 7.64 (m, 1H), 8.00 (d, 2H), 7.59 – 7.57 (m, 2H); ¹³C NMR (126 MHz, DMSO-d₆): δ 166.5, 145.6, 142.7, 134.4, 132.5, 128.8, 128.1, 125.0, 120.1; HRMS (ESI) for C₁₃H₁₀N₂O₃ (m/z) [M + H]⁺ calcd: 243.0691, found: 243.0684.

***N*-(2-chlorophenyl)benzamide (3h)¹¹**



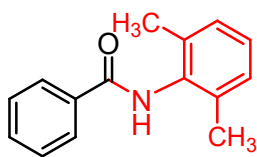
Yield 80%; white solid; m.p. 97 °C; ¹H NMR (500 MHz, CDCl₃): δ 8.58 (d, 1H), 8.45 (s, 1H), 7.93 (d, 2H), 7.60 – 7.58 (t, 1H), 7.57 – 7.50 (t, 2H), 7.42 (dd, 1H), 7.34 – 7.26 (m, 1H), 7.08 – 7.07 (m, 1H); ¹³C NMR (126 MHz, CDCl₃): δ 165.6, 135.0, 134.9, 132.5, 129.3, 129.2, 128.2, 127.4, 125.0, 123.3, 121.8; HRMS (ESI) for C₁₃H₁₀ClNO (m/z) [M + H]⁺ calcd: 231.0451, found: 231.0434.

***N*-(4-cyanophenyl)benzamide (3i)¹²**



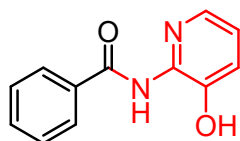
Yield 80%; yellow solid; m.p. 168–170 °C; ¹H NMR (500 MHz, CDCl₃): δ 8.03 (s, 1H), 7.88 (d, 1H), 7.79 (d, 1H), 7.67 – 7.64 (m, 3H), 7.61 – 7.50 (m, 2H), 7.48 – 7.34 (m, 2H); ¹³C NMR (126 MHz, CDCl₃): δ 173.2, 142.1, 133.6, 133.5, 129.5, 129.2, 128.9, 127.3, 120.0; HRMS (ESI) for C₁₄H₁₀N₂O (m/z) [M + H]⁺ calcd: 223.0793, found: 223.0798.

***N*-(2,6-dimethylphenyl)benzamide (3j)**¹³



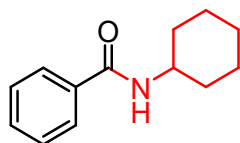
Yield 84%; white solid; m.p. 167–169 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.95 (d, 2H), 7.61 – 7.51 (t, 1H), 7.43 (t, 2H), 7.28 (s, 1H), 7.19 – 7.14 (m, 3H), 2.31 (s, 6H); ¹³C NMR (126 MHz, CDCl₃): δ 166.3, 135.9, 134.9, 134.2, 132.2, 129.1, 128.6, 127.9, 127.6, 18.8; HRMS (ESI) for C₁₅H₁₅NO (m/z) [M + H]⁺ calcd: 226.1154, found: 226.1173.

***N*-(3-hydroxypyridin-2-yl)benzamide (3k)**¹⁴



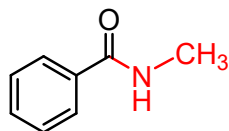
Yield 90%; White solid; m.p. 95–97 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 7.97 (d, 1H), 7.71 (d, 2H), 7.40 – 7.35 (m, 1H), 7.29 (m, 2H), 7.27 (s, 1H), 7.24 (d, 1H), 7.23 (m, 1H), 4.54 (s, 1H); ¹³C NMR (126 MHz, DMSO-d₆): δ 168.9, 146.7, 143.3, 138.9, 134.5, 131.4, 129.9, 129.9, 124.1, 120.4; HRMS (ESI) for C₁₂H₁₀N₂O₂ (m/z) [M + H]⁺ calcd: 215.0742, found: 215.0778.

***N*-cyclohexylbenzamide (3l)**¹³

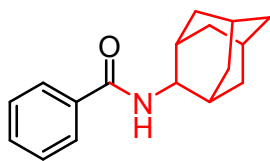


Yield 93%; white solid; m.p. 154–156 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.75 – 7.74 (m, 2H), 7.49 – 7.42 (m, 1H), 7.40 – 7.26 (d, 2H), 5.99 (s, 1H), 4.00 – 3.95 (d, 1H), 2.04 – 2.02 (m, 2H), 1.76 – 1.74 (m, 2H), 1.69 – 1.64 (m, 1H), 1.46 – 1.41 (m, 2H), 1.39 – 1.20 (m, 2H); ¹³C NMR (126 MHz, CDCl₃): δ 166.9, 135.4, 131.6, 128.8, 127.2, 49.0, 33.6, 25.9, 25.2; HRMS (ESI) for C₁₃H₁₇NO (m/z) [M + H]⁺ calcd: 204.1310, found: 204.1329.

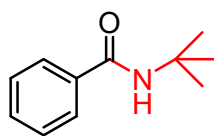
***N*-methylbenzamide (3m)**¹³



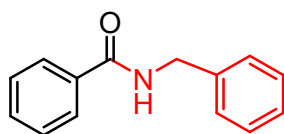
Yield 86%; yellow solid; m.p. 82–83 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.76 – 7.74 (m, 2H), 7.48 – 7.39 (m, 1H), 7.38 – 7.26 (m, 2H), 6.42 (s, 1H), 2.99 – 2.98 (m, 3H); ¹³C NMR (126 MHz, CDCl₃): δ 168.6, 134.8, 131.5, 128.7, 127.0, 27.0; HRMS (ESI) for C₈H₉NO (m/z) [M + H]⁺ calcd: 136.0684, found: 136.0670.

***N*-(3*s*,5*s*,7*s*)-adamantan-1-yl)benzamide (3n)¹⁵**

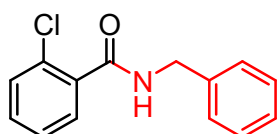
Yield 78%; white solid; (m.p. 143–145 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.74 – 7.72 (m, 2H), 7.50 – 7.46 (m, 1H), 7.44 – 7.28 (t, 2H), 5.82 (s, 1H), 2.15 (s, 9H), 1.77 (d, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 166.9, 162.6, 136.3, 134.8, 131.3, 130.8, 129.1, 129.1, 128.7, 126.9, 52.5, 41.9, 36.6, 29.7; HRMS (ESI) for C₁₇H₂₁NO (m/z) [M + H]⁺ calcd: 256.1623, found: 256.1641.

***N*-(tert-butyl)benzamide (3o)¹³**

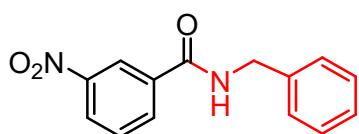
Yield 81%; white solid; m.p. 136 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.73 – 7.71 (m, 2H), 7.47 – 7.43 (m, 1H), 7.40 – 7.26 (m, 2H), 1.48 (m, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 167.0, 136.0, 131.23, 128.6, 126.8, 51.7, 29.0; HRMS (ESI) for C₁₁H₁₅NO (m/z) [M + H]⁺ calcd: 178.1154, found: 178.1169.

***N*-benzylbenzamide (3p)¹⁶**

Yield 92%; white solid; m.p. 103–105 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.83 (d, 2H), 7.55 (t, 1H), 7.48 – 7.39 (m, 2H), 7.35 – 7.29 (m, 4H), 6.43 (m, 1H), 4.69 (d, 2H); ¹³C NMR (126 MHz, CDCl₃): δ 167.5, 138.4, 134.6, 131.8, 129.0, 128.8, 128.1, 127.8, 127.1, 44.4; HRMS (ESI) for C₁₄H₁₃NO (m/z) [M + H]⁺ calcd: 212.0997, found: 212.0981.

***N*-benzyl-4-fluorobenzamide (3q)⁶**

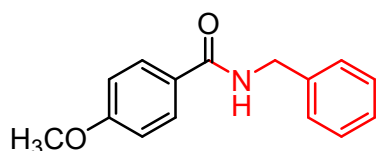
Yield 92%; white solid; m.p. 104–106 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.67 – 7.37 (m, 1H), 7.35 – 7.26 (m, 7H), 6.54 (d, 1H), 4.66 (d, 2H); ¹³C NMR (126 MHz, CDCl₃): δ 165.7, 152.4, 149.2, 144.6, 131.8, 129.0, 120.7, 99.3, 59.8, 53.9, 18.3, 14.5; HRMS (ESI) for C₁₄H₁₂ClNO (m/z) [M + H]⁺ calcd: 246.0607, found: 246.0618.

***N*-benzyl-3-nitrobenzamide (3r)⁶**

Yield 92%; white solid; m.p. 94–96 °C; ¹H NMR (500 MHz, CDCl₃): δ 8.62 (s, 1H), 8.35 (d, 1H), 8.19 (d, 1H), 7.64 (m, 1H), 7.36 – 7.35 (m, 5H), 6.90 (t, 1H), 4.67 (d, 2H); ¹³C NMR (126

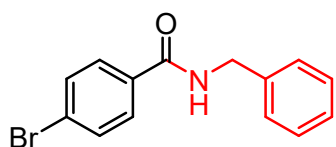
MHz, CDCl₃): δ 165.0, 148.1, 137.5, 135.9, 133.3, 129.9, 128.9, 127.9, 127.9, 126.1, 121.8, 44.4; HRMS (ESI) for C₁₄H₁₂N₂O₃ (m/z) [M + H]⁺ calcd: 257.0848, found: 257.0832.

***N*-benzyl-4-methoxybenzamide (3s)**¹⁷



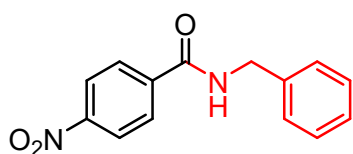
Yield 86%; white solid; m.p. 125 °C; ¹H NMR (500 MHz, CDCl₃): δ 8.00 (d, 1H), 7.67 (d, 2H), 7.25 – 7.16 (m, 4H), 6.89 (d, 2H), 6.80 (s, 1H), 4.54 (d, 2H), 3.80 (s, 3H); ¹³C NMR (126 MHz, CDCl₃): δ 167.1, 167.1, 164.8, 162.5, 162.5, 138.6, 133.0, 129.08, 129.0, 128.1, 127.8, 126.9, 121.5, 114.3, 114.0, 55.6, 44.3; HRMS (ESI) for C₁₅H₁₅NO₂ (m/z) [M + H]⁺ calcd: 242.1103, found: 242.1137.

***N*-benzyl-4-bromobenzamide (3t)**⁶



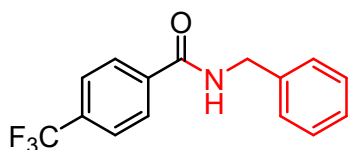
Yield 86%; white solid; m.p. 161–162 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 8.41 (s, 1H), 7.47 (d, 2H), 7.35 (d, 2H), 6.69 – 6.60 (m, 5H), 4.49 (d, 2H); ¹³C NMR (126 MHz, DMSO-d₆): δ 165.6, 140.0, 138.0, 131.3, 130.4, 130.3, 128.7, 127.7, 127.2, 121.0, 120.9, 53.9; HRMS (ESI) for C₁₄H₁₂BrNO (m/z) [M + H]⁺ calcd: 290.0102, found: 290.0126.

***N*-benzyl-4-nitrobenzamide (3u)**¹⁷



Yield 91%; white solid; (m.p. 138–140 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 9.06 (s, 1H), 7.99 (d, 2H), 7.33 – 7.23 (m, 7H), 4.49 (d, 2H); ¹³C NMR (126 MHz, DMSO-d₆): δ 165.6, 163.4, 140.0, 131.3, 130.4, 130.3, 128.7, 127., 127.2, 115.9, 115.6, 43.1; HRMS (ESI) for C₁₄H₁₂N₂O₃ (m/z) [M + H]⁺ calcd: 257.0848, found: 257.0819.

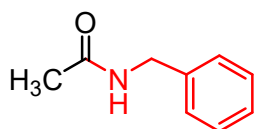
***N*-benzyl-4-fluorobenzamide (3v)**⁶



Yield 92%; white solid; m.p. 146–167 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.79 – 7.66 (m, 3H), 7.65 – 7.61 (m, 3H), 7.49 (t, 2H), 7.26 – 7.18 (m, 1H), 4.98 (s, 2H); ¹³C NMR (126 MHz, CDCl₃): δ 166.1, 137.7, 137.6, 133.4 (q, *J* = 32.7 Hz, 1C), 128.9, 127.9,

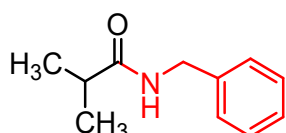
127.8, 127.5, 125.7 (q, $J = 3.8$ Hz, 1C), 124.7 (q, $J = 273.4$ Hz, 1C), 44.2; ^{19}F NMR (471 MHz, CDCl_3): δ - 62.9; HRMS (ESI) for $\text{C}_{15}\text{H}_{12}\text{F}_3\text{NO}$ (m/z) [$\text{M} + \text{H}$] $^+$ calcd: 280.0871, found: 280.0855.

***N*-benzylacetamide (3w)⁶**



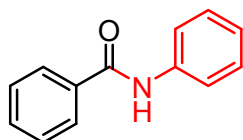
Yield 89%; white solid; m.p. 60 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.27 – 7.15 (m, 5H), 6.23 (s, 1H), 4.27 (d, 2H), 1.87 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3): δ 170.2, 138.4, 128.7, 127.9, 127.5, 59.7, 43.7, 23.2; HRMS (ESI) for $\text{C}_9\text{H}_{11}\text{NO}$ (m/z) [$\text{M} + \text{H}$] $^+$ calcd: 150.0841, found: 150.0868.

***N*-benzylisobutyramide (3x)¹⁸**



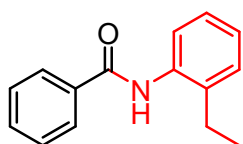
Yield 89%; white solid; m.p. 91 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.33 (d, 2H), 7.32 – 7.26 (m, 3H), 5.82 (s, 1H), 4.43 (d, 2H), 2.41 – 2.36 (m, 1H), 1.19 (d, 6H); ^{13}C NMR (126 MHz, DMSO-d_6): δ 176.9, 138.7, 128.9, 127.9, 127.6, 43.6, 35.8, 19.8; HRMS (ESI) for $\text{C}_{11}\text{H}_{15}\text{NO}$ (m/z) [$\text{M} + \text{H}$] $^+$ calcd: 178.1154, found: 178.1163.

***N*-phenylbenzamide (3y)¹¹**



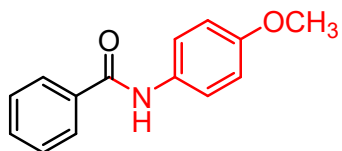
Yield 89%; white solid; m.p. 164 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.90-7.88 (d, 3H), 7.68 – 7.57 (d, 2H), 7.56 – 7.48 (t, 1H), 7.40-7.37 (t, 2H), 7.19 (t, 2H), 7.16 (t, 1H); ^{13}C NMR (126 MHz, CDCl_3): δ 165.8, 137.9, 135.0, 131.8, 129.1, 128.8, 127.0, 124.6, 120.3; HRMS (ESI) for $\text{C}_{13}\text{H}_{11}\text{NO}$ (m/z) [$\text{M} + \text{H}$] $^+$ calcd: 198.0841, found: 198.0853.

***N*-(2-ethylphenyl)benzamide (3z)⁶**



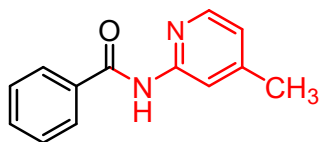
Yield 82%; white solid; m.p. 159 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.97 (d, 1H), 7.90 (d, 2H), 7.60 (s, 1H), 7.58 (t, 1H), 7.52 (t, 2H), 7.29 (t, 2H), 7.19 (t, 1H), 2.73 (q, 2H), 1.32 (t, 3H); ^{13}C NMR (126 MHz, CDCl_3): δ 166.0, 135.6, 135.3, 135.2, 132.0, 129.0, 128.8, 127.2, 127.0, 125.9, 124.1, 24.6, 14.2; HRMS (ESI) for $\text{C}_{15}\text{H}_{15}\text{NO}$ (m/z) [$\text{M} + \text{H}$] $^+$ calcd: 226.1154, found: 226.1132.

***N*-(4-methoxyphenyl)benzamide (3aa)¹⁵**



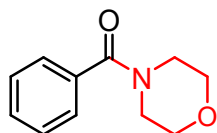
Yield 91%; green solid; m.p. 154–156 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.89 (m, 3H), 7.55 (m, 3H), 7.50 – 7.47 (m, 2H), 6.29 (d, 2H), 3.83 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3): δ 165.7, 135.0, 131.7, 131.0, 128.7, 127.0, 122.2, 114.3, 99.3, 55.2; HRMS (ESI) for $\text{C}_{14}\text{H}_{13}\text{NO}_2$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 228.0946, found: 228.0977.

***N*-(4-methylpyridin-2-yl)benzamide (3ab)¹⁴**



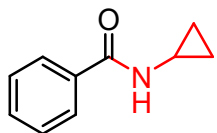
Yield 90%; White solid; m.p. 115–116 °C; ^1H NMR (500 MHz, DMSO-d_6): δ 8.12 (d, 1H), 7.76 (d, 2H), 7.63 (s, 1H), 7.63 – 7.51 (m, 1H), 7.33 – 7.28 (m, 2H), 7.23 (s, 1H), 7.06 (s, 1H), 2.31 (s, 3H); ^{13}C NMR (126 MHz, DMSO-d_6): δ 165.8, 152.8, 152.3, 148.2, 133.0, 131.9, 129.5, 128.8, 127.7, 120.8, 19.2; HRMS (ESI) for $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 213.0950, found: 213.0978.

Morpholino(phenyl)methanone (3ac)⁹



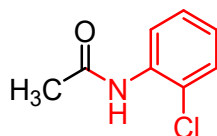
Yield 91%; yellow solid; m.p. 73–75 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.42 – 7.40 (m, 5H), 3.77 – 3.45 (m, 8H), ^{13}C NMR (126 MHz, CDCl_3): δ 170.5, 135.3, 129.9, 128.6, 127.0, 66.9, 48.2, 42.6; HRMS (ESI) for $\text{C}_{11}\text{H}_{13}\text{NO}_2$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 192.0946, found: 192.0955.

***N*-cyclopropylbenzamide (3ad)⁶**



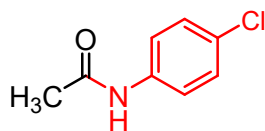
Yield 83%; white solid; m.p. 55–57 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.65 (d, 2H), 7.38 – 7.17 (m, 3H), 6.36 (d, 1H), 2.80 – 2.77 (m, 1H), 0.75 (s, 2H), 0.52 (s, 2H); ^{13}C NMR (126 MHz, CDCl_3): δ 169.1, 134.6, 131.6, 128.7, 127.0, 23.3, 6.9; HRMS (ESI) for $\text{C}_{10}\text{H}_{11}\text{NO}$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 162.0841, found: 162.0867.

***N*-(2-chlorophenyl)acetamide (3ae)¹⁹**



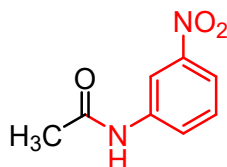
Yield 84%; white solid; m.p. 84–86 °C; ^1H NMR (500 MHz, CDCl_3): δ 8.28 (d, 1H), 7.55 (s, 1H, NH), 7.29 (d, 1H), 7.20 (m, 1H), 7.19 – 6.19 (m, 1H), 2.16 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3): δ 168.4, 134.7, 129.1, 127.8, 124.7, 122.7, 121.8, 25.0; HRMS (ESI) for $\text{C}_8\text{H}_8\text{ClNO}$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 170.0294, found: 170.0263.

***N*-(4-chlorophenyl)acetamide (3af)²⁰**



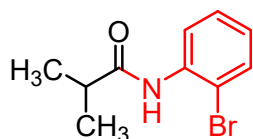
Yield 87%; white solid; m.p. 178 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.75 (s, 1H), 7.42 (m, 4H), 2.17 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3): δ 168.4, 136.9, 131.9, 121.5, 116.9, 24.6; HRMS (ESI) for $\text{C}_8\text{H}_8\text{ClNO}$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 170.0294, found: 170.0298.

***N*-(2-nitrophenyl)acetamide (3ag)²⁰**



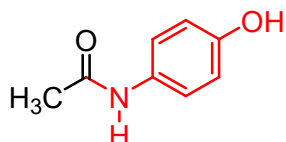
Yield 83%; white solid; m.p. 93 °C; ^1H NMR (500 MHz, CDCl_3): δ 8.57 (s, 1H), 8.08 (s, 1H), 7.73 (d, 1H), 7.54 (d, 1H), 7.33 – 7.28 (m, 1H), 2.17 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3): δ 168.8, 150.7, 138.0, 127.3, 124.3, 122.4, 120.1, 24.5; HRMS (ESI) for $\text{C}_8\text{H}_8\text{N}_2\text{O}_3$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 181.0535, found: 181.0553.

***N*-(2-bromophenyl)isobutyramide (3ah)¹⁸**



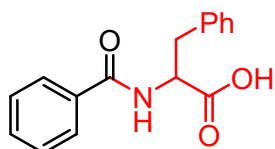
Yield 82%; white solid; m.p. 95 °C; ^1H NMR (500 MHz, CDCl_3): δ 8.42 (d, 1H), 7.73 (s, 1H), 7.39 (d, 1H), 7.30 (m, 2H), 7.29 – 7.03 (m, 1H), 4.01 (d, 2H), 2.65 – 2.59 (m, 1H), 1.10 (d, 6H); ^{13}C NMR (126 MHz, CDCl_3): δ 175.29, 134.80, 129.04, 127.88, 124.57, 122.57, 129.02, 121.67, 37.12, 19.70; HRMS(ESI) for $\text{C}_{10}\text{H}_{12}\text{BrNO}$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 242.0102, found: 242.0132.

***N*-(4-hydroxyphenyl)acetamide (3ai)**



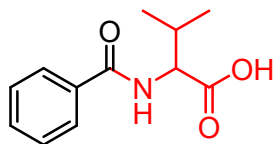
Yield 84%; white solid; m.p. 168 °C; ^1H NMR (500 MHz, DMSO-d_6): δ 9.64 (s, 1H), 9.13 (s, 1H), 7.34 – 7.32 (m, 2H), 6.68 – 6.66 (m, 2H), 1.98 (s, 3H); ^{13}C NMR (126 MHz, DMSO-d_6): δ 167.5, 153.1, 131.0, 120.8, 114.9, 23.7; HRMS (ESI) for $\text{C}_8\text{H}_9\text{NO}_2$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 152.0633, found: 152.0701.

benzoylphenylalanine (6a)



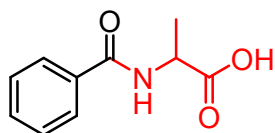
Yield 87%; white solid; m.p. 181–183 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.61 (d, 2H), 7.43 (t, 1H), 7.33 (t, 2H), 7.23 – 7.16 (m, 3H), 7.13 (d, 2H), 6.59 (d, 1H), 5.01 (t, 1H), 3.30 – 3.16 (dd, 2H); ^{13}C NMR (126 MHz, CDCl_3): δ 174.6, 167.8, 135.7, 133.4, 132.0, 129.4, 128.7, 127.3, 127.1, 53.7, 37.3; HRMS (ESI) for $\text{C}_{16}\text{H}_{15}\text{NO}_3$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 270.1052, found: 270.1073.

Benzoylvaline (6b)



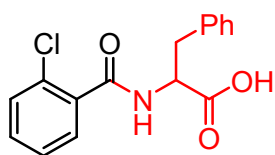
Yield 85%; white solid; m.p. 130 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.81 (d, 2H), 7.50 (t, 1H), 7.45 (t, 2H), 6.80 (d, 1H), 4.80 – (m, 1H), 2.36 (t, 1H), 1.05 (d, 6H); ^{13}C NMR (126 MHz, DMSO-d_6): δ 175.8, 168.1, 168.0, 133.8, 131.9, 128.7, 127.1, 57.6, 31.3, 19.0, 17.8; HRMS (ESI) for $\text{C}_{12}\text{H}_{15}\text{NO}_3$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 222.1051, found: 222.1040.

Benzoylalanine (6c)²⁰



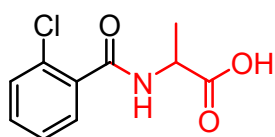
Yield 86%; white solid; m.p. 163 – 165 °C; ^1H NMR (500 MHz, DMSO-d_6): δ 8.67 (d, 1H), 7.90 (d, 2H), 7.56 (t, 1H), 7.48 (t, 2H), 5.14 (s, 1H), 4.45 – 4.42 (m, 1H), 1.41 (d, 3H); ^{13}C NMR (126 MHz, DMSO-d_6): δ 174.7, 166.7, 134.4, 131.8, 128.1, 127.9, 18.6, 17.3; HRMS (ESI) for $\text{C}_{10}\text{H}_{11}\text{NO}_3$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 194.0739, found: 194.0714.

2-chlorobenzoyl)phenylalanine (6d)



Yield 88%; white solid; m.p. 190 – 192 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.75 (d, 1H), 7.40 (d, 1H), 7.35 (t, 1H), 7.29 (t, 1H), 7.24 (dd, 2H), 7.18 (dd, 2H), 7.15 (t, 1H), 6.61 (d, 1H), 5.01 (t, 1H), 3.30 (dd, 2H); ^{13}C NMR (126 MHz, CDCl_3): δ 174.6, 167.8, 135.7, 134.0, 133.4, 132.0, 129.4, 128.7, 127.3, 59.8, 127.1, 53.7, 38.0; HRMS (ESI) for $\text{C}_{16}\text{H}_{14}\text{ClNO}_3$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 304.0662, found: 304.0683.

(2-chlorobenzoyl)alanine (6e)



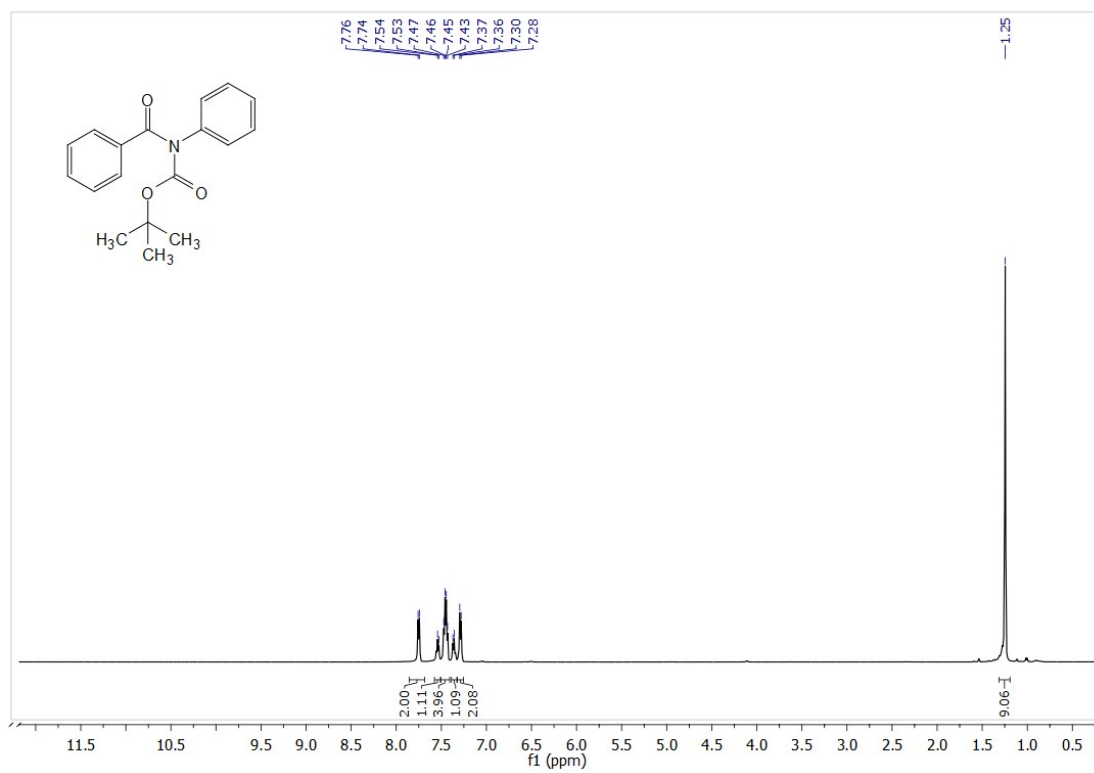
Yield 87%; white solid; m.p. 171–173°C; ^1H NMR (500 MHz, DMSO-d_6): δ 8.69 (d, 1H), 8.33 (d, 1H), 8.32 (t, 1H), 7.64 (t, 1H), 7.57 (d, 1H), 4.45 (m, 1H), 1.43 (d, 3H); ^{13}C NMR (126 MHz, DMSO-d_6): δ 174.8, 166.7, 134.4, 131.9, 129.4, 128.7, 127.9, 49.6, 19.7; HRMS (ESI) for $\text{C}_{10}\text{H}_{10}\text{ClNO}_3$ (m/z) $[\text{M} + \text{H}]^+$ calcd: 228.0349, found: 228.0356.

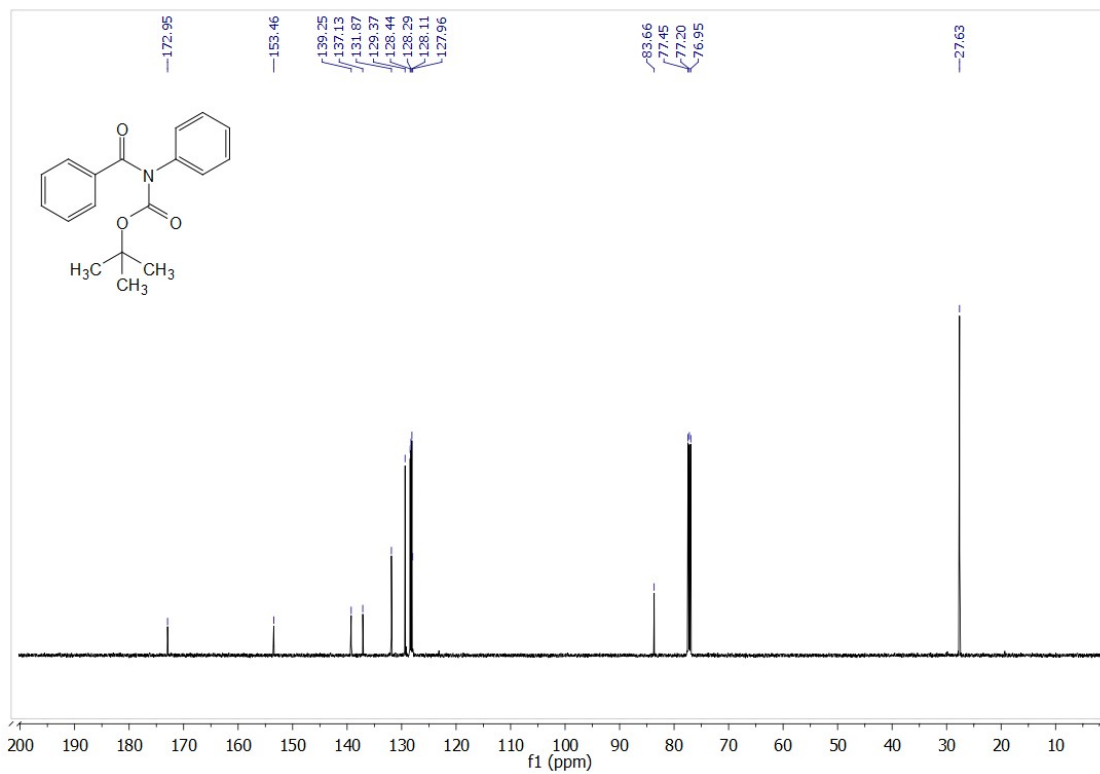
4. References

- 1 R. M. de Figueiredo, J. S. Suppo and J. M. Campagne, *Chem. Rev.*, 2016, **19**, 12029.
- 2 Y. Liu, S. Shi, M. Achtenhagen, R. Liu and M. Szostak, *Org. Lett.*, 2017, **19**, 1614.
- 3 X. Li and G. Zou, *Chem. Commun.*, 2015, **51**, 5089.
- 4 G. Meng, P. Lei and M. Szostak, *Org. Lett.*, 2017, **19**, 2158.
- 5 M. M. Rahman, G. Li and M. Szostak, *J. Org. Chem.*, 2019, **84**, 12091.
- 6 A. Mishra, S. Chauhan, P. Verma, S. Singh and V. Srivastava, *Asian J. Org. Chem.*, 2019, **8**, 853.
- 7 M. Raju, S. Mäeorg, O. Tšubrik and U. Mäeorg, *Arkivoc*, 2009, **6**, 291.
- 8 L. Grehn, K. Gunnarsson, U. Ragnarsson, T. Anthonsen and R. Kivekäs, *Acta Chem. Scand. B*, 1986, **40**, 745.
- 9 T. Ohshima, T. Iwasaki, Y. Maegawa, A. Yoshiyama and K. Mashima, *Synfacts*, 2008, **6**, 0627.
- 10 E. Racine, F. Monnier, J. P. Vors and M. Taillefer, *Org. Lett.*, 2011, **13**, 2818.
- 11 J. Bai, S. Li, R. Zhu, Y. Li and W. Li., *J. Org. Chem.* 2023, **6**, 3714.
- 12 I. A. P. S. Rajan and S. Rajendran, *New J. Chem.*, 2023, **47**, 10480.
- 13 A. Mishra, S. Singh and V. Srivastava, *Asian J. Org. Chem.*, 2018, **7**, 1600.
- 14 S. Yang, H. Yan, X. Ren, X. Shi, J. Li, Y. Wang and G. Huang, *Tetrahedron*, 2013, **69**, 6431.
- 15 C. A. Faler and M. M. Joullié, *Tetrahedron Lett.*, 2006, **47**, 7229.
- 16 F. Nasiri, J. Mokhtari, S. Taheri and Z. Mirjafary, *Tetrahedron Lett.*, 2023, **118**, 154392.
- 17 P. Sureshbabu, S. Azeez, P. Chaudhary and J. Kandasamy, *Org. Biomol. Chem.*, 2019, **17**, 845.
- 18 D. Joseph, M. Seong Park and S. Lee, *Org. Biomol. Chem.*, 2021, **19**, 6227.
- 19 X. Wan, Z. Ma, B. Li, K. Zhang, S. Cao, S. Zhang, Z. Shi, *J. Am. Chem. Soc.*, 2006, **23**, 7416.
- 20 I. A. P. S. Rajan and S. Rajendran, *Org. Biomol. Chem.*, 2023, **21**, 4760.

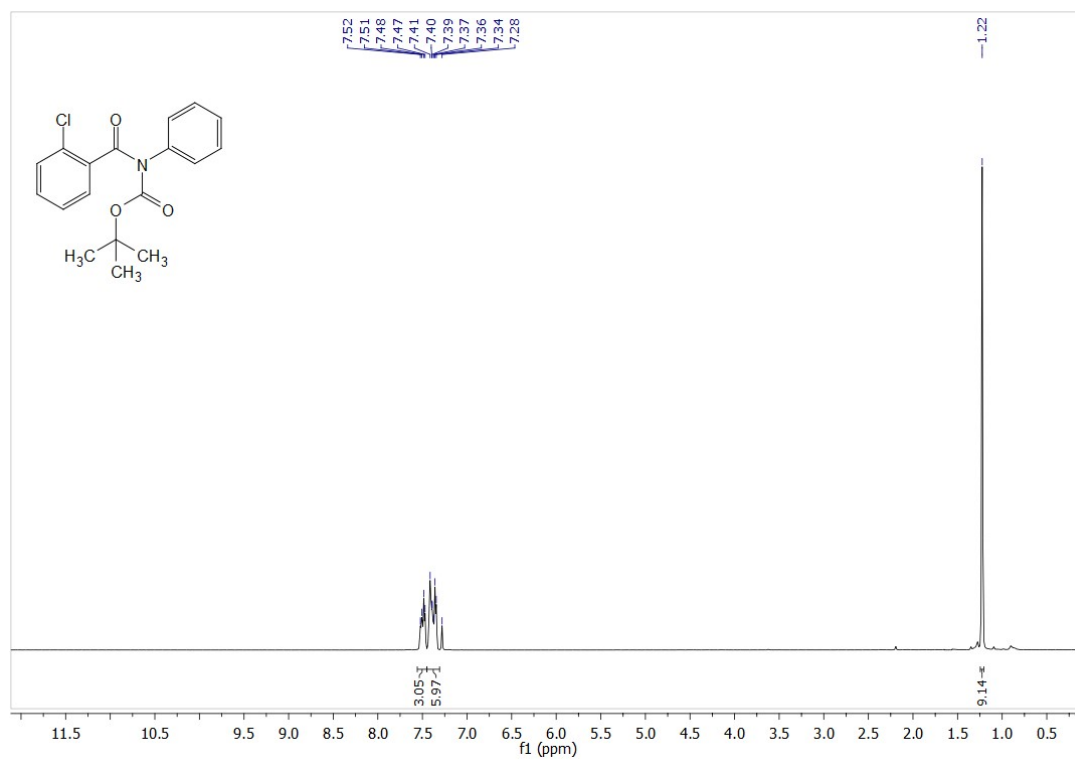
¹H and ¹³C NMR Spectra of starting products

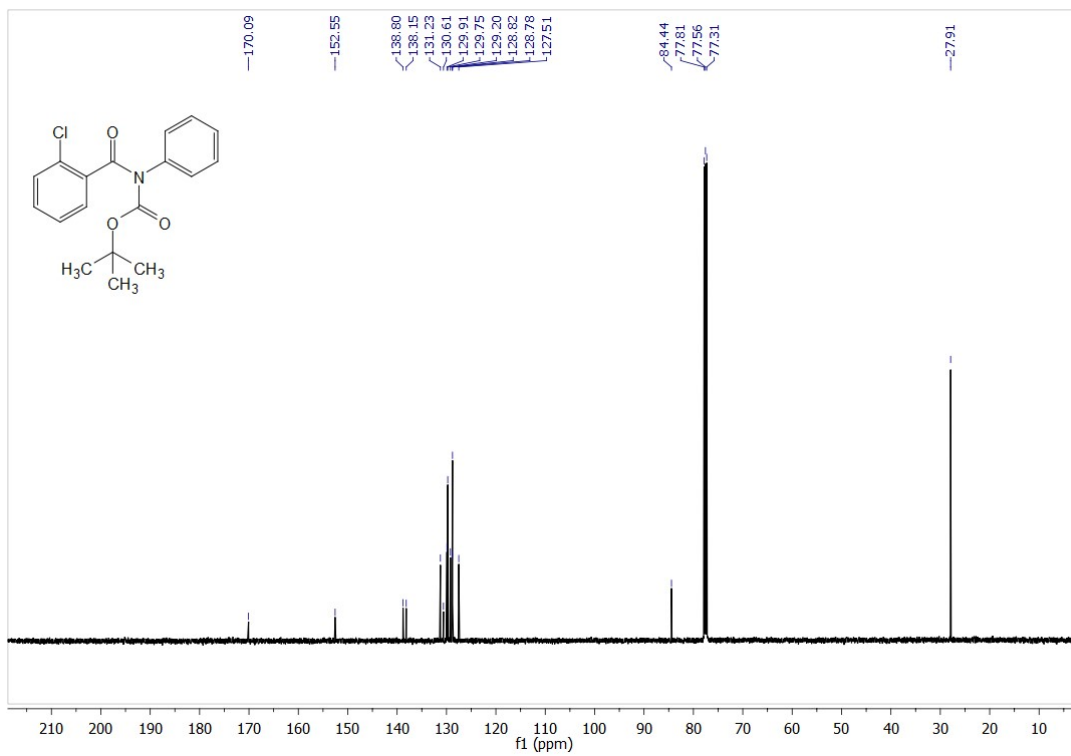
¹H and ¹³C NMR Spectra of *tert*-butyl benzoyl(phenyl)carbamate (1a)



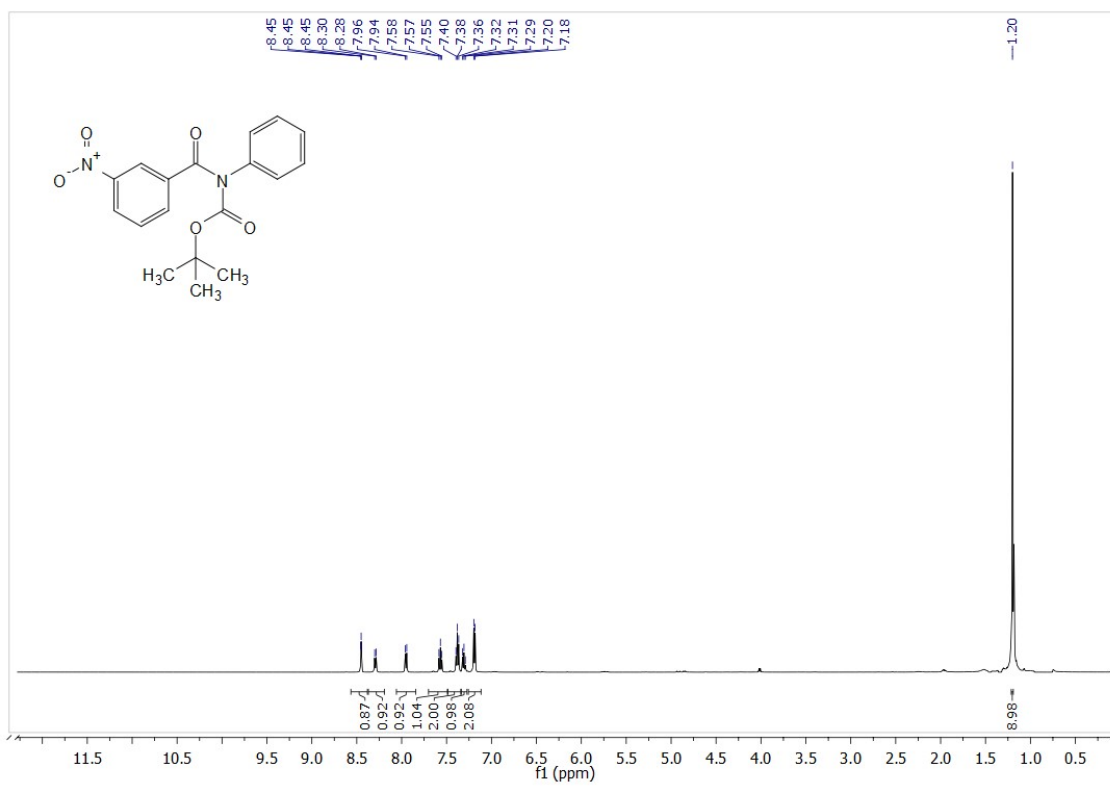


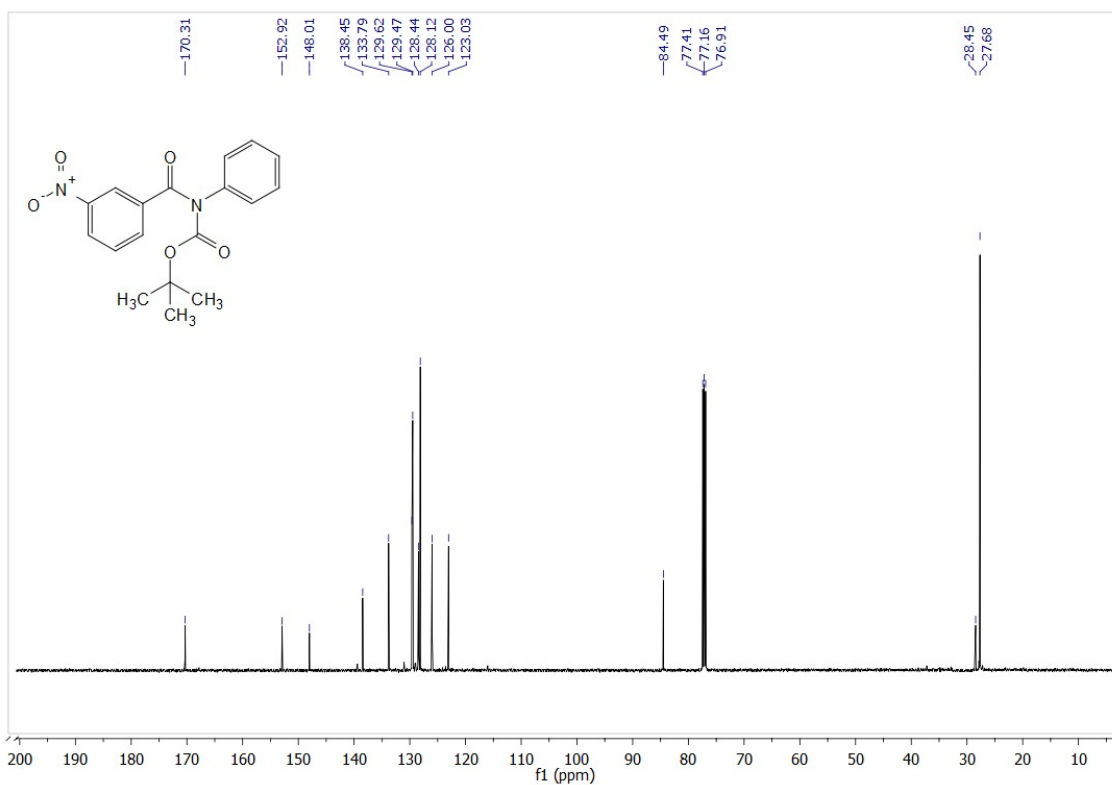
¹H and ¹³C NMR Spectra of *tert*-butyl (2-chlorobenzoyl)(phenyl)carbamate (1b)



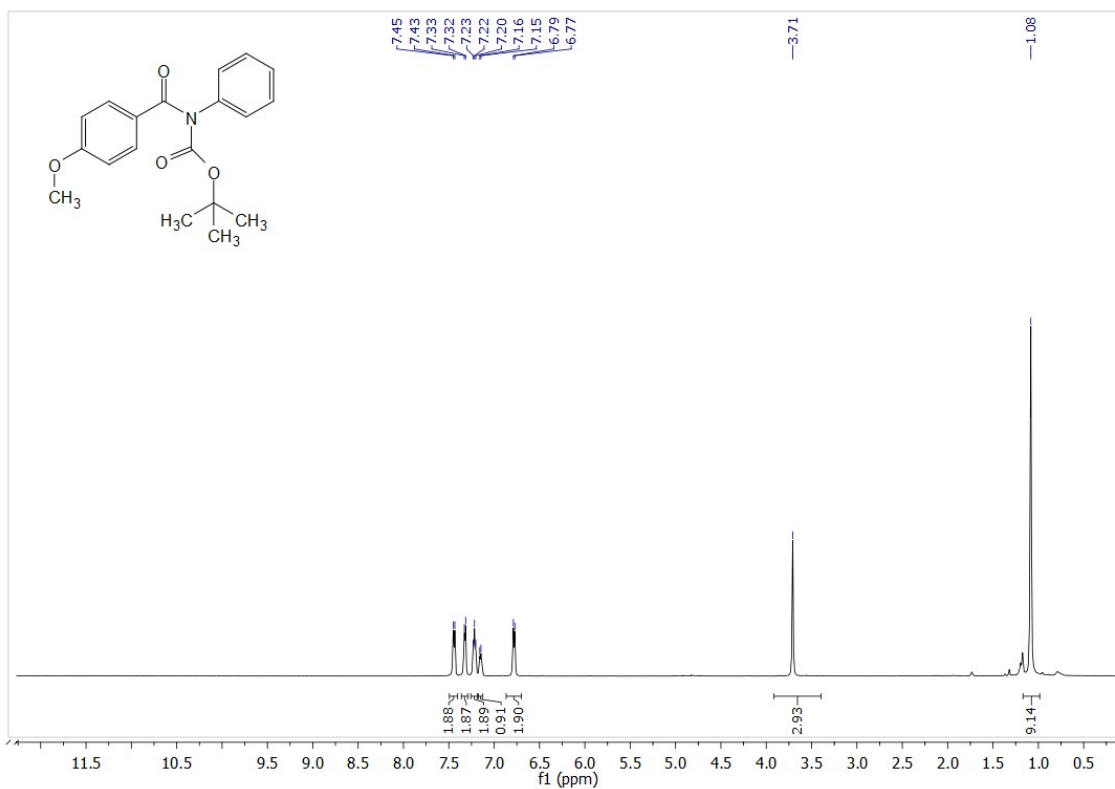


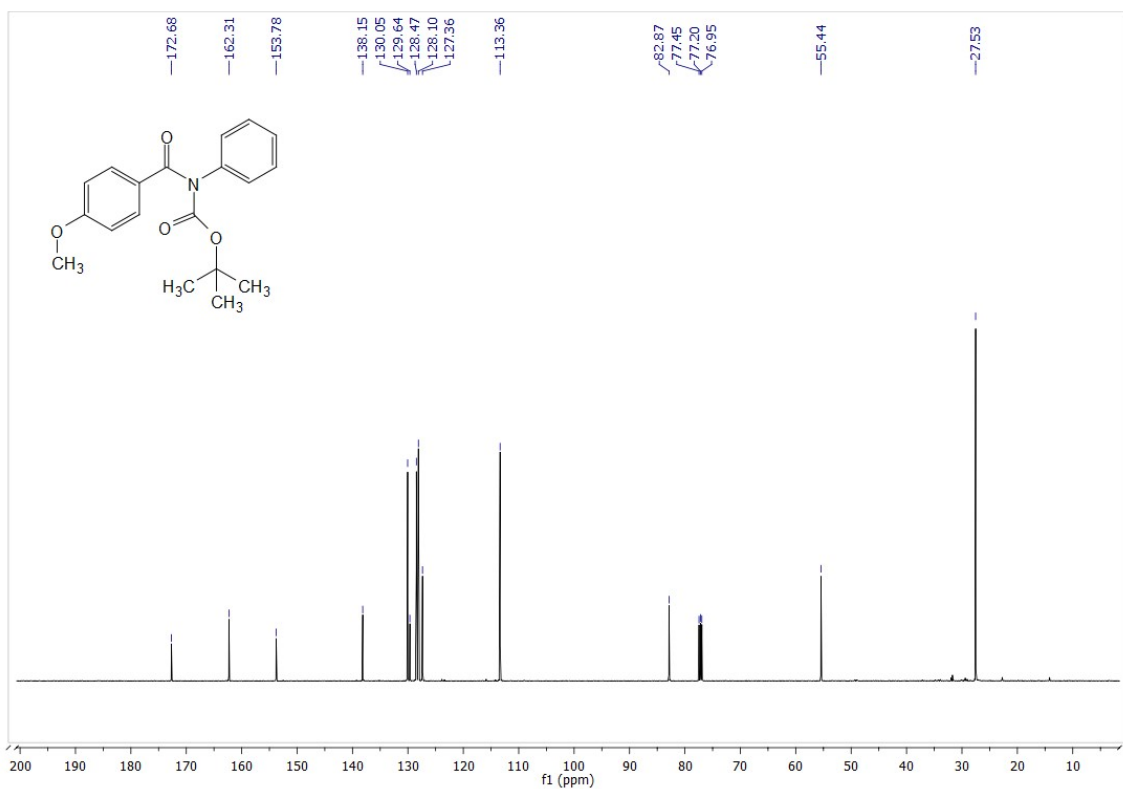
¹H and ¹³C NMR Spectra of *tert*-butyl (3-nitrobenzoyl)(phenyl)carbamate (1c)



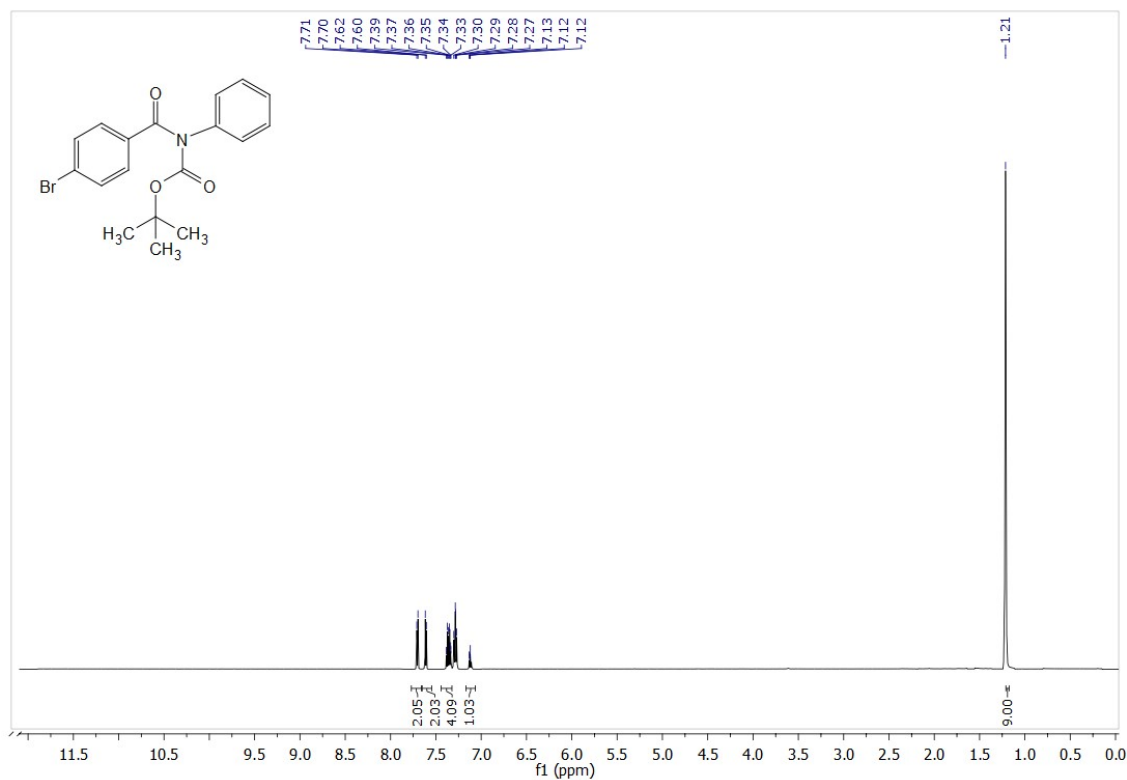


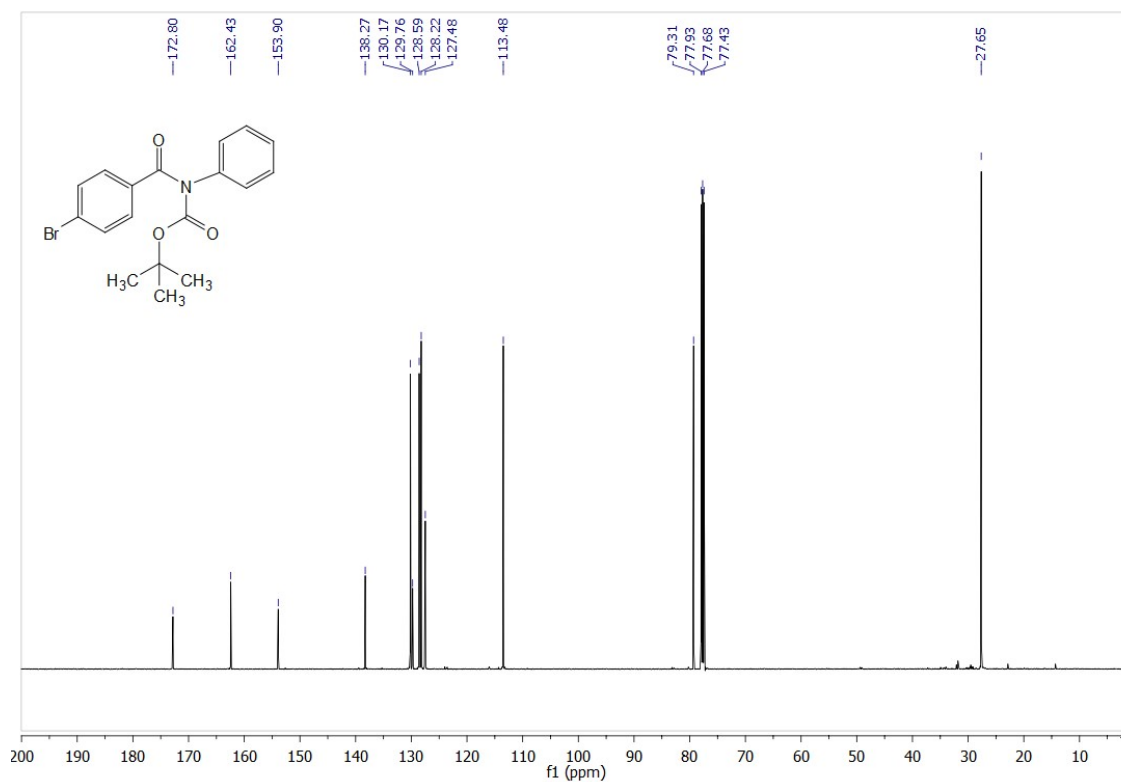
¹H and ¹³C NMR Spectra of *tert*-butyl (4-methoxybenzoyl)(phenyl)carbamate (1d)



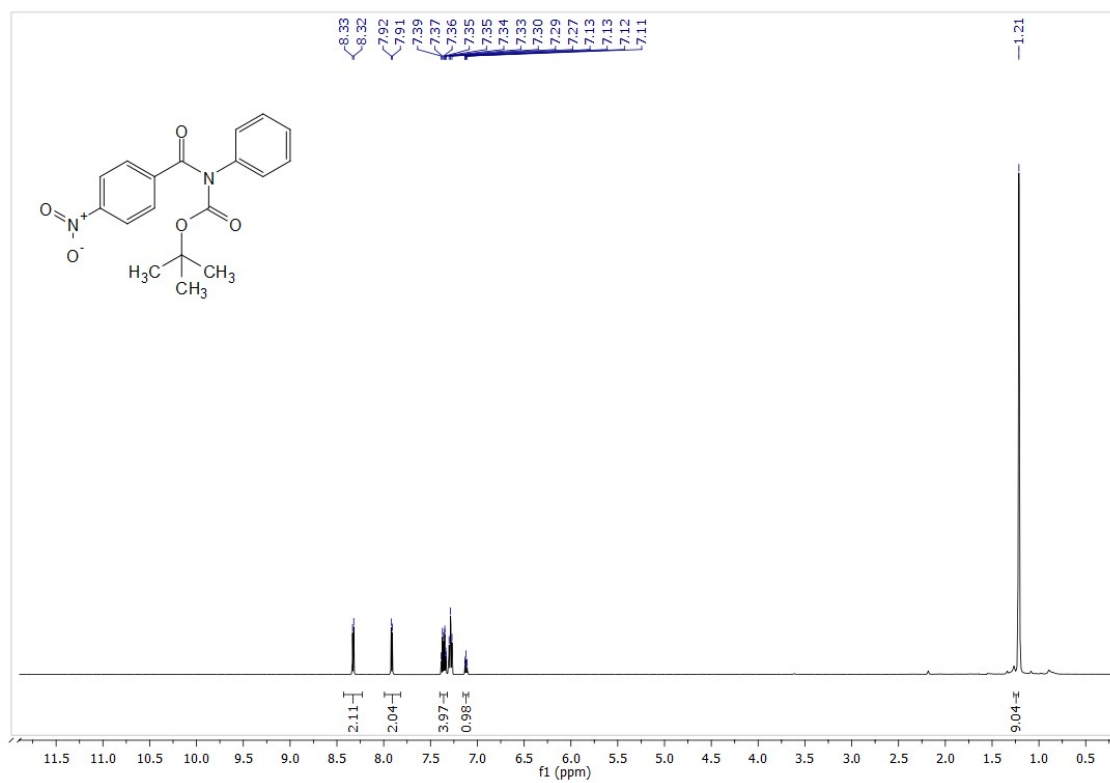


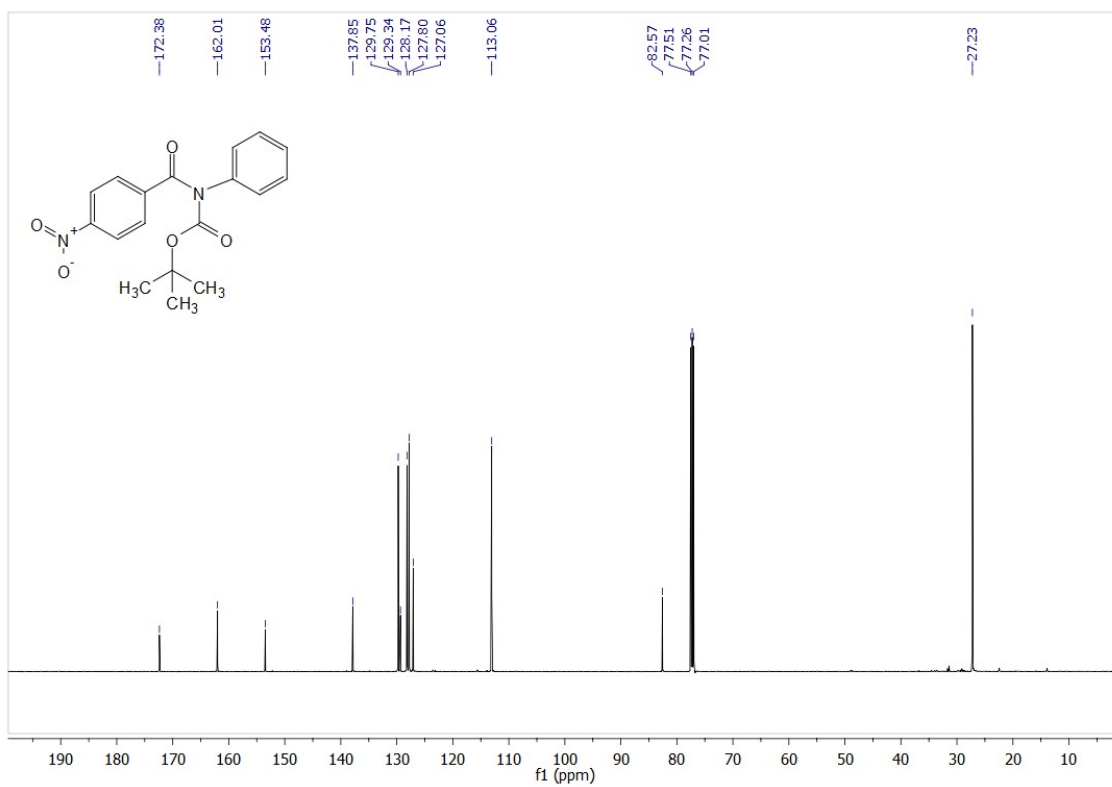
¹H and ¹³C NMR Spectra of *tert*-butyl (4-bromobenzoyl)(phenyl)carbamate (1e)



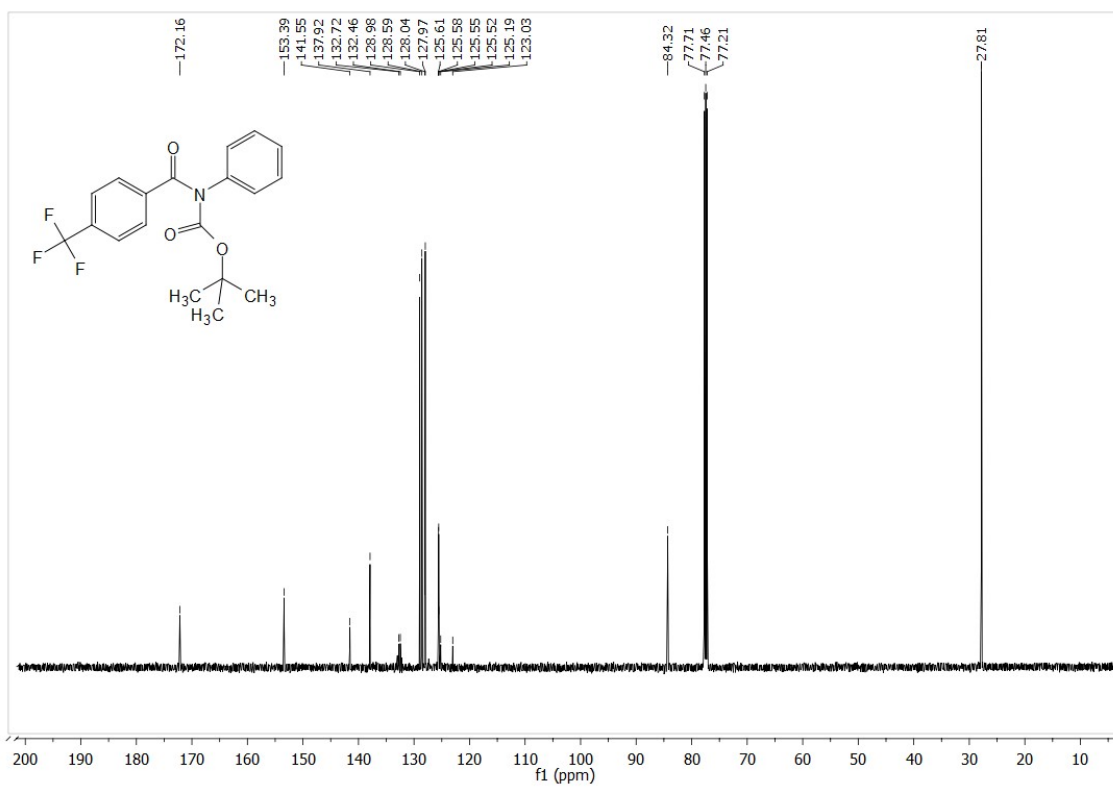
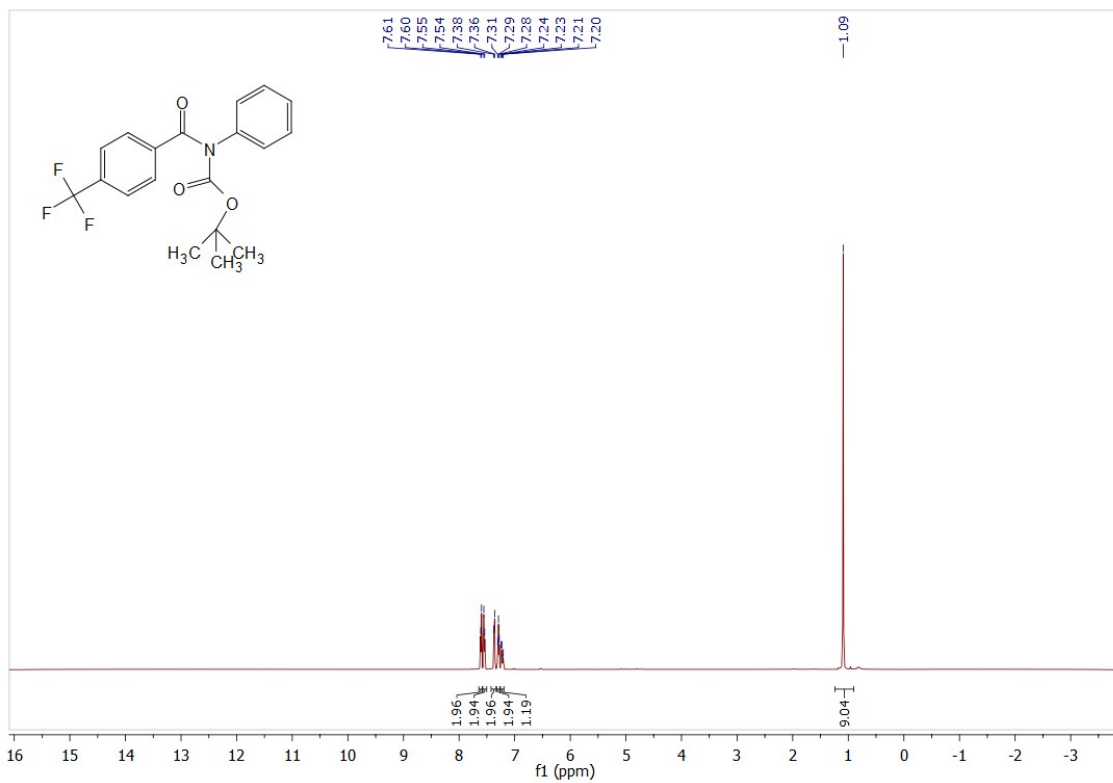


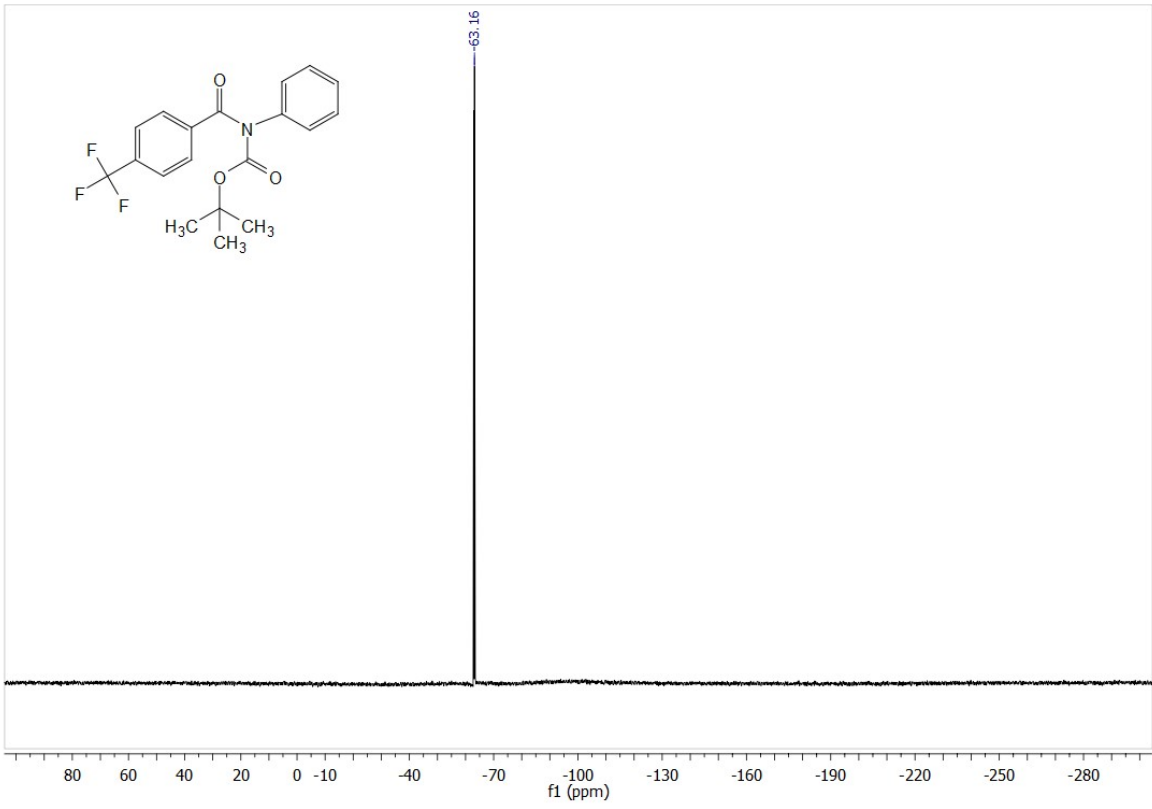
¹H and ¹³C NMR Spectra of *tert*-butyl (4-nitrobenzoyl)(phenyl)carbamate (1f)



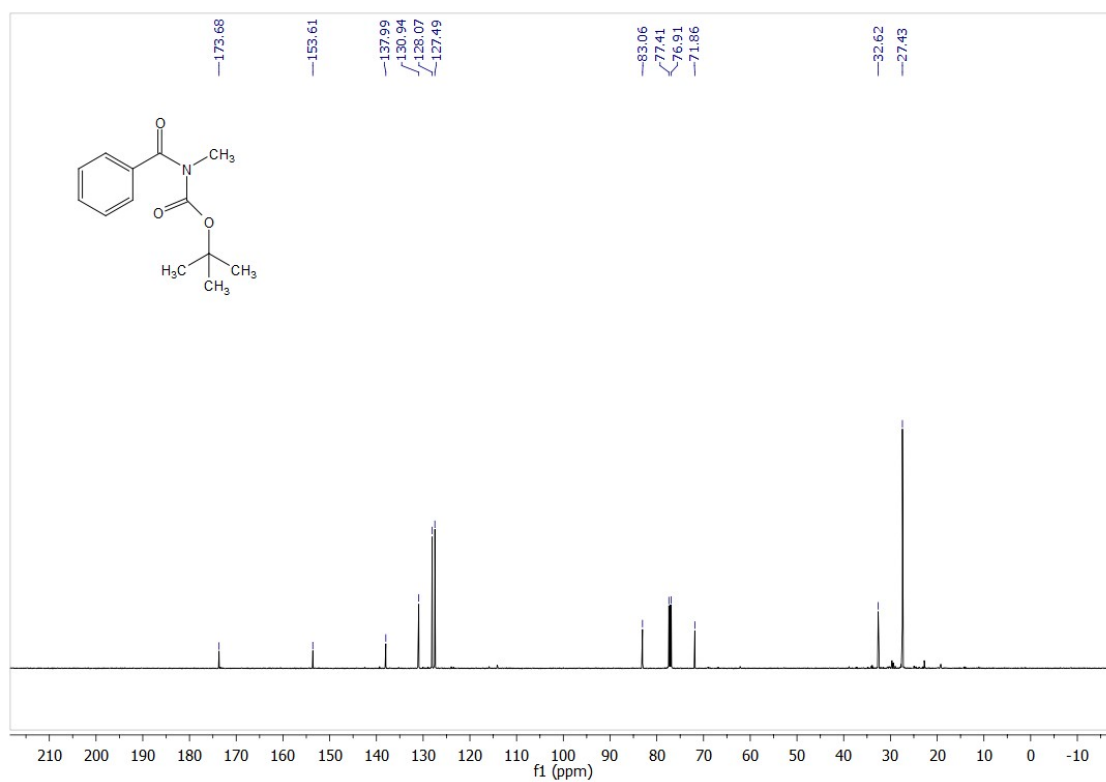
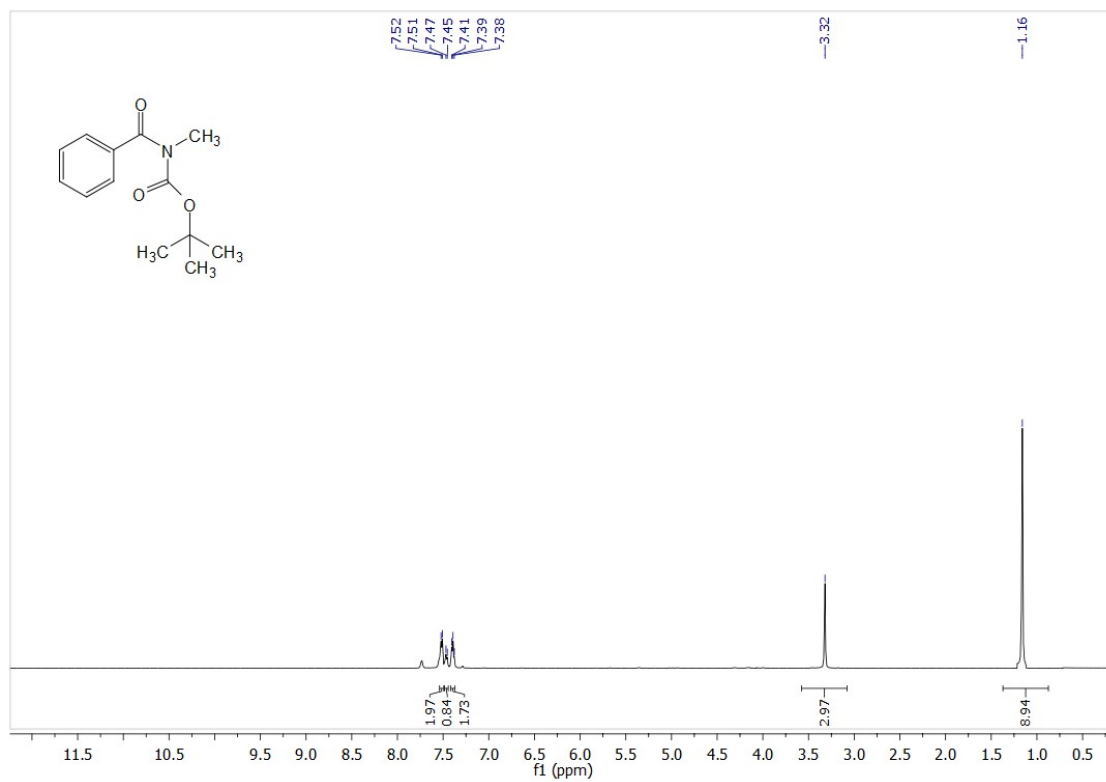


¹H, ¹³C and ¹⁹F NMR Spectra of *tert*-butyl (4-trifluoromethyl)(benzoyl)(phenyl)carbamate (1g)

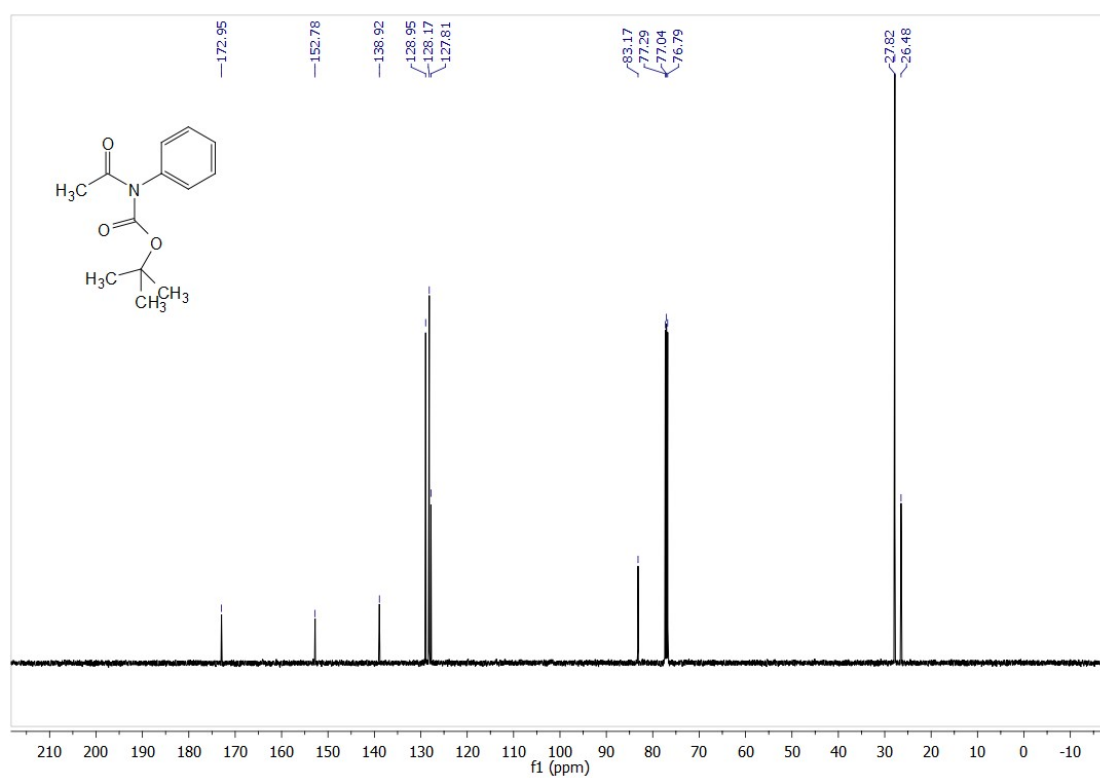
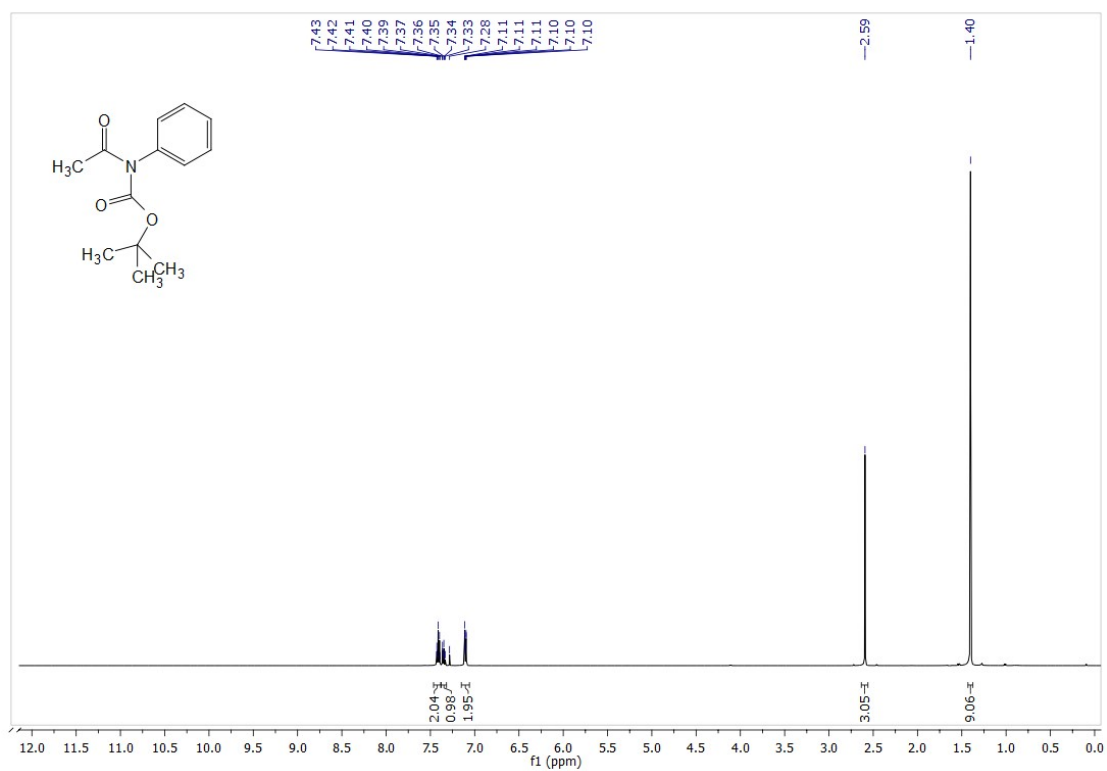




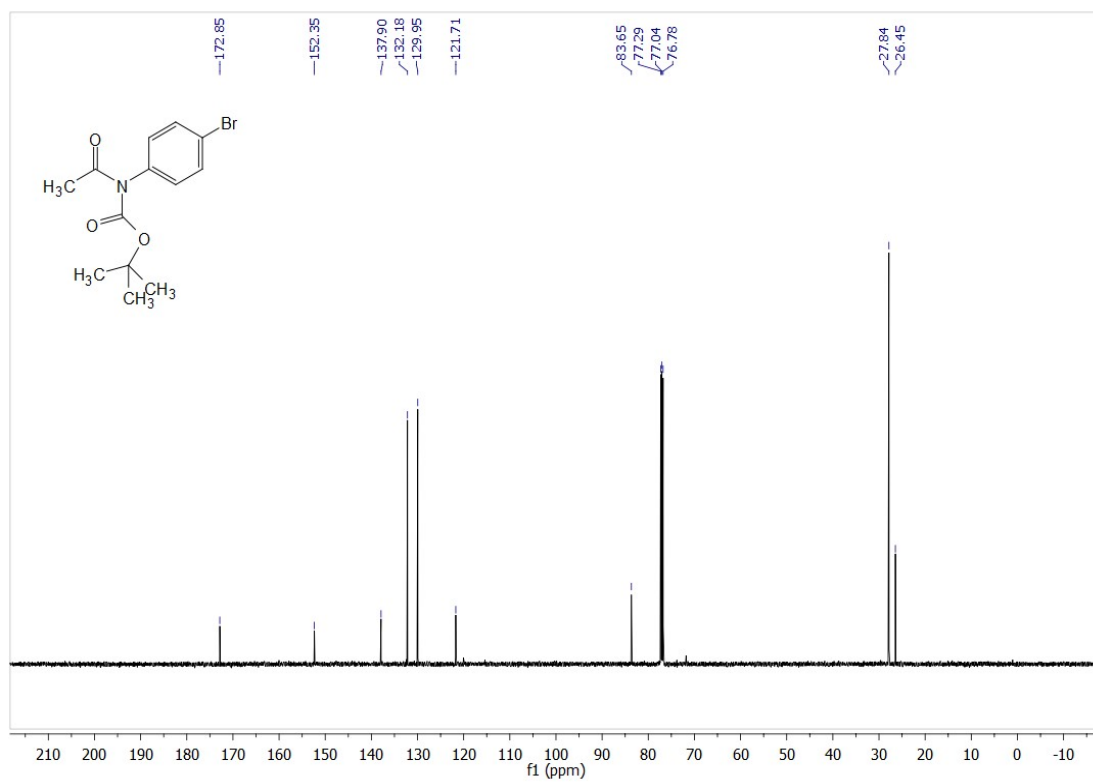
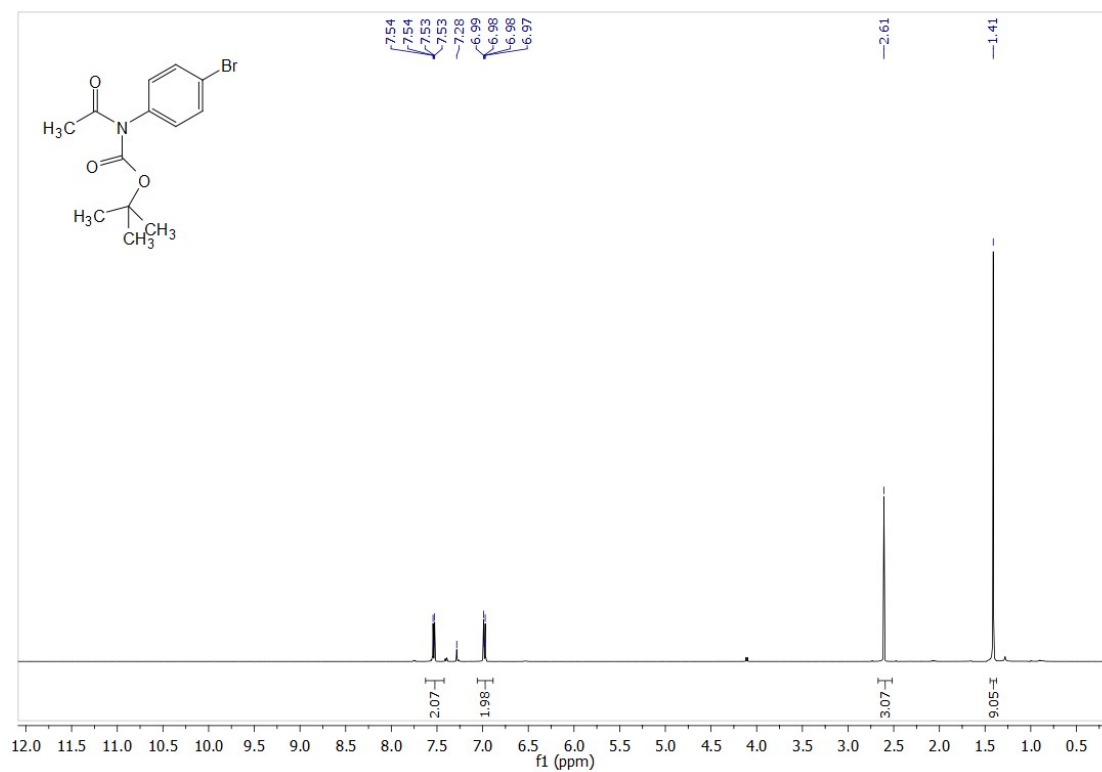
^1H and ^{13}C NMR Spectra of *tert*-butyl benzoyl(methyl)carbamate (1h)



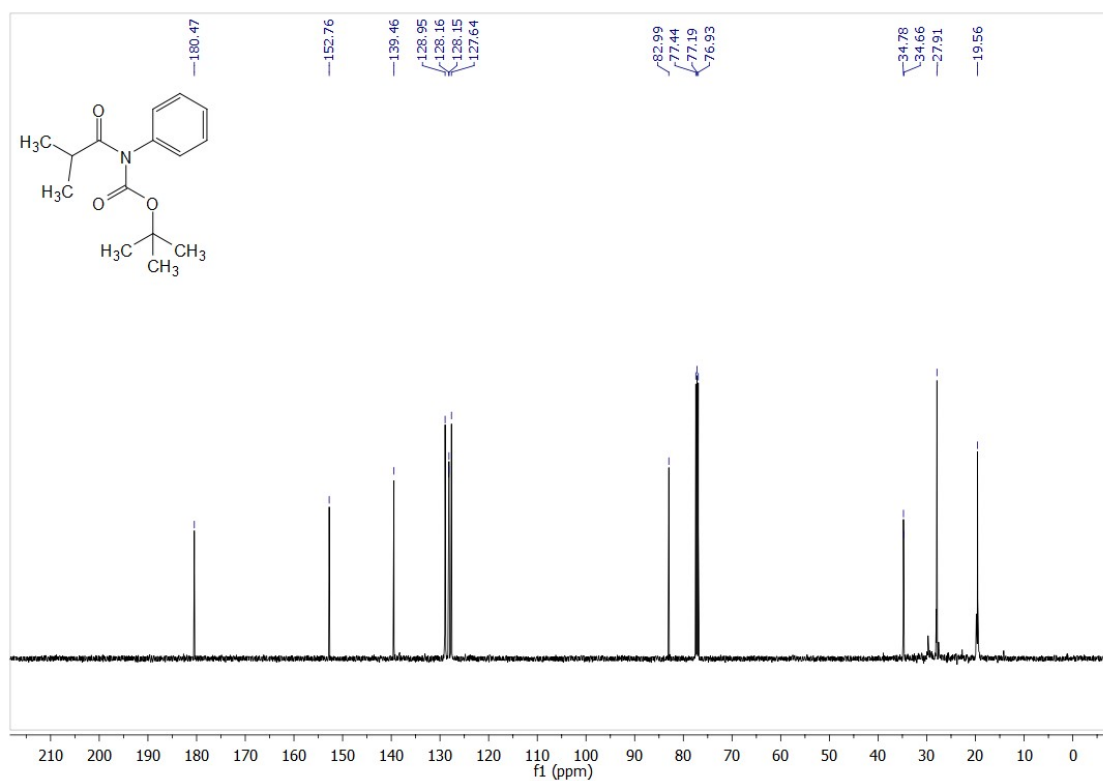
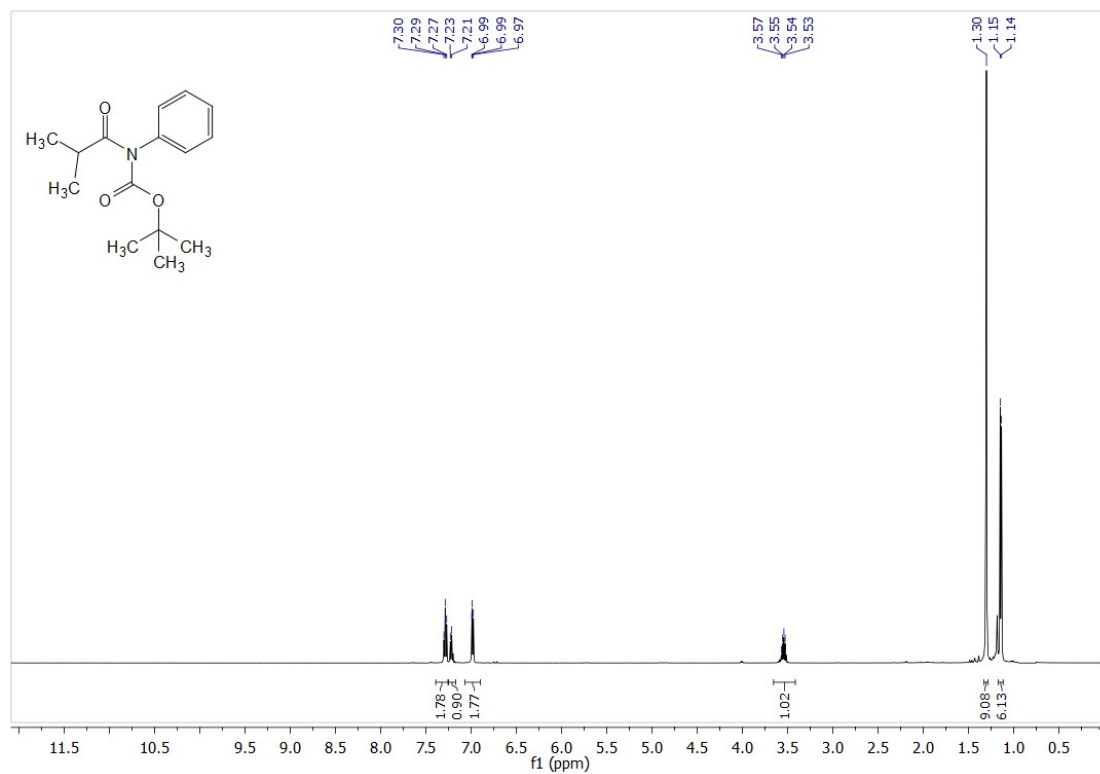
^1H and ^{13}C NMR Spectra of *tert*-butyl acetyl(phenyl)carbamate (1i)



¹H and ¹³C NMR Spectra of *tert*-butyl acetyl(4-bromophenyl)carbamate (1j)

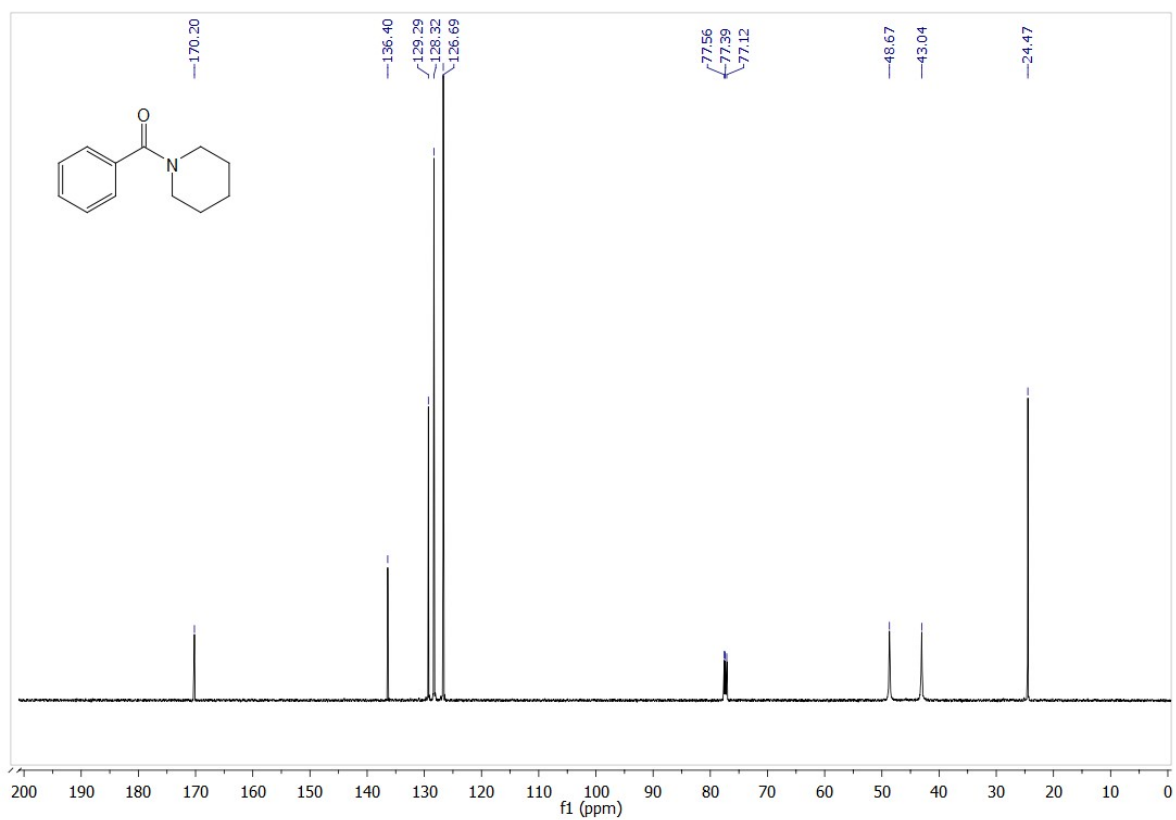
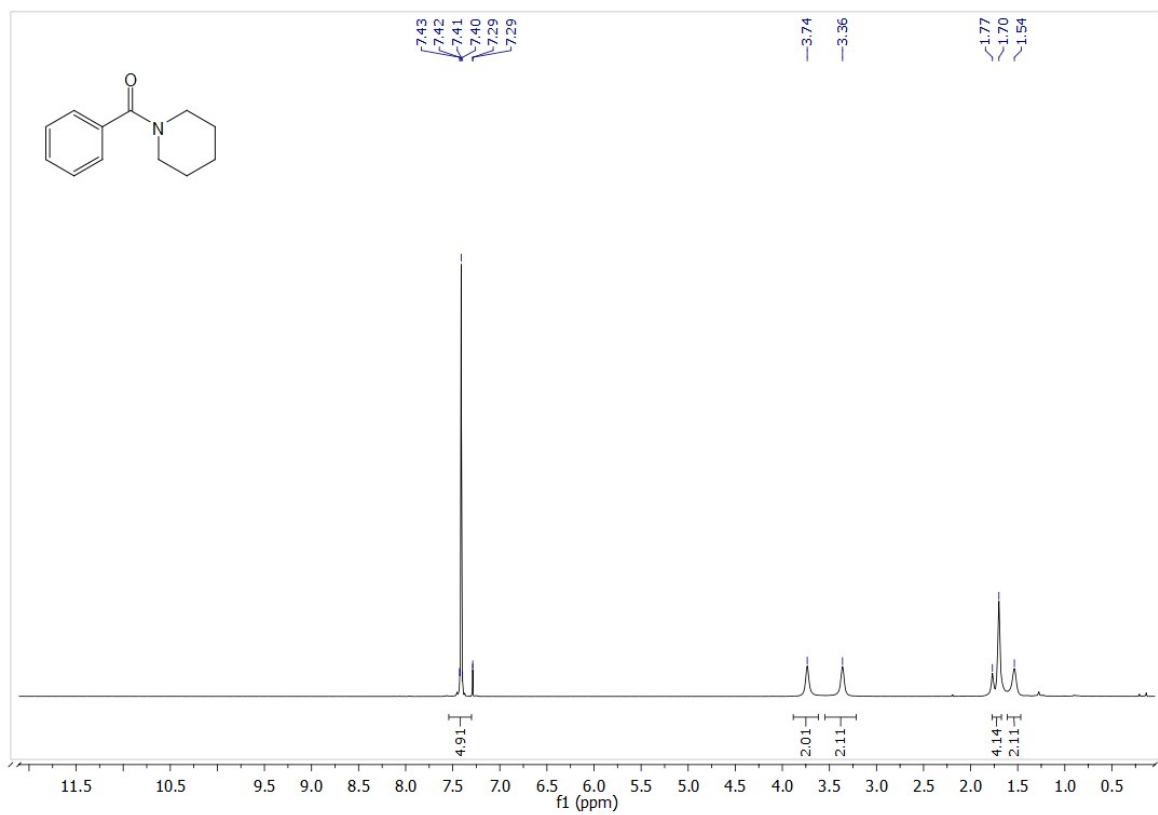


¹H and ¹³C NMR Spectra of *tert*-butyl isobutyryl(phenyl)carbamate (1k)



^1H and ^{13}C NMR Spectra of Transamidation Products

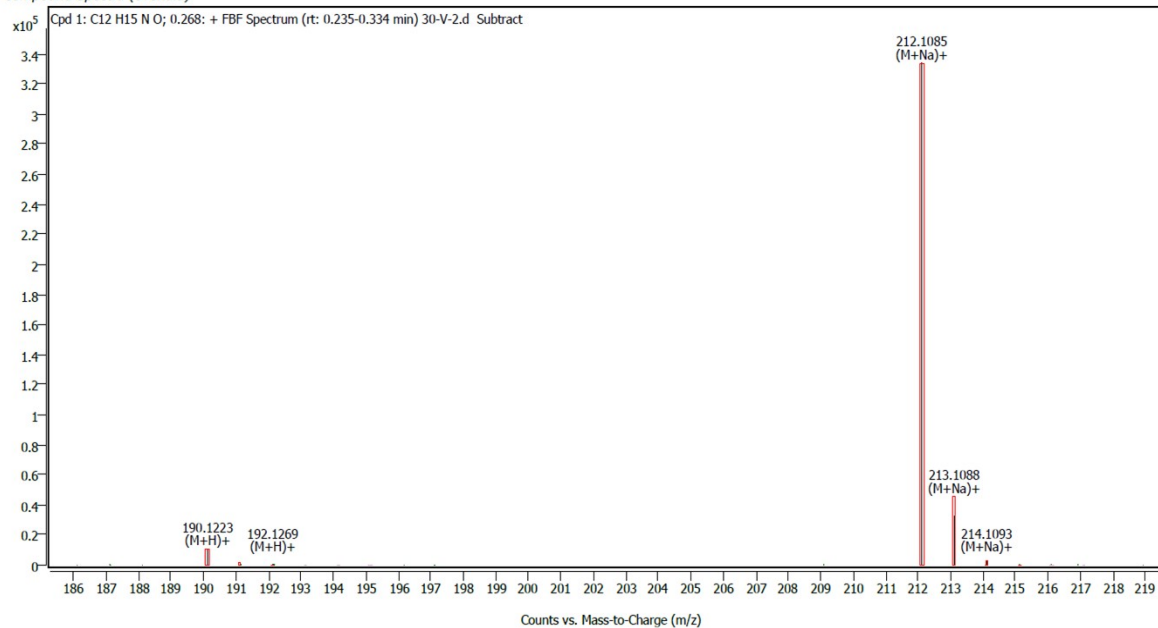
^1H and ^{13}C NMR Spectra of phenyl(piperidin-1-yl)methanone (3a)



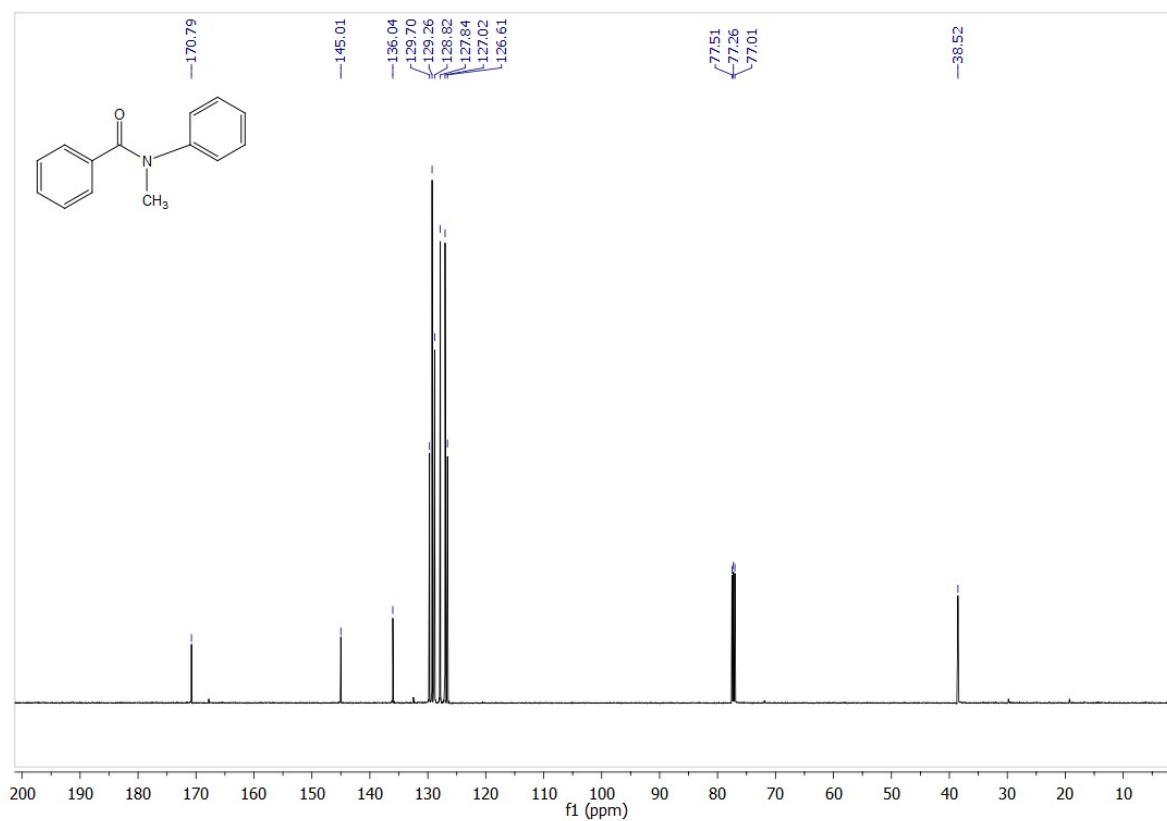
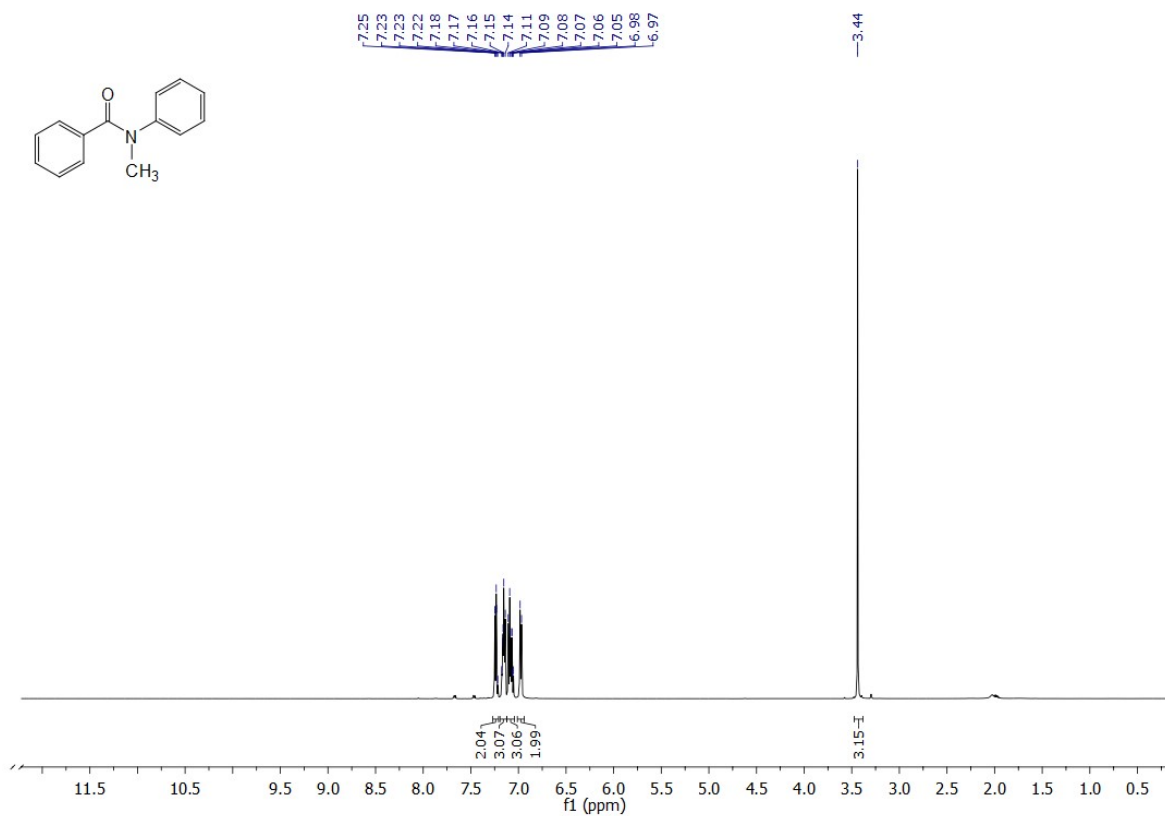
Mass Spectra of phenyl(piperidin-1-yl)methanone (3a)

Cpd. 1: C₁₂ H₁₅ N O

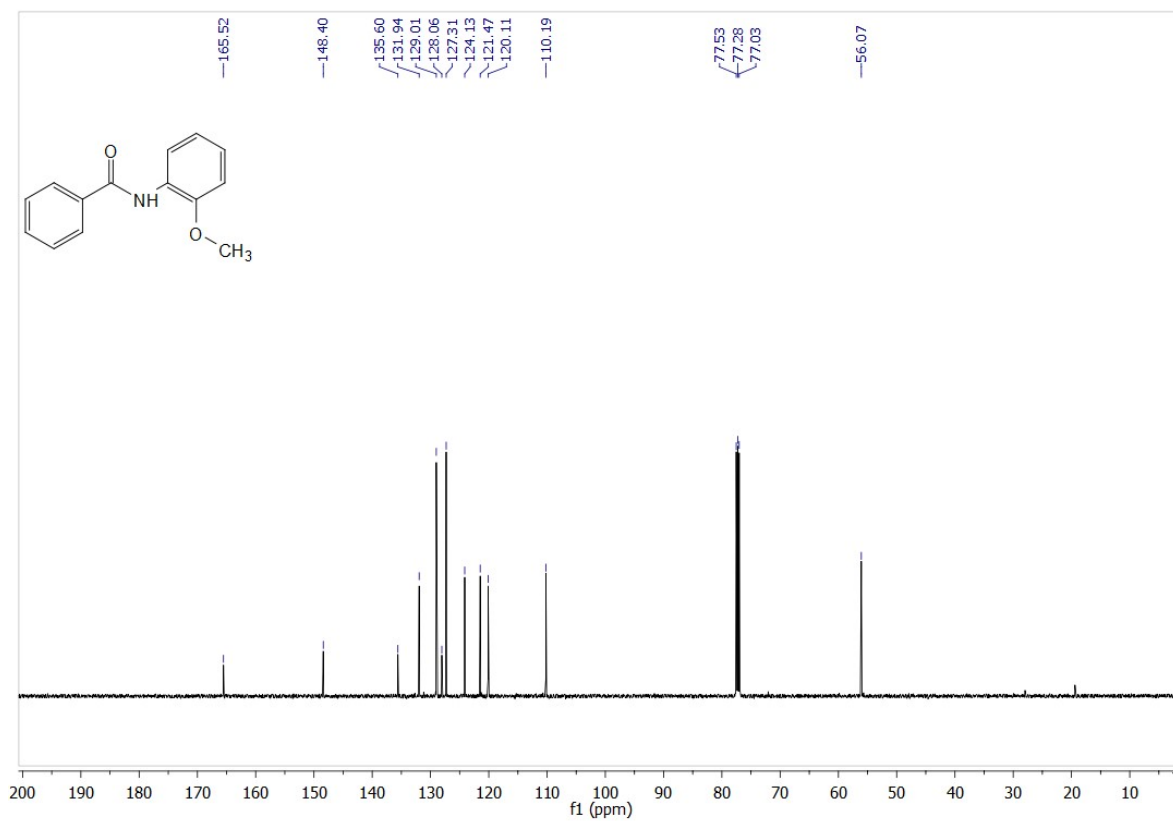
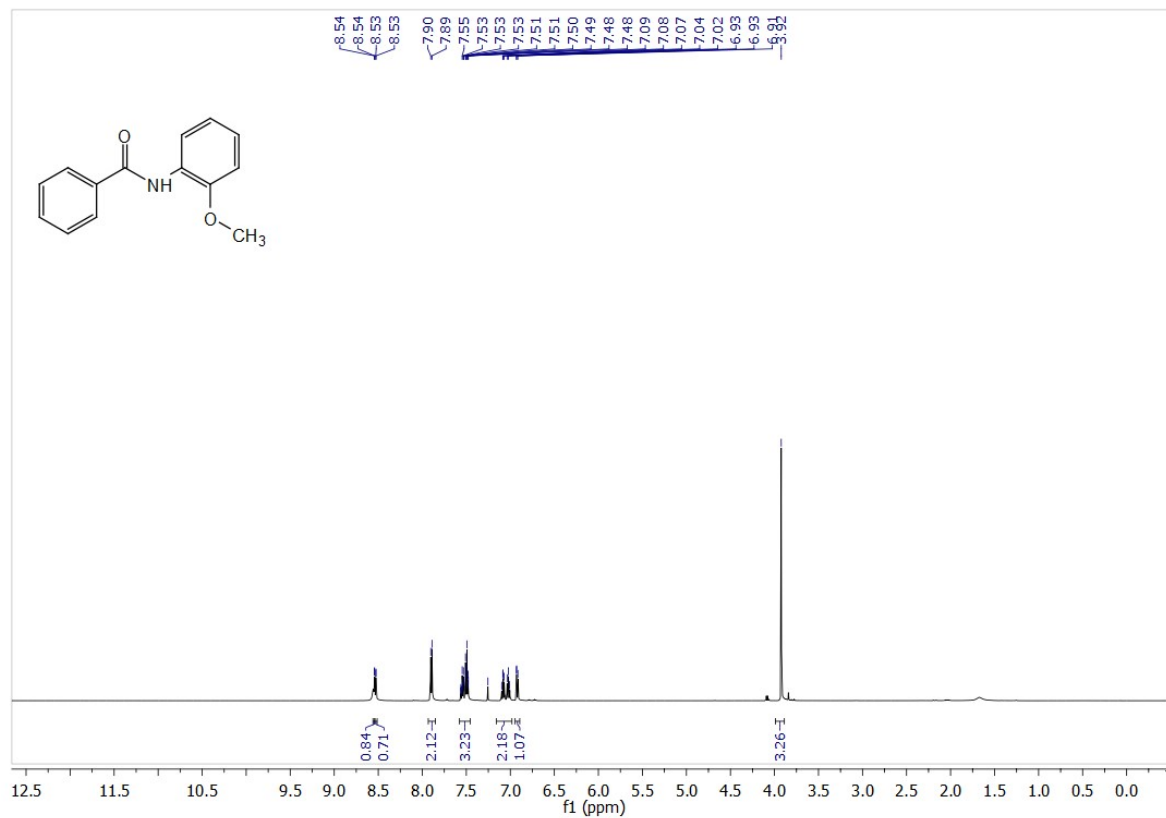
Compound Spectra (overlaid)



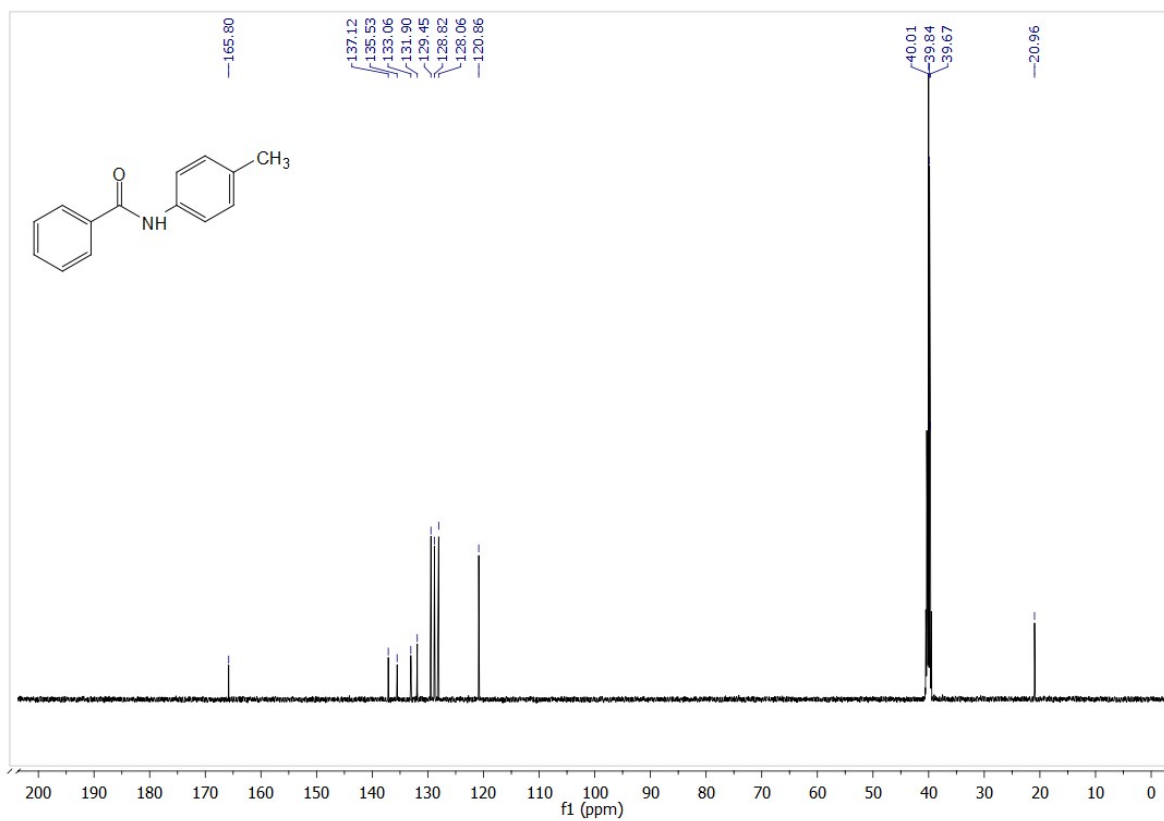
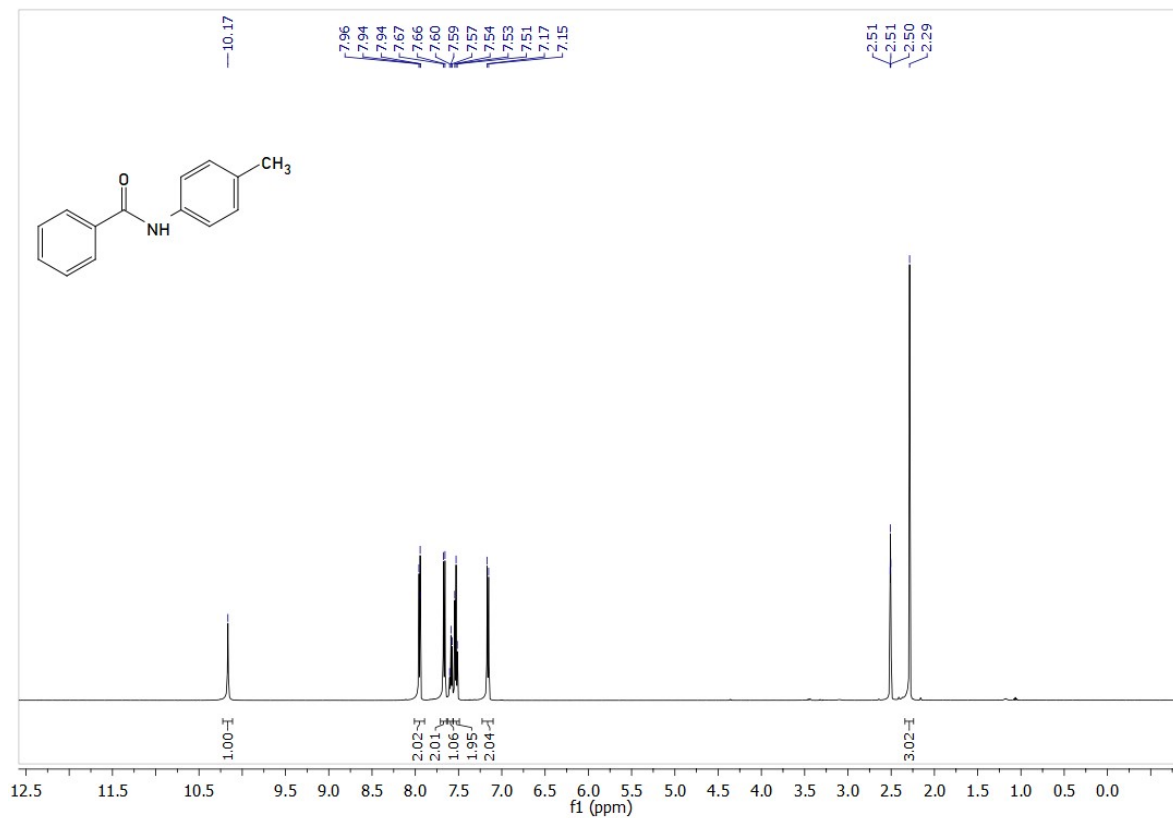
^1H and ^{13}C NMR Spectra of *N*-methyl-*N*-phenylbenzamide (3b)



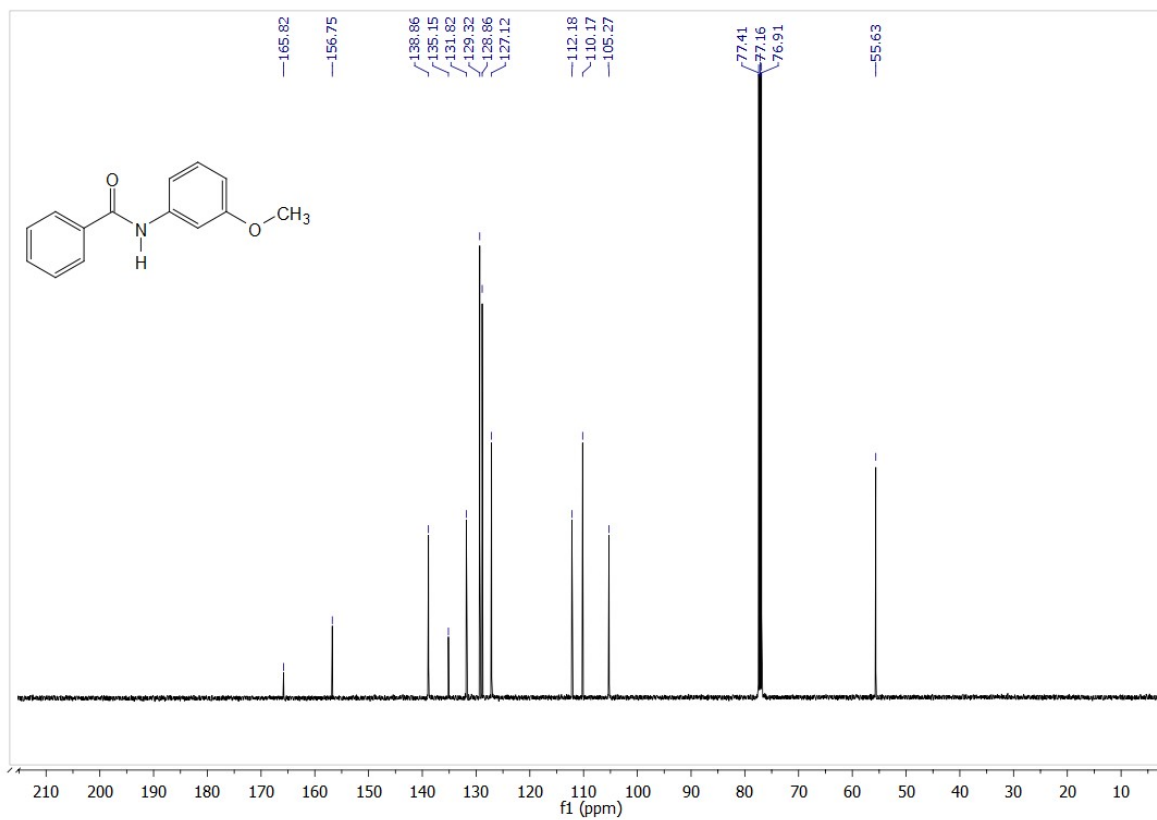
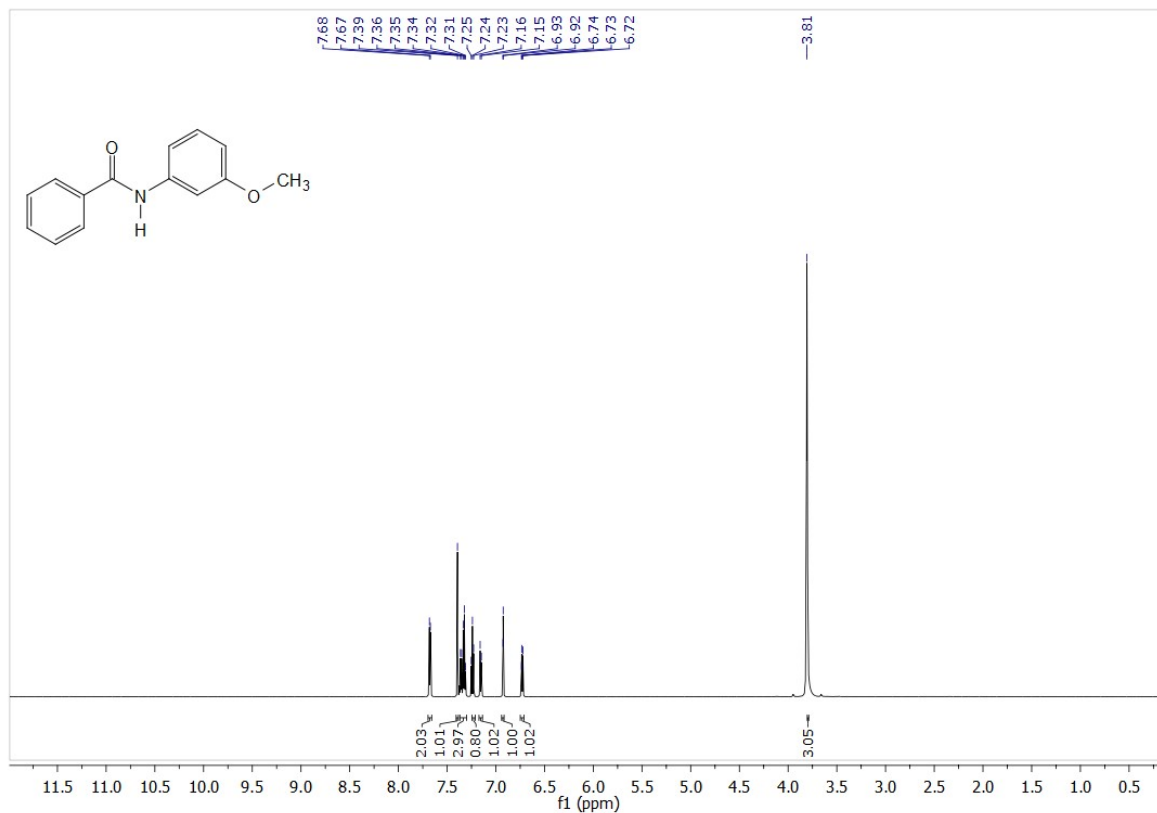
¹H and ¹³C NMR Spectra of *N*-(2-methoxyphenyl)benzamide (3c)



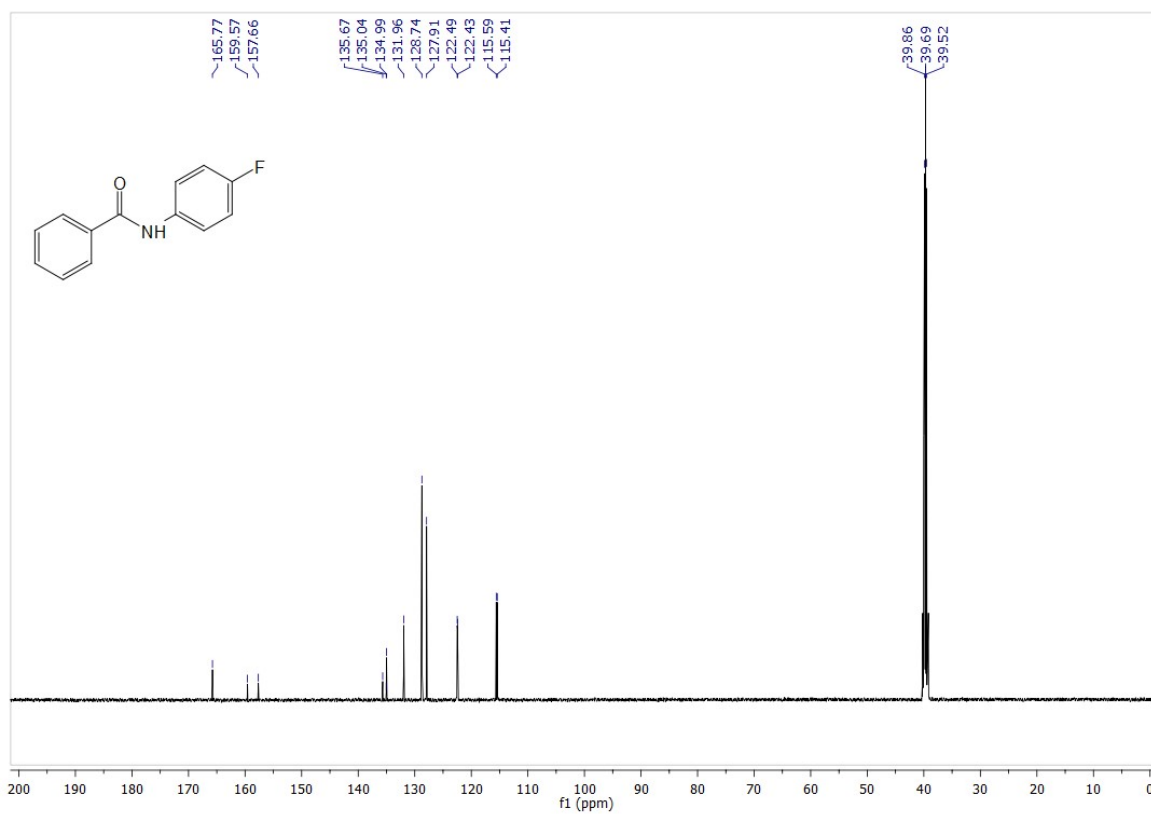
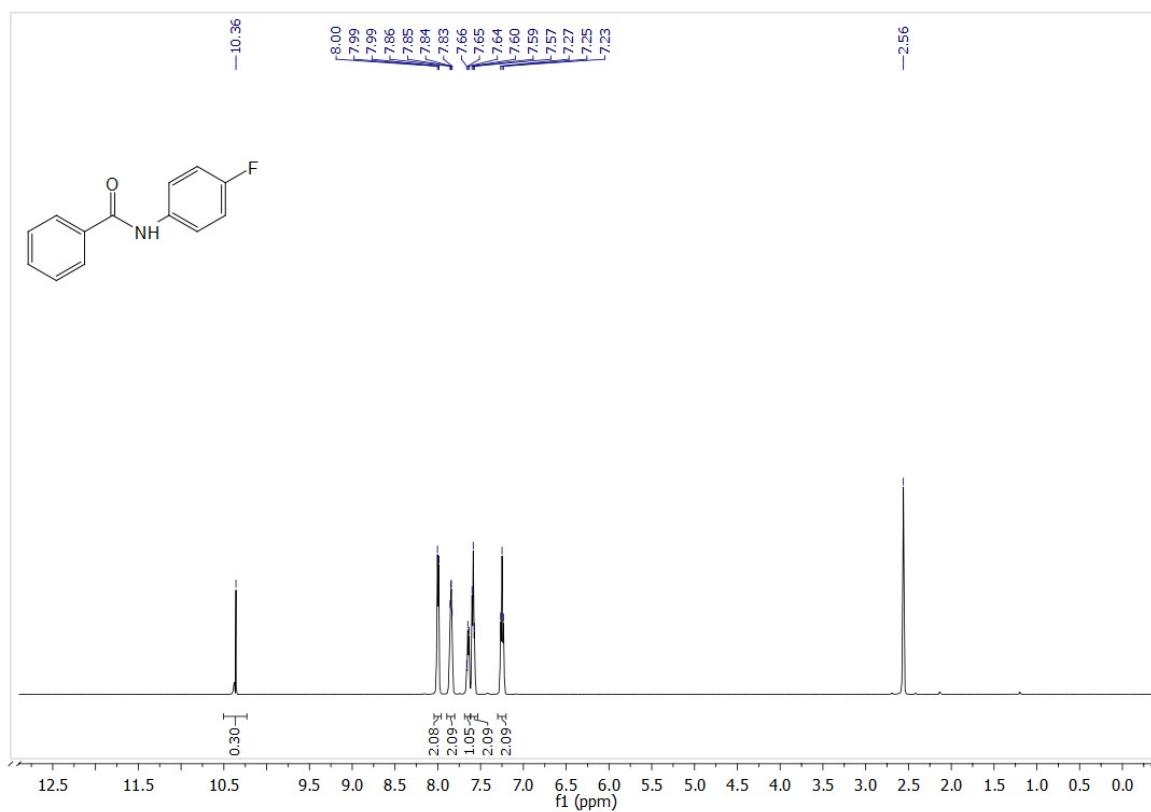
¹H and ¹³C NMR Spectra of *N*-(4-methylphenyl)benzamide (3d)

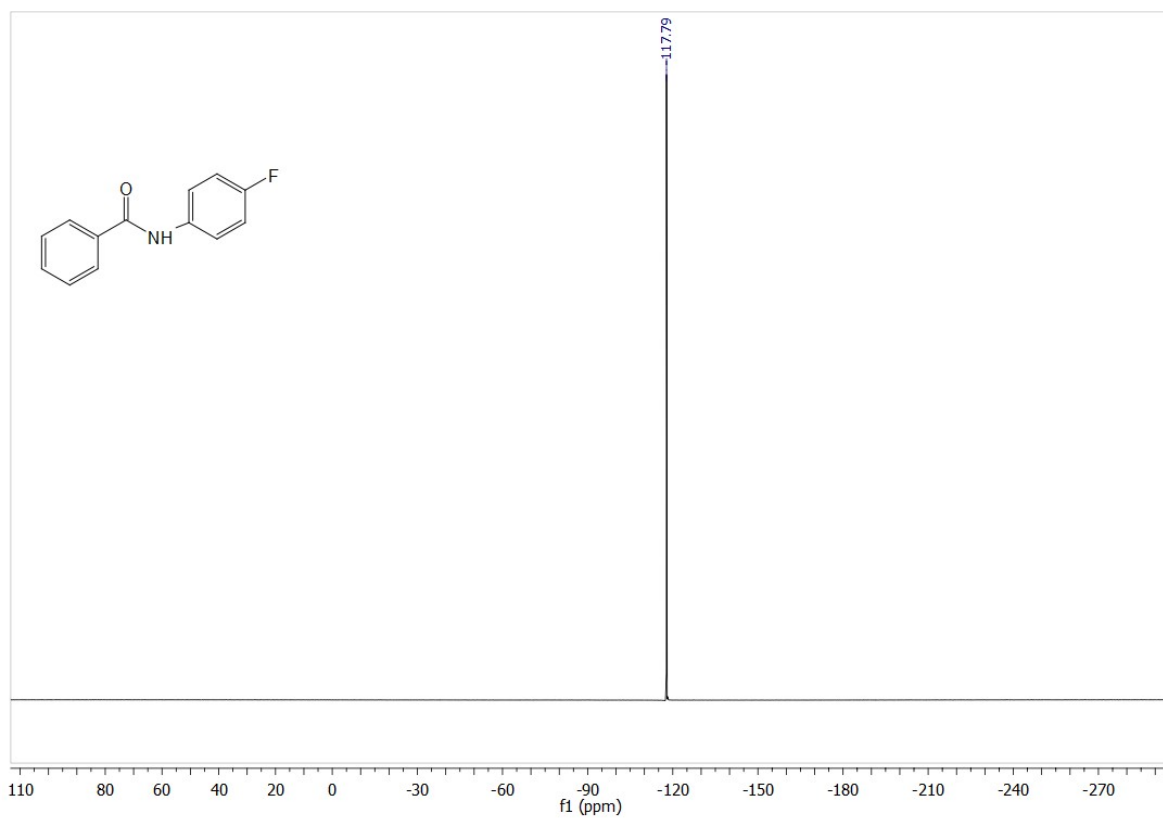


¹H and ¹³C NMR Spectra of *N*-(3-methoxyphenyl)benzamide (3e)

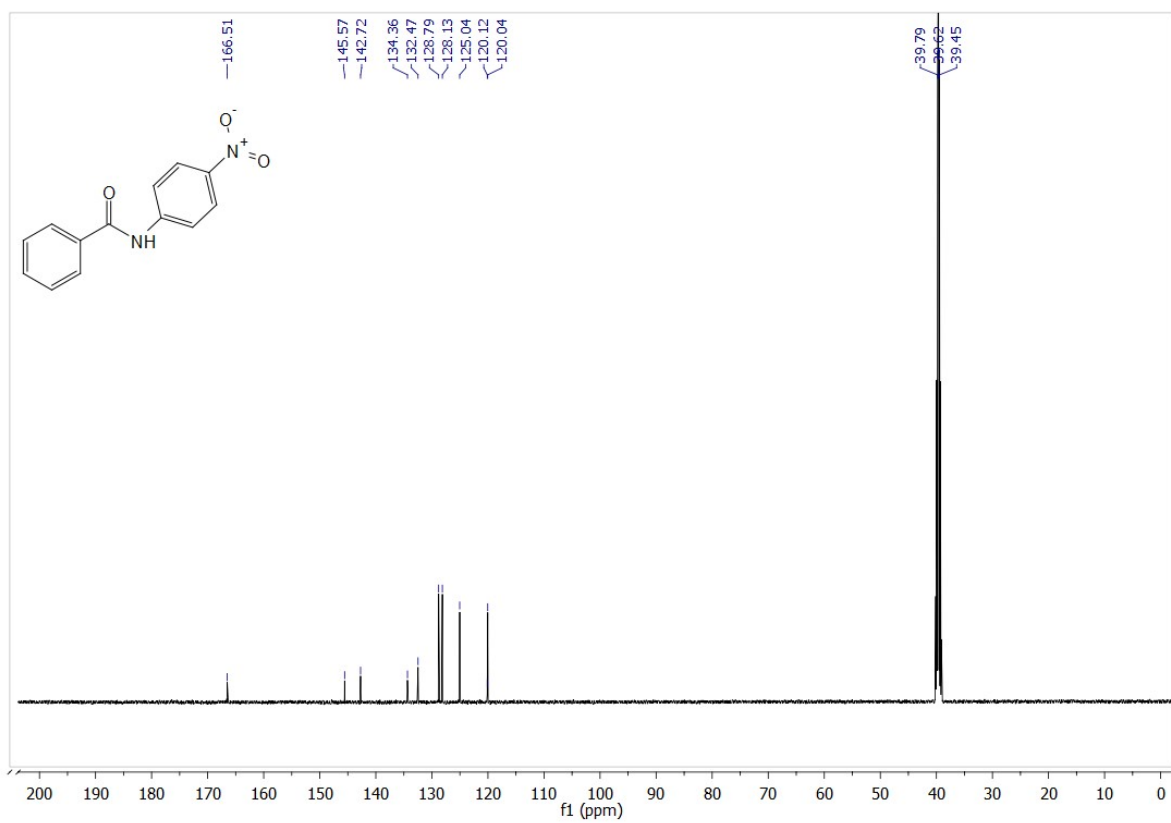
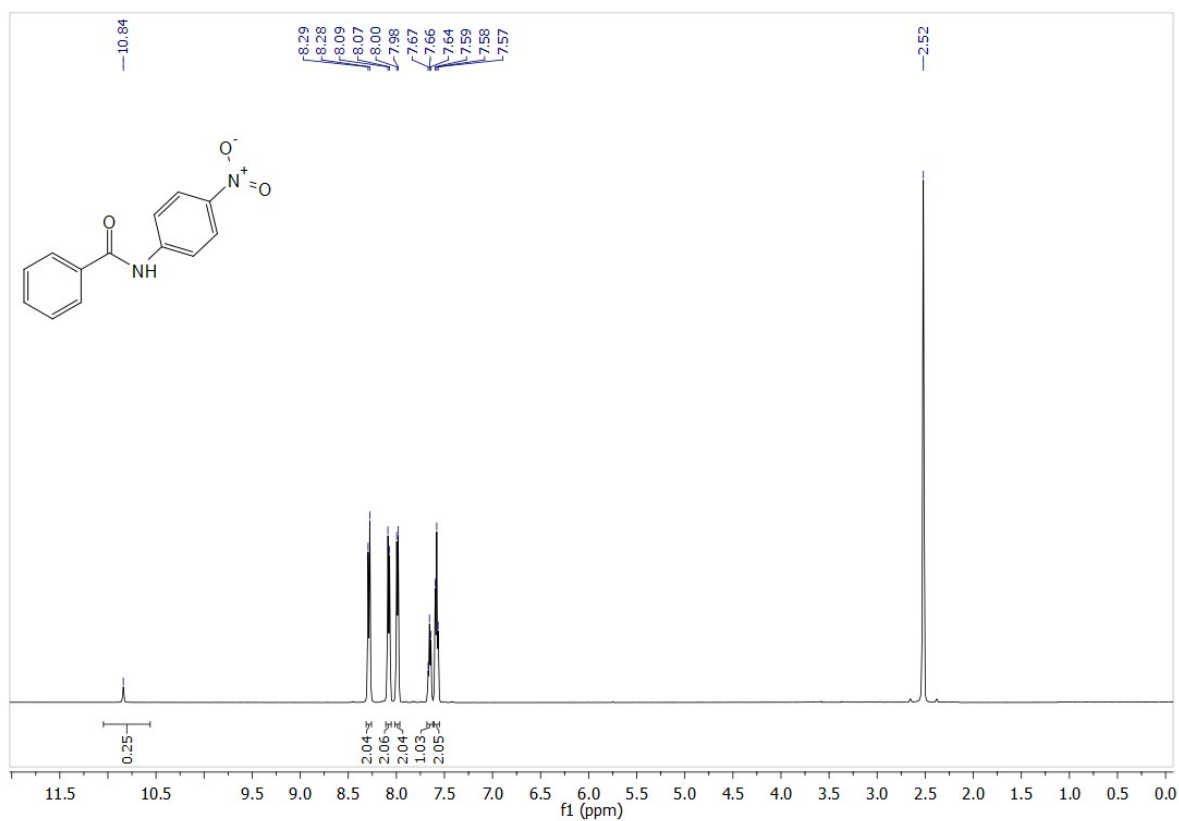


^1H , ^{13}C and ^{19}F NMR Spectra of *N*-(4-fluorophenyl)benzamide (3f)

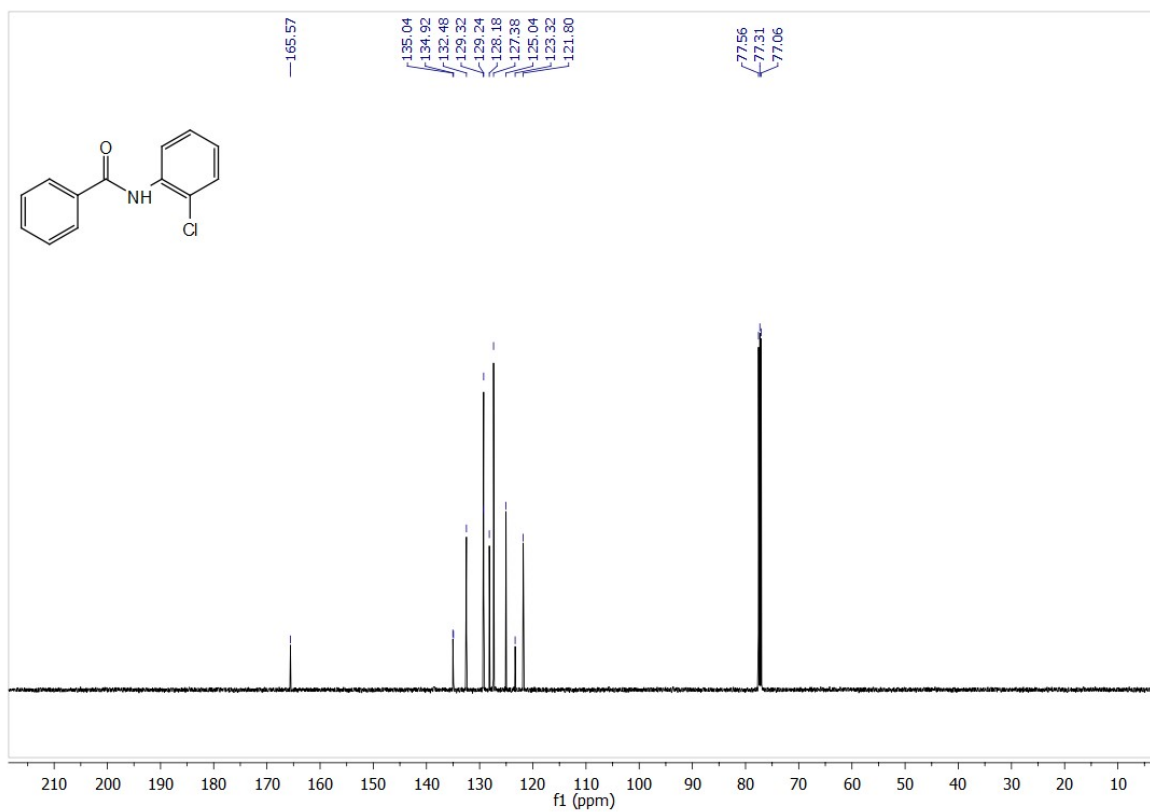
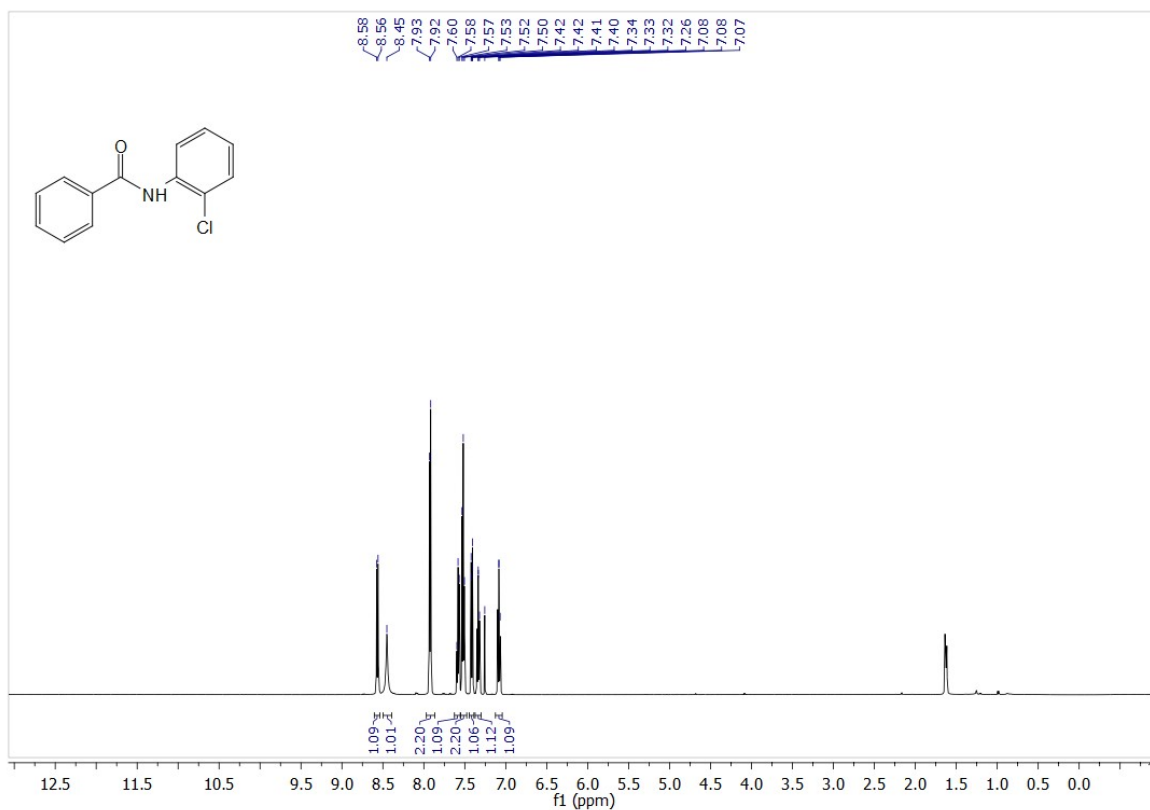




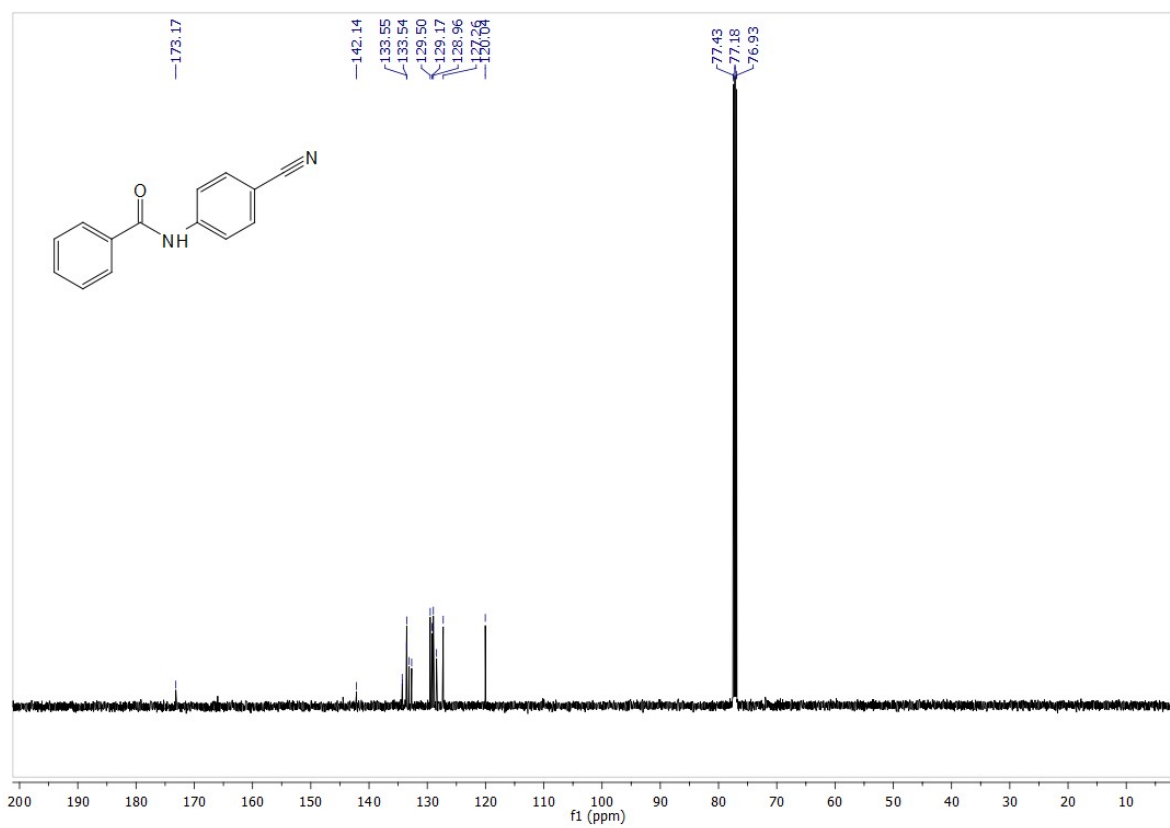
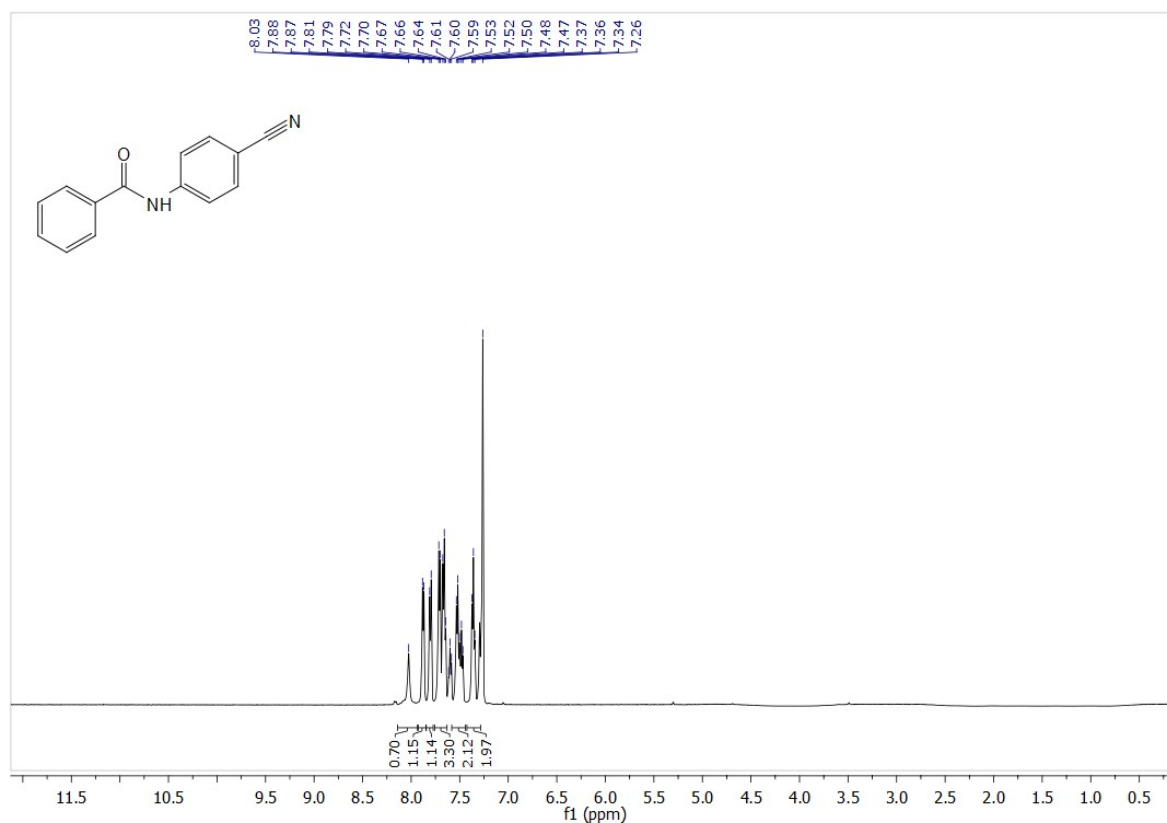
¹H and ¹³C NMR Spectra of *N*-(4-nitrophenyl)benzamide (3g)



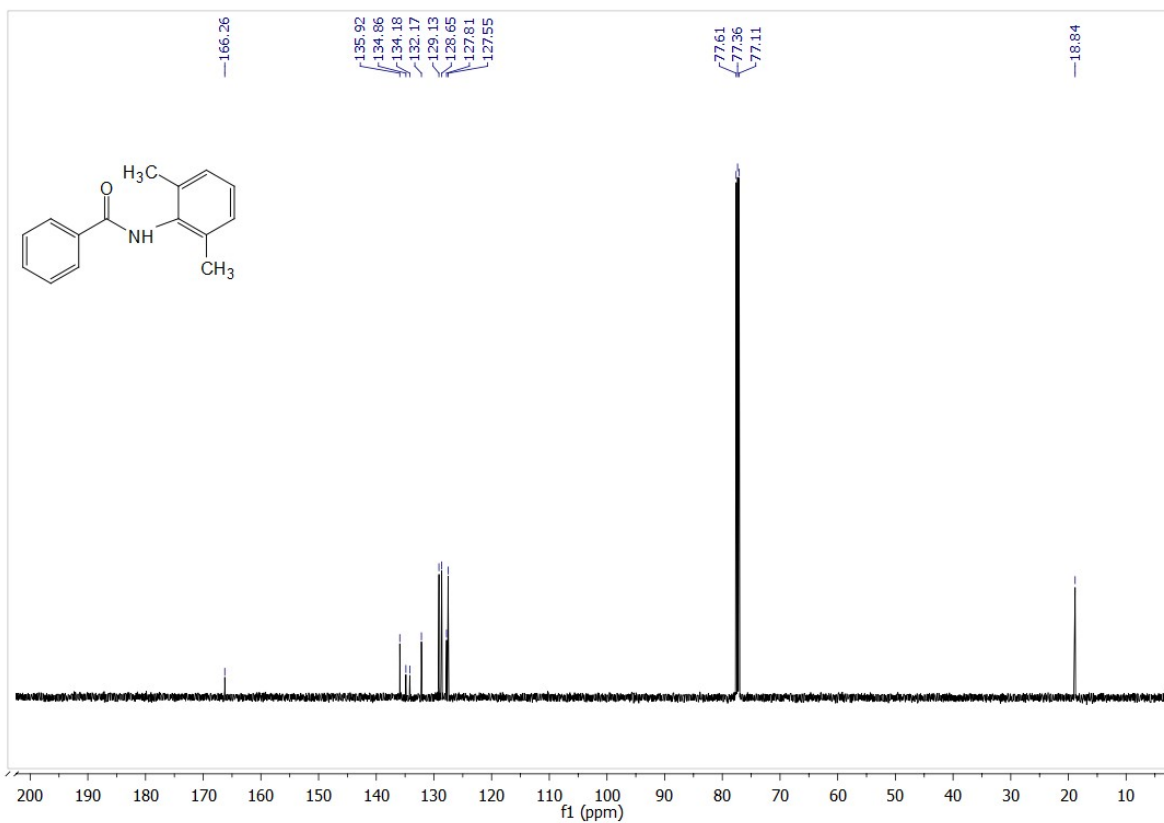
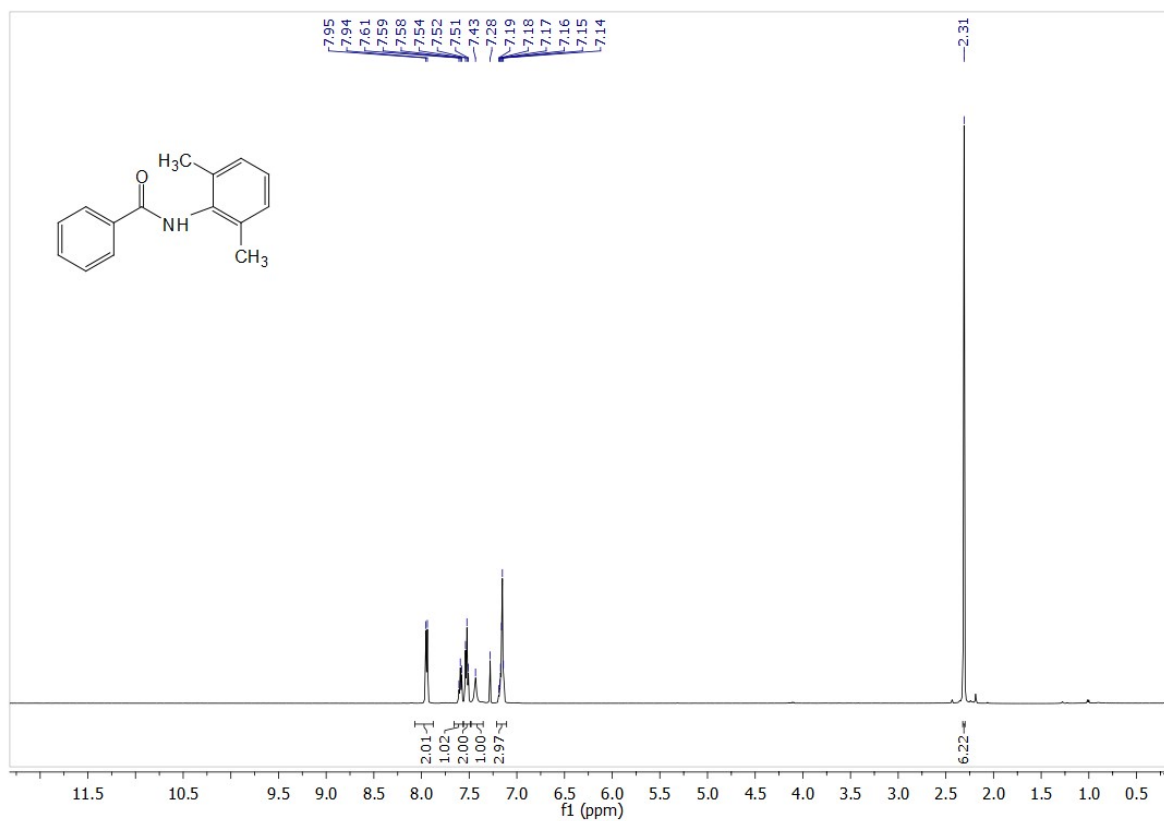
¹H and ¹³C NMR Spectra of *N*-(2-chlorophenyl)benzamide (3h)



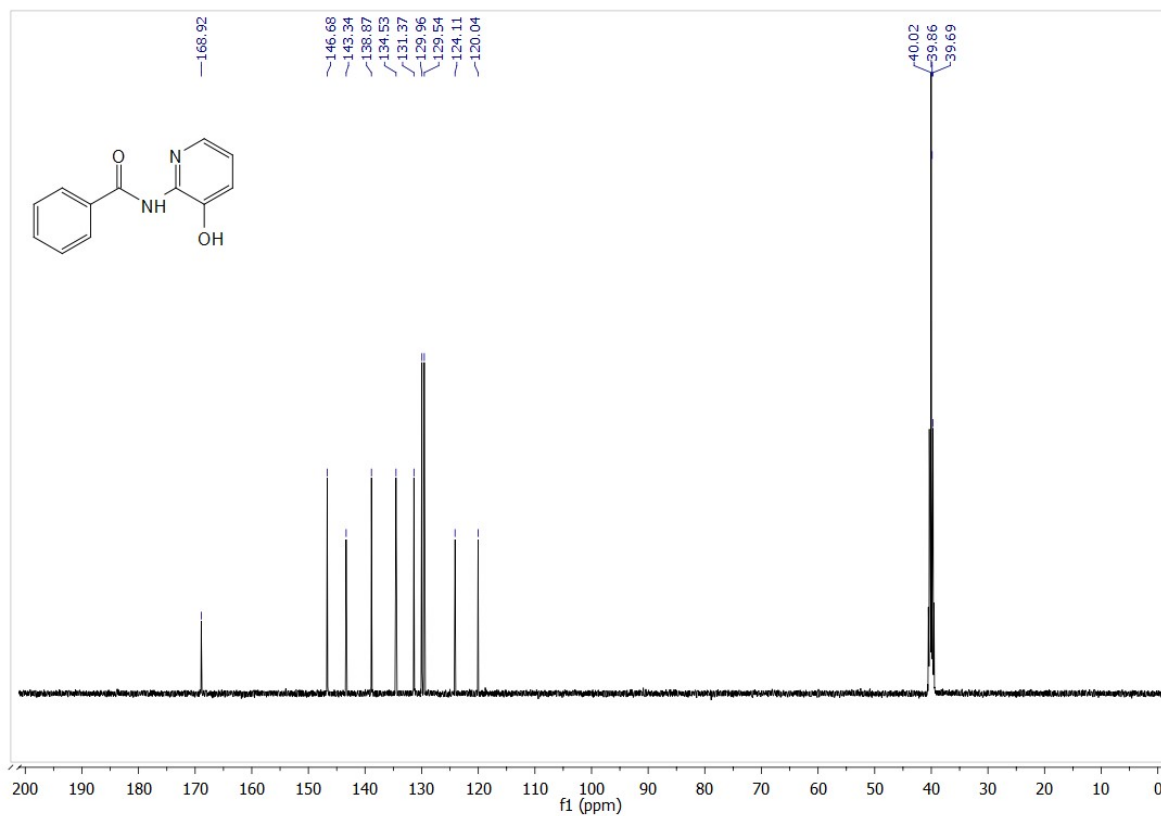
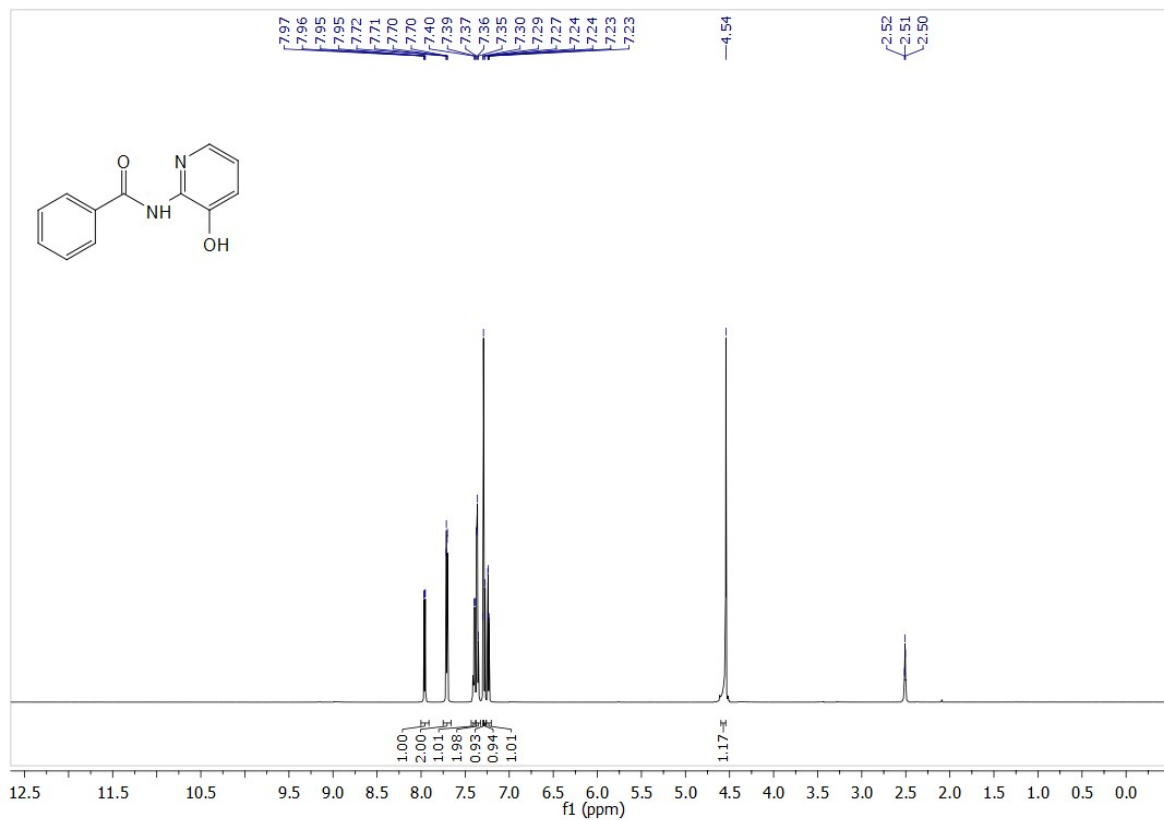
^1H and ^{13}C NMR Spectra of *N*-(4-cynophenyl)benzamide (3i)



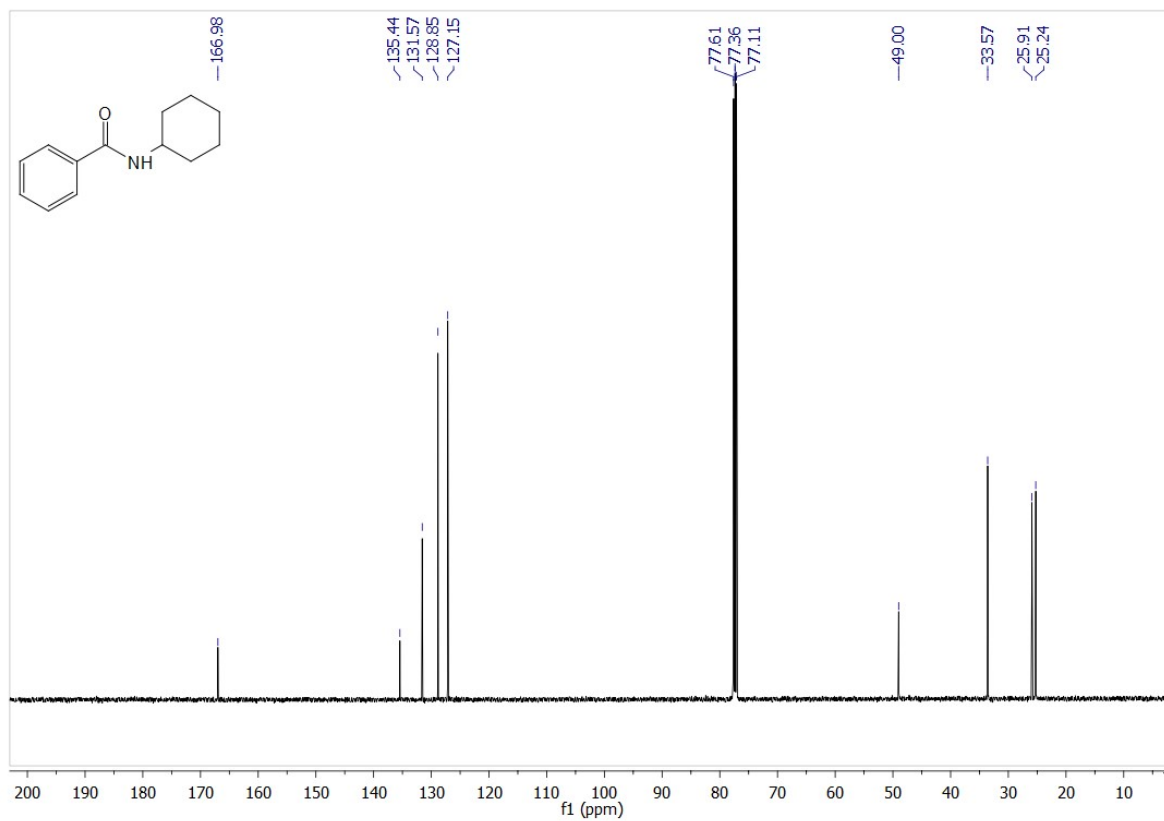
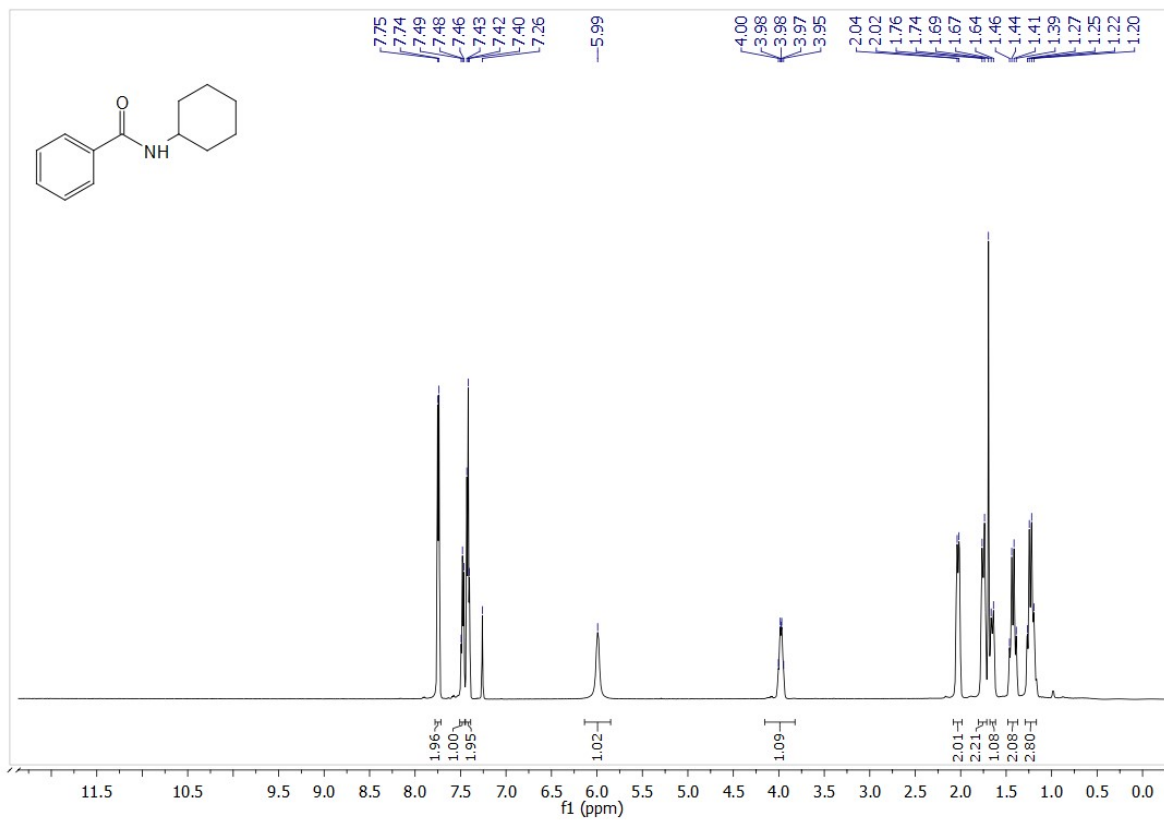
¹H and ¹³C NMR Spectra of *N*-(2,6-dimethylphenyl)benzamide (3j)



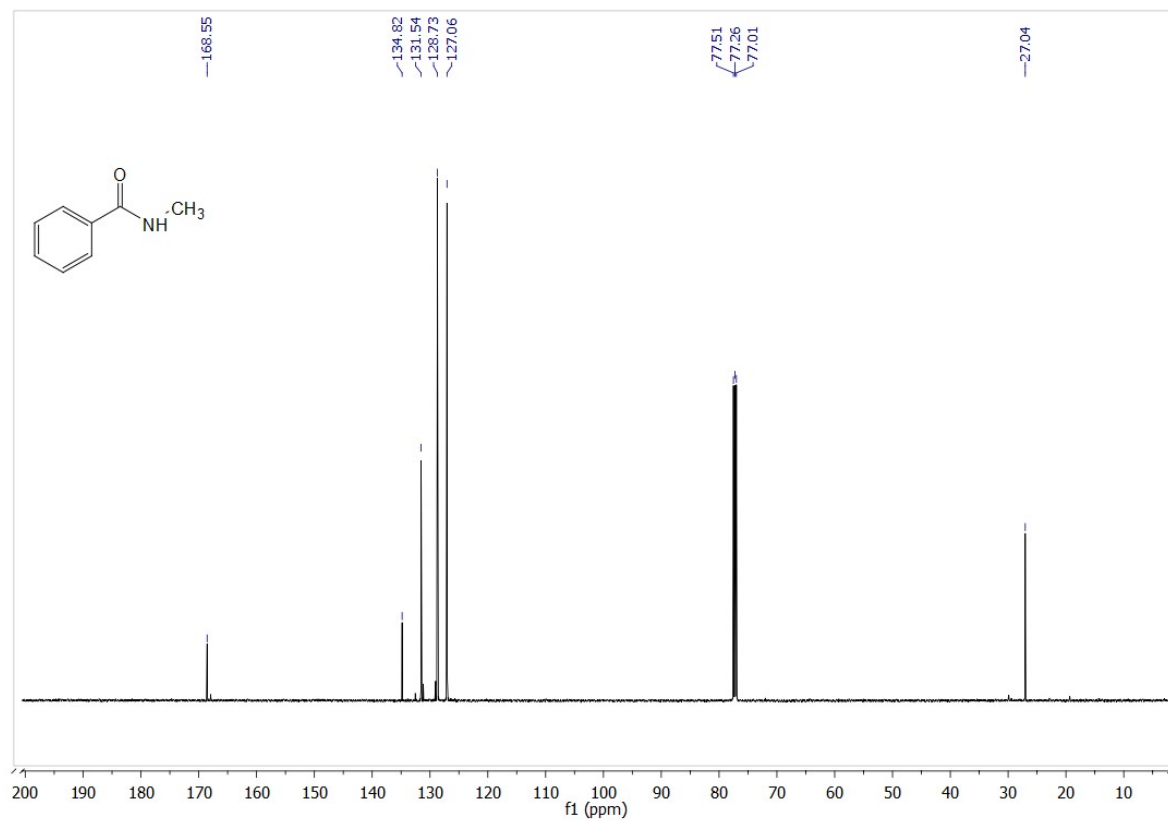
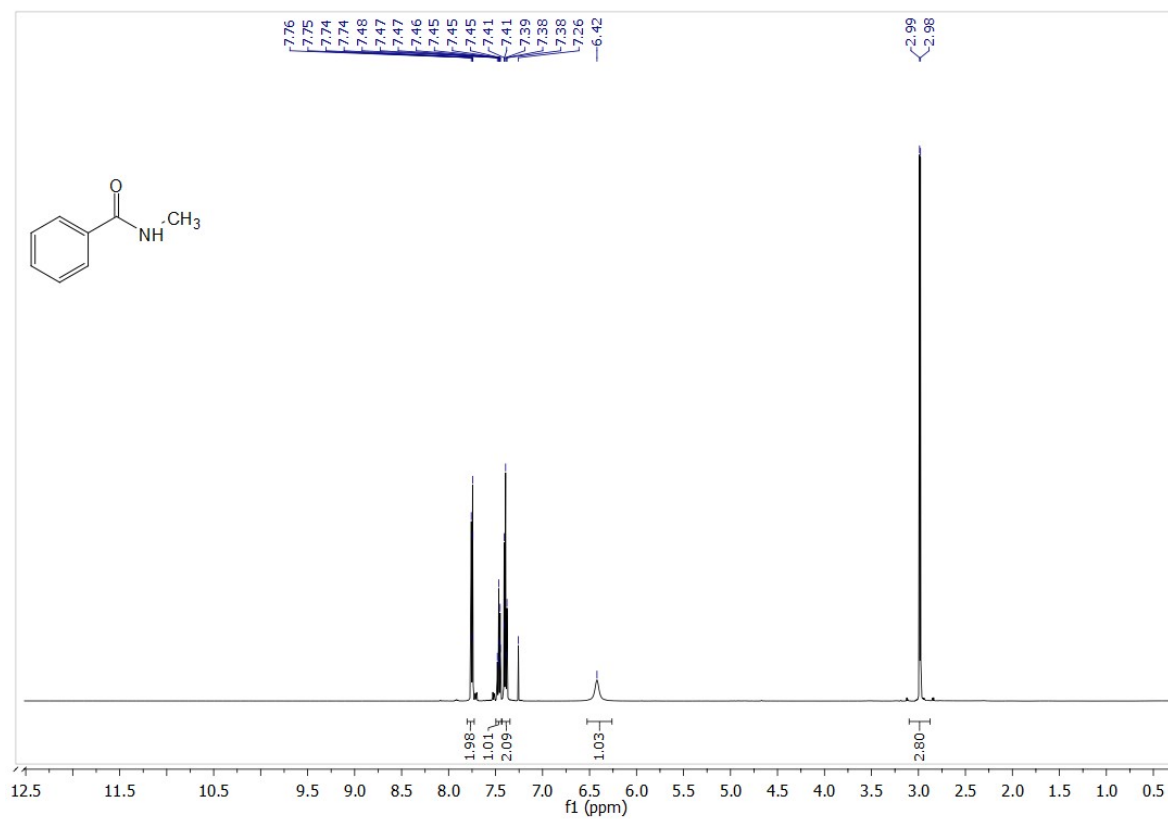
¹H and ¹³C NMR Spectra of *N*-(3-hydroxypyridin-2-yl)benzamide (3k)



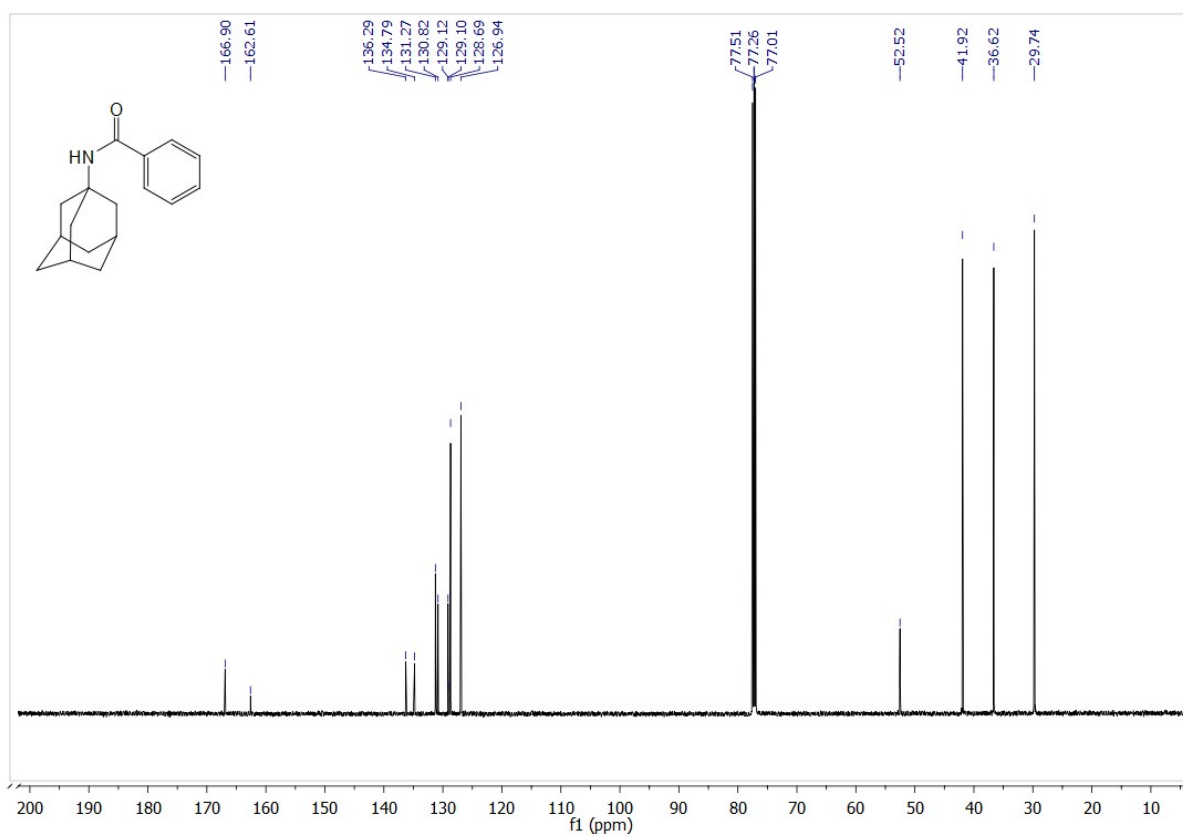
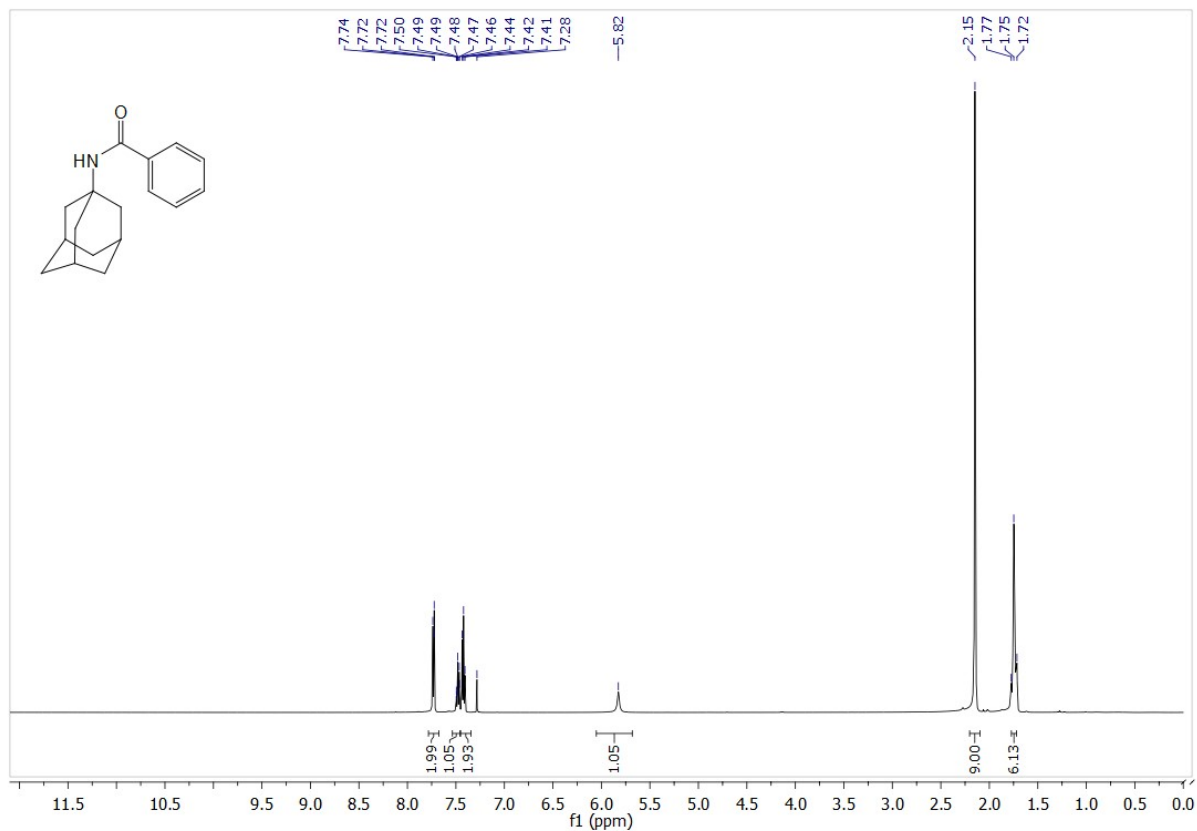
¹H and ¹³C NMR Spectra of *N*-cyclohexylbenzamide (31)



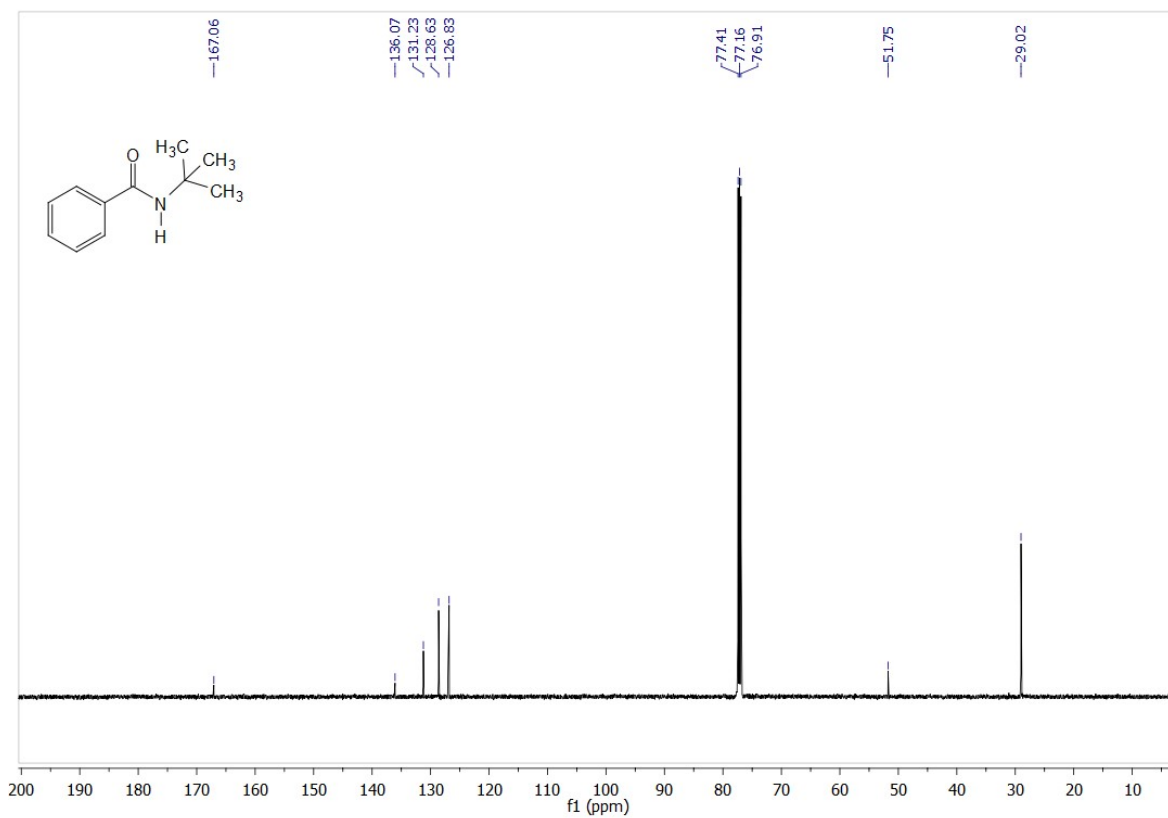
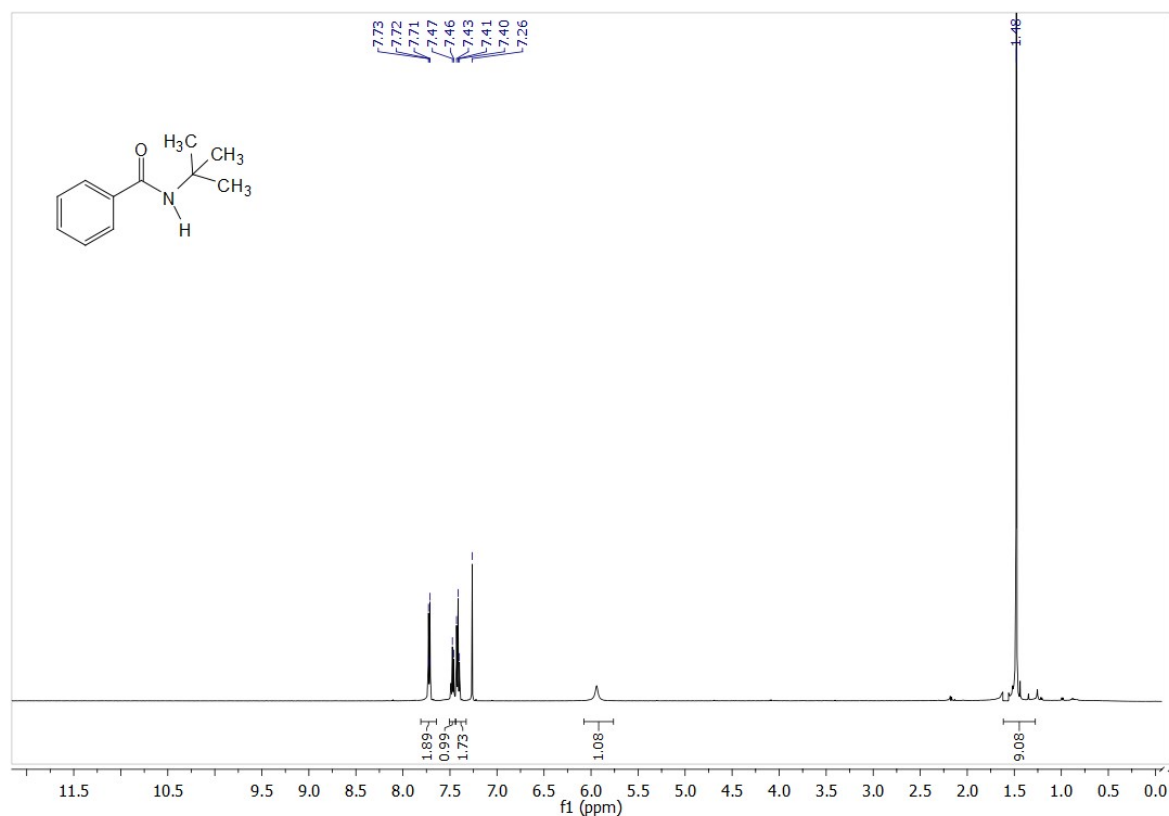
^1H and ^{13}C NMR Spectra of *N*-methylbenzamide (3m)



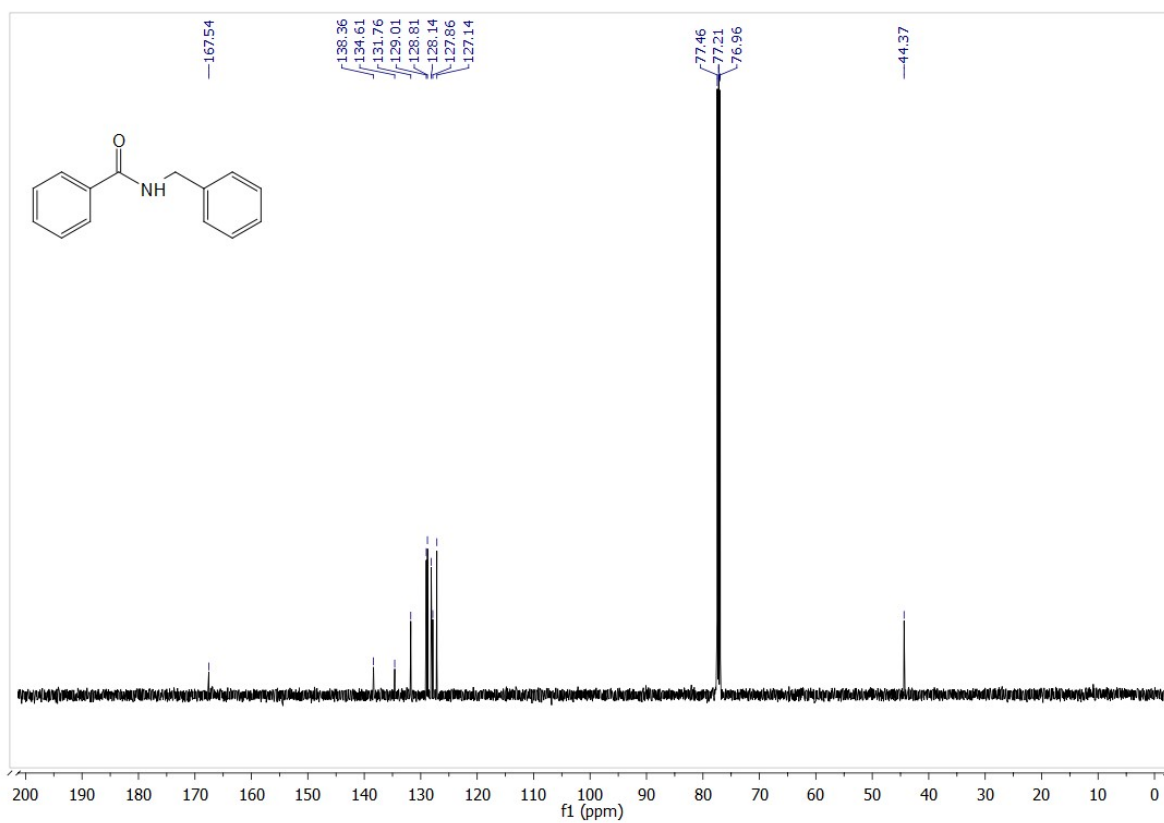
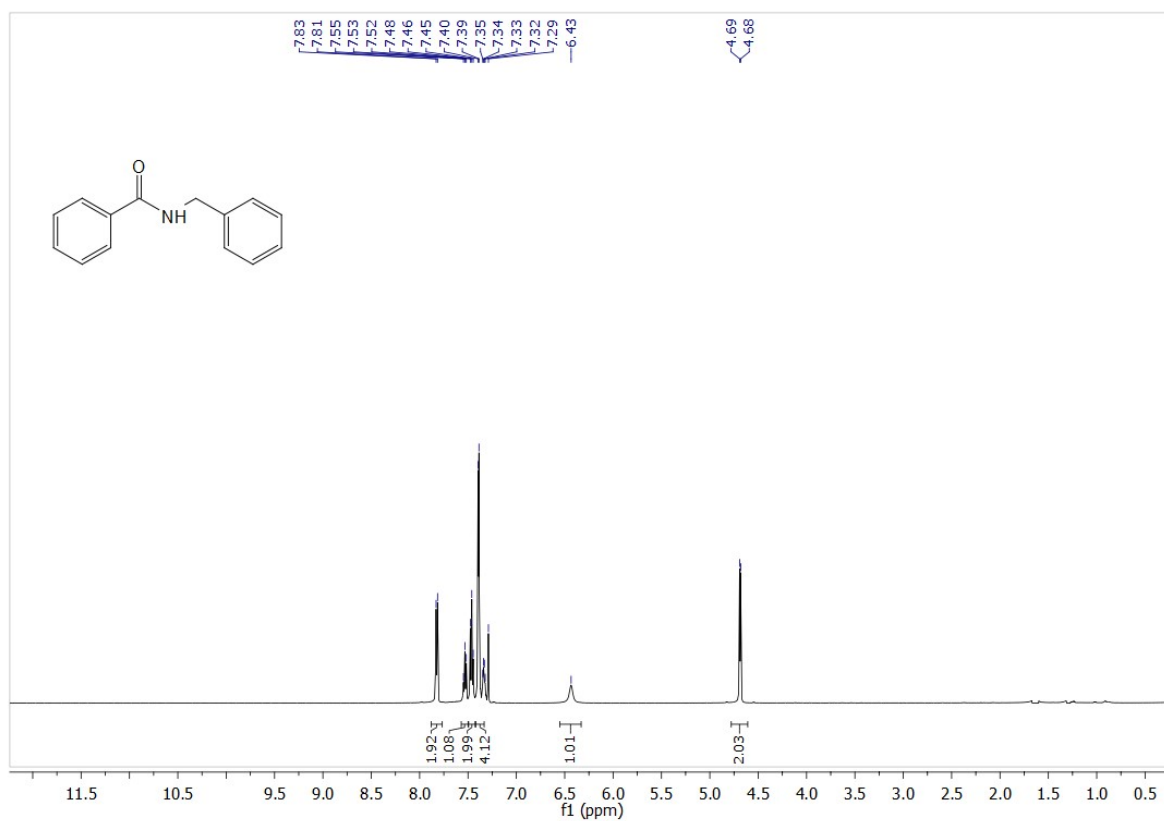
^1H and ^{13}C NMR Spectra of *N*-(3*s*,5*s*,7*s*)-adamantan-1-yl)benzamide (3n)



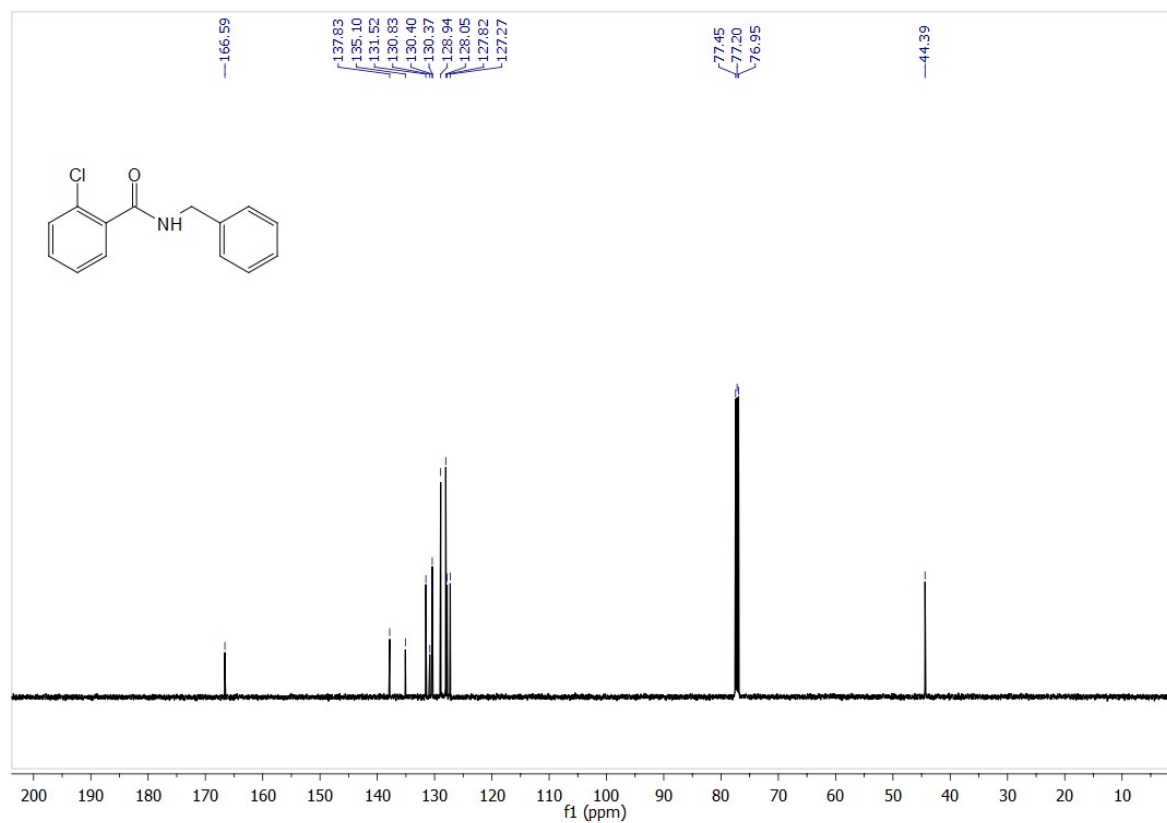
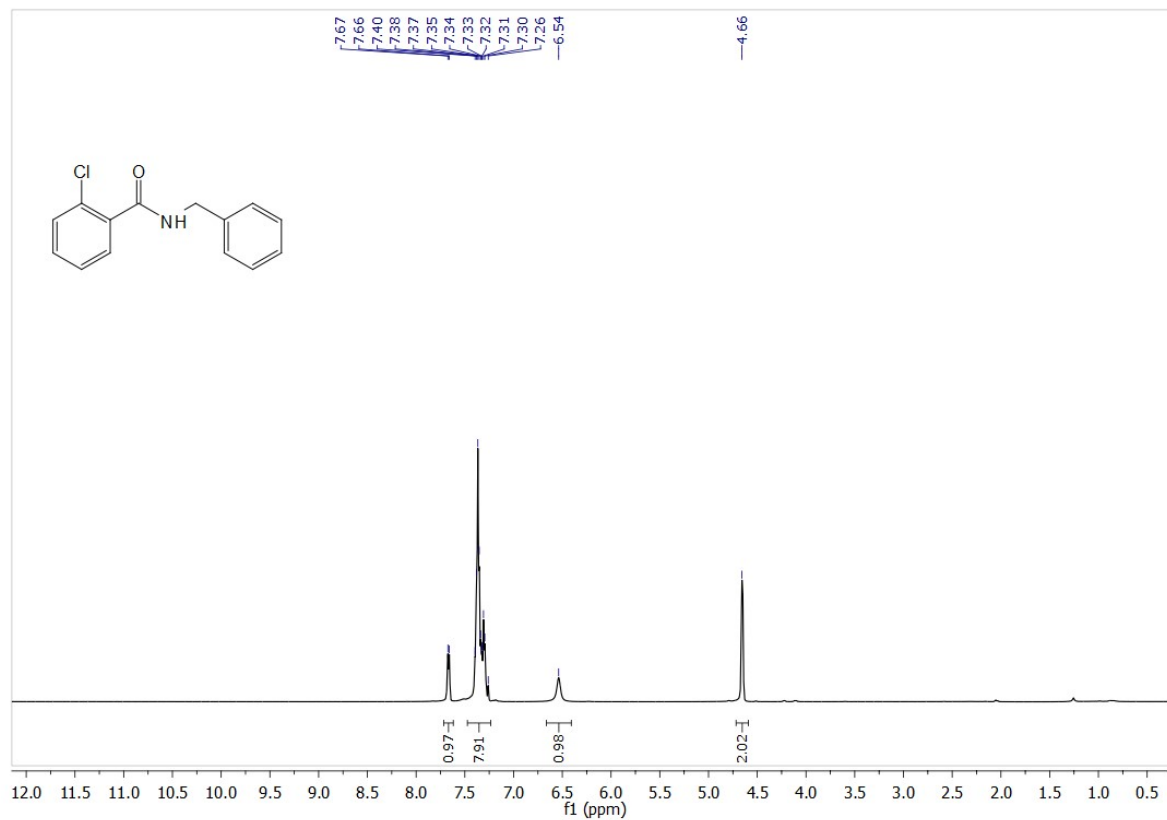
^1H and ^{13}C NMR Spectra of *N*-(*tert*-butyl)benzamide (3o)



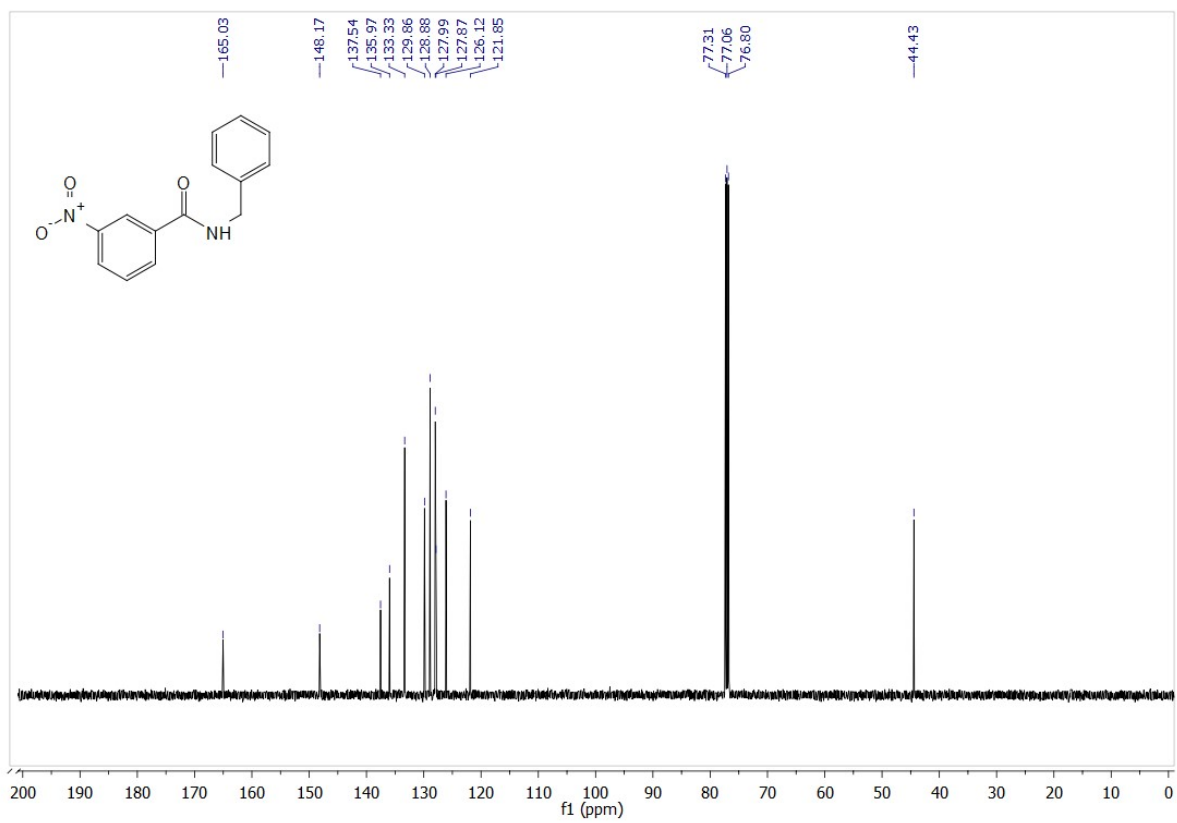
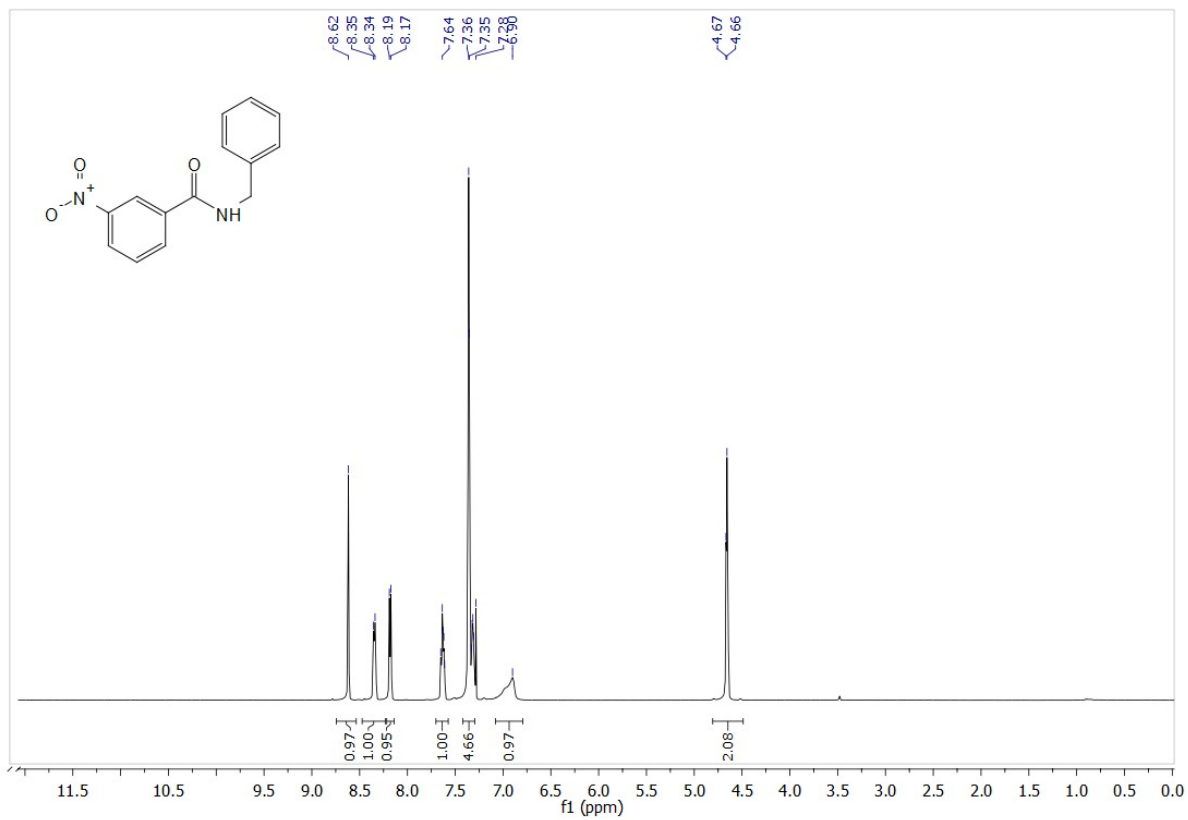
^1H and ^{13}C NMR Spectra of *N*-benzylbenzamide (3p)



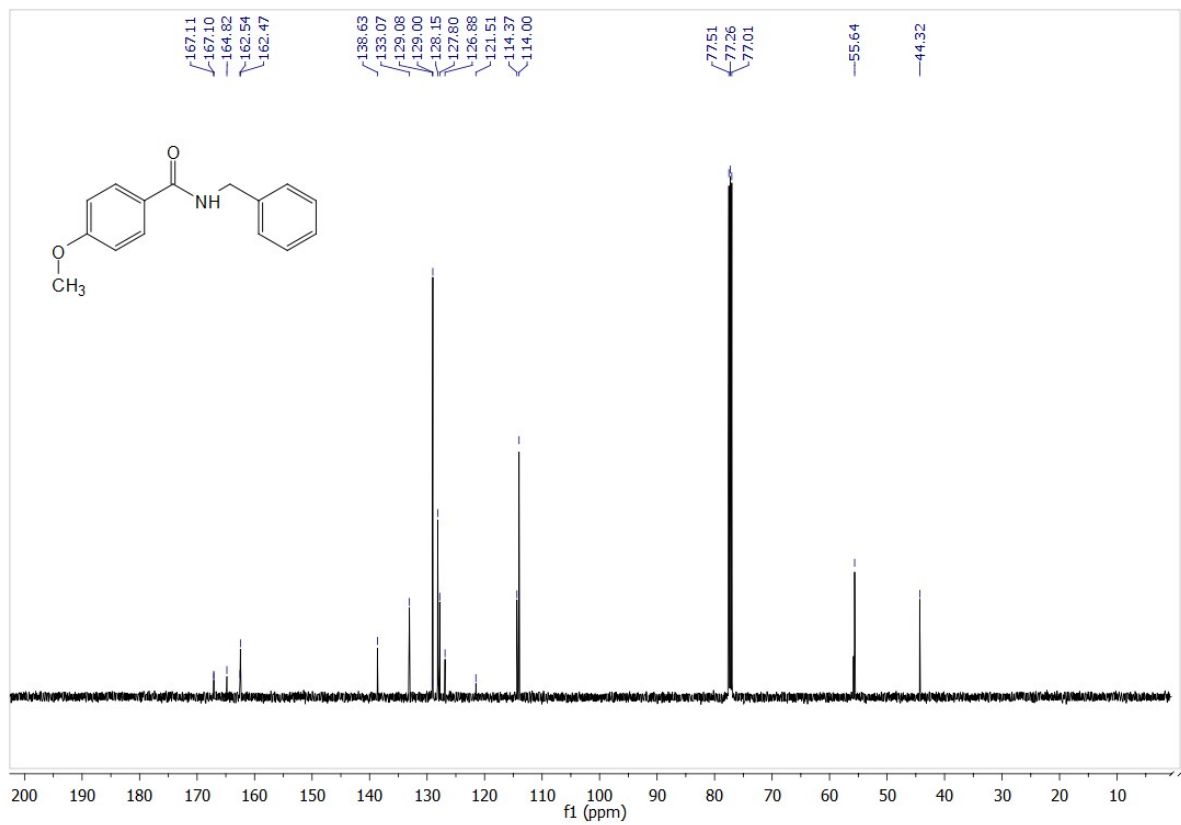
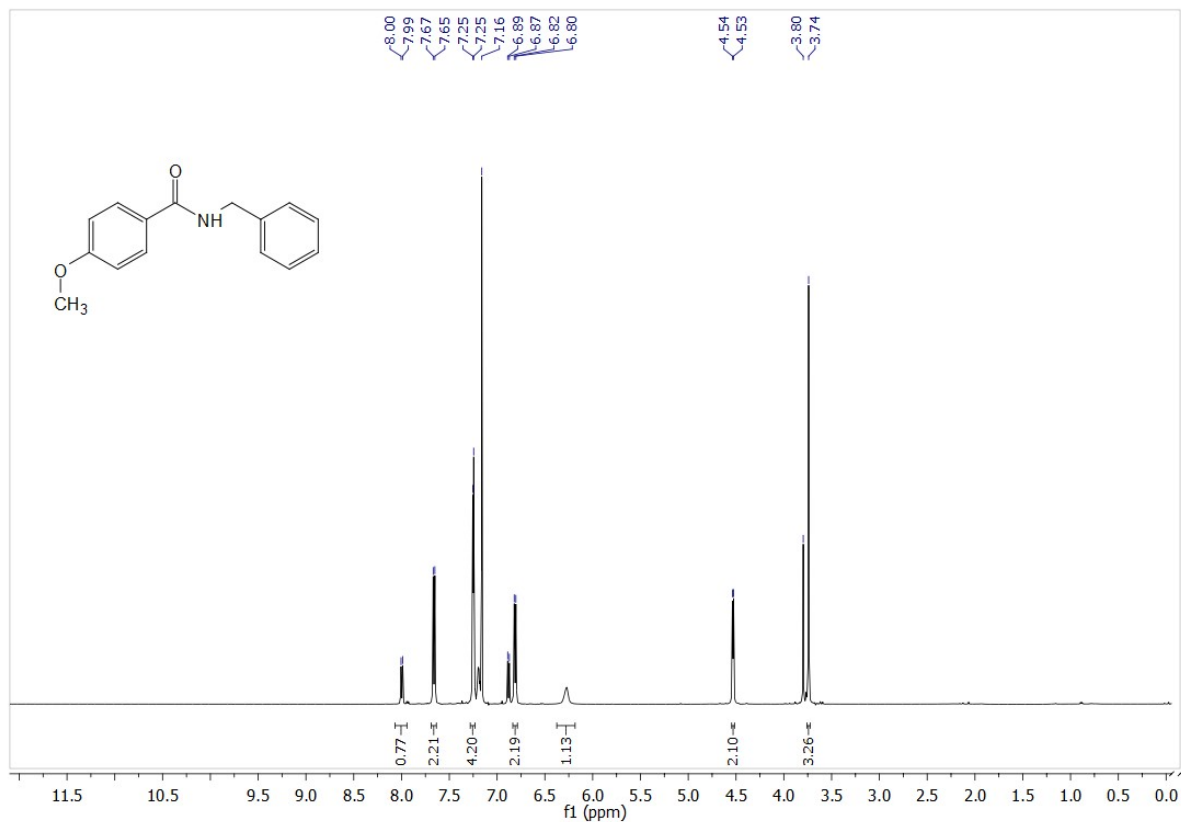
^1H and ^{13}C NMR Spectra of *N*-benzyl-2-chlorobenzamide (3q)



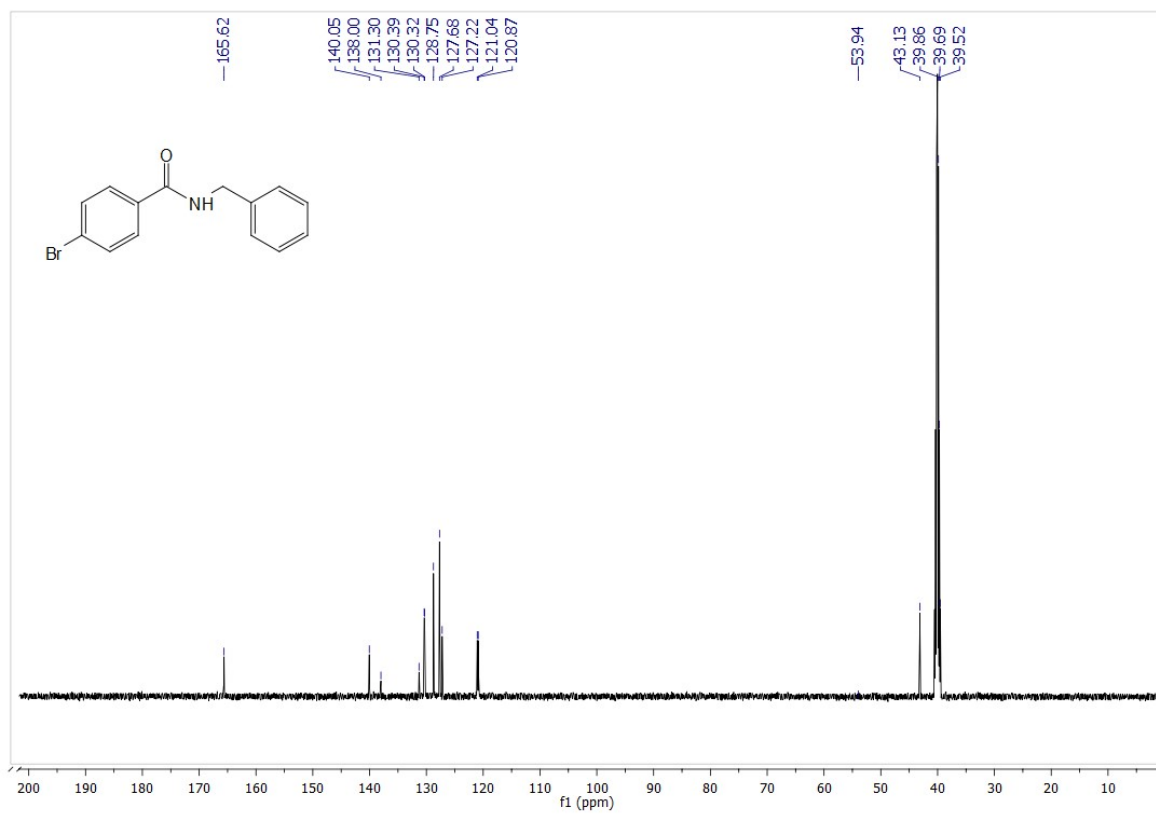
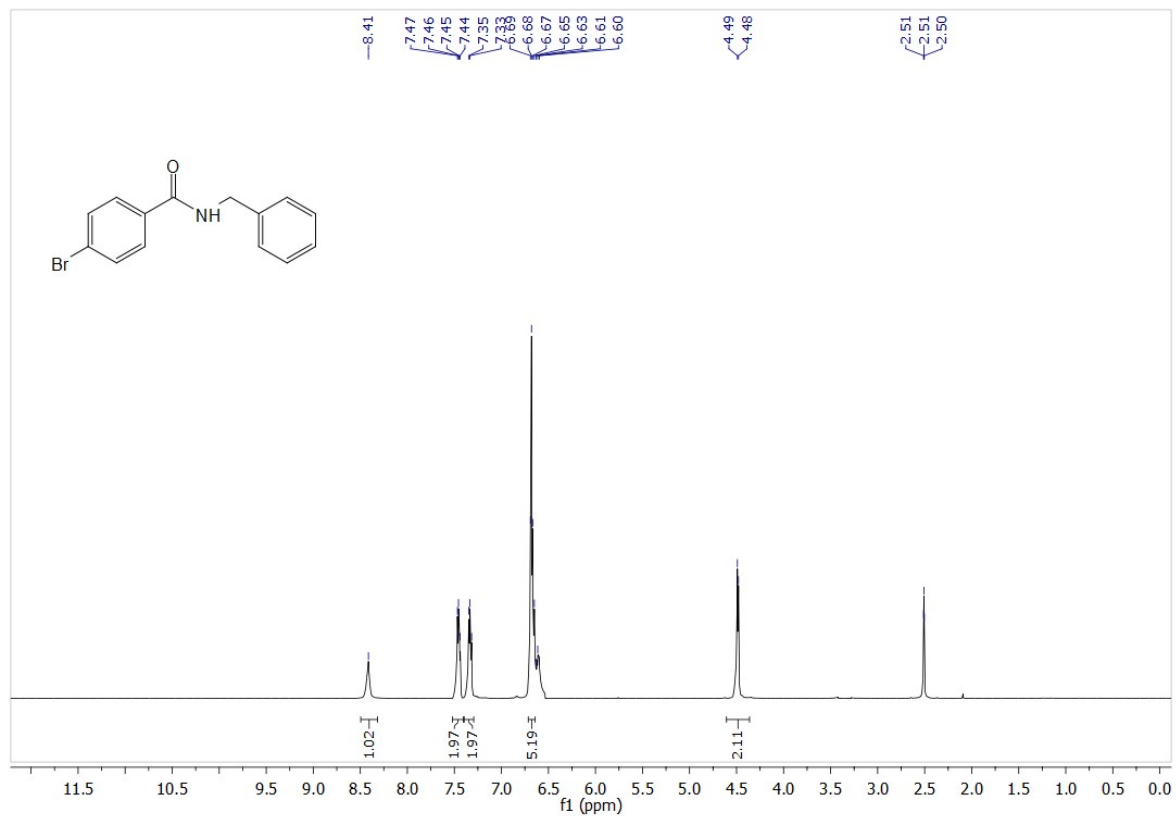
¹H and ¹³C NMR Spectra of *N*-benzyl-3-nitrobenzamide (3r)



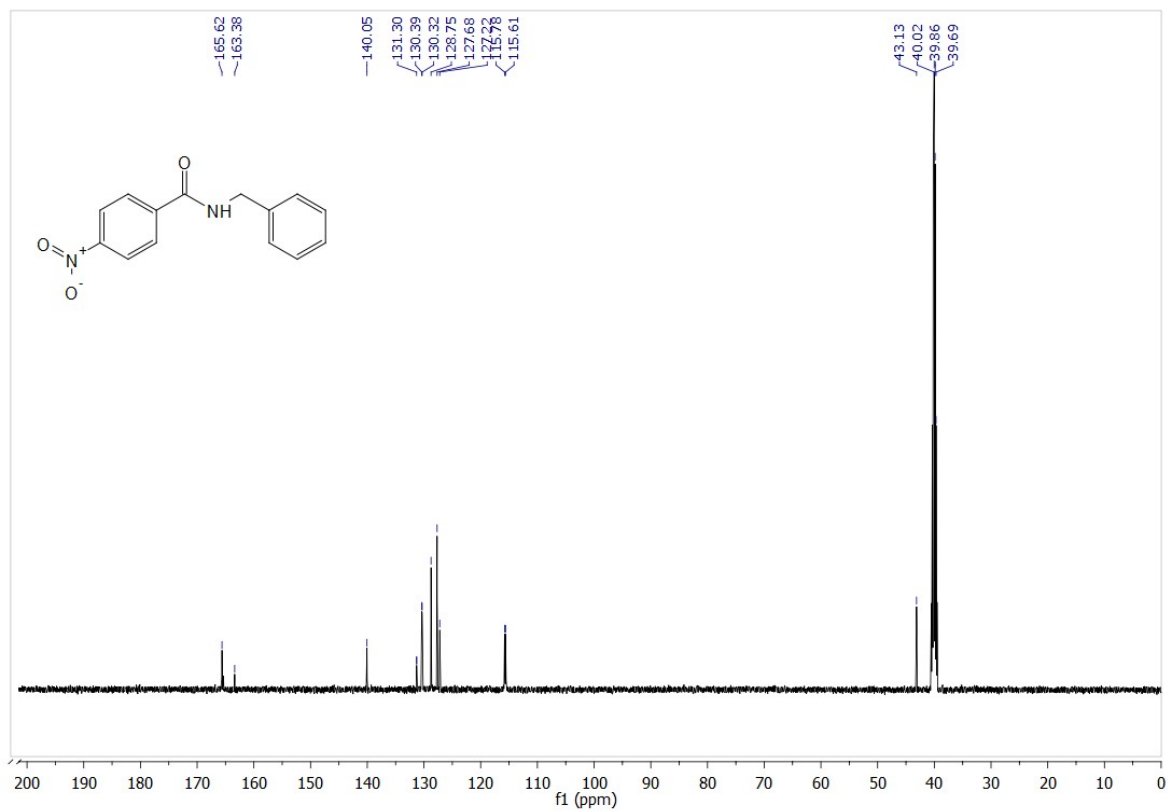
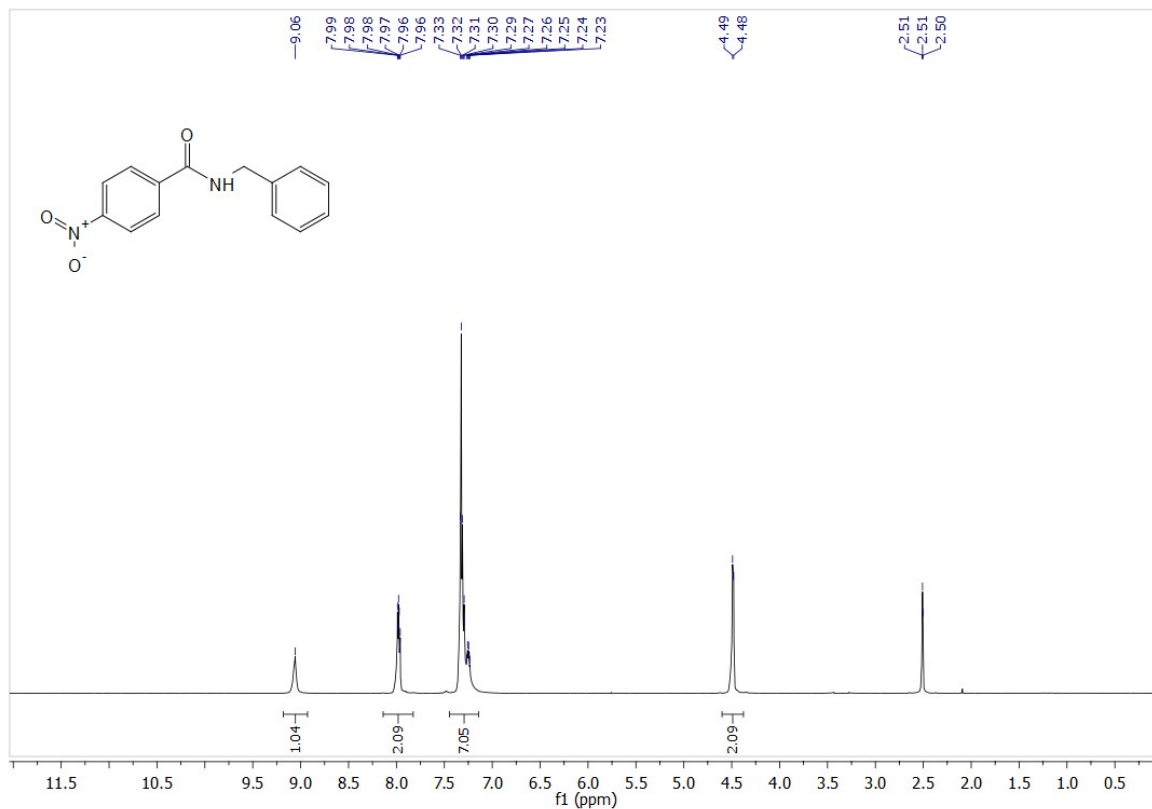
¹H and ¹³C NMR Spectra of *N*-benzyl-4-methoxybenzamide (3s)



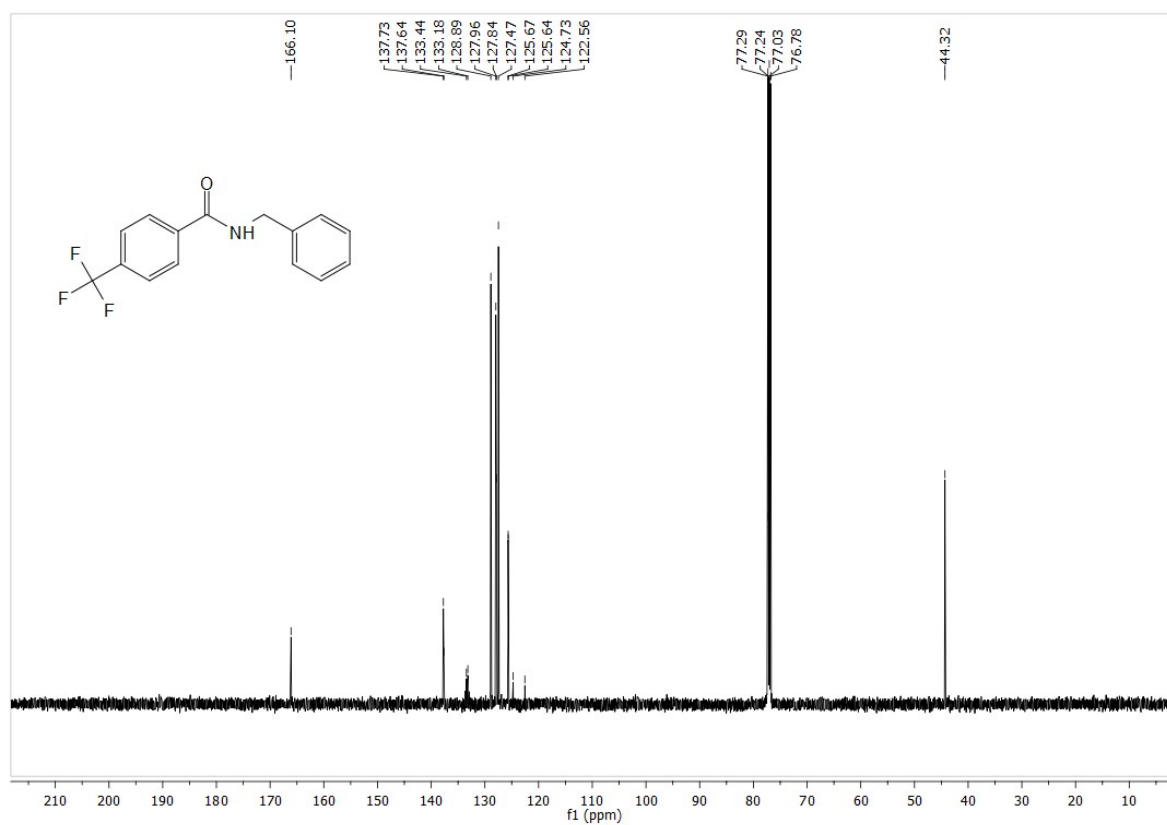
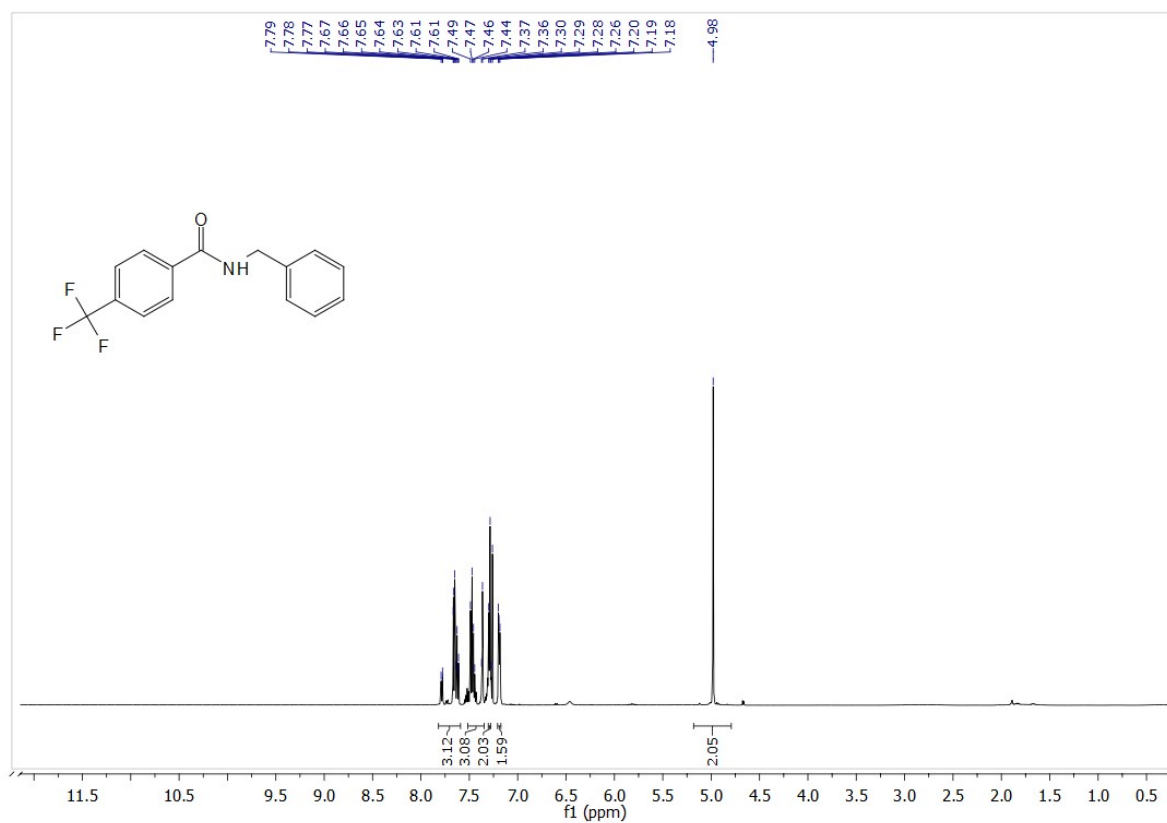
¹H and ¹³C NMR Spectra of *N*-benzyl-4-bromobenzamide (3t)

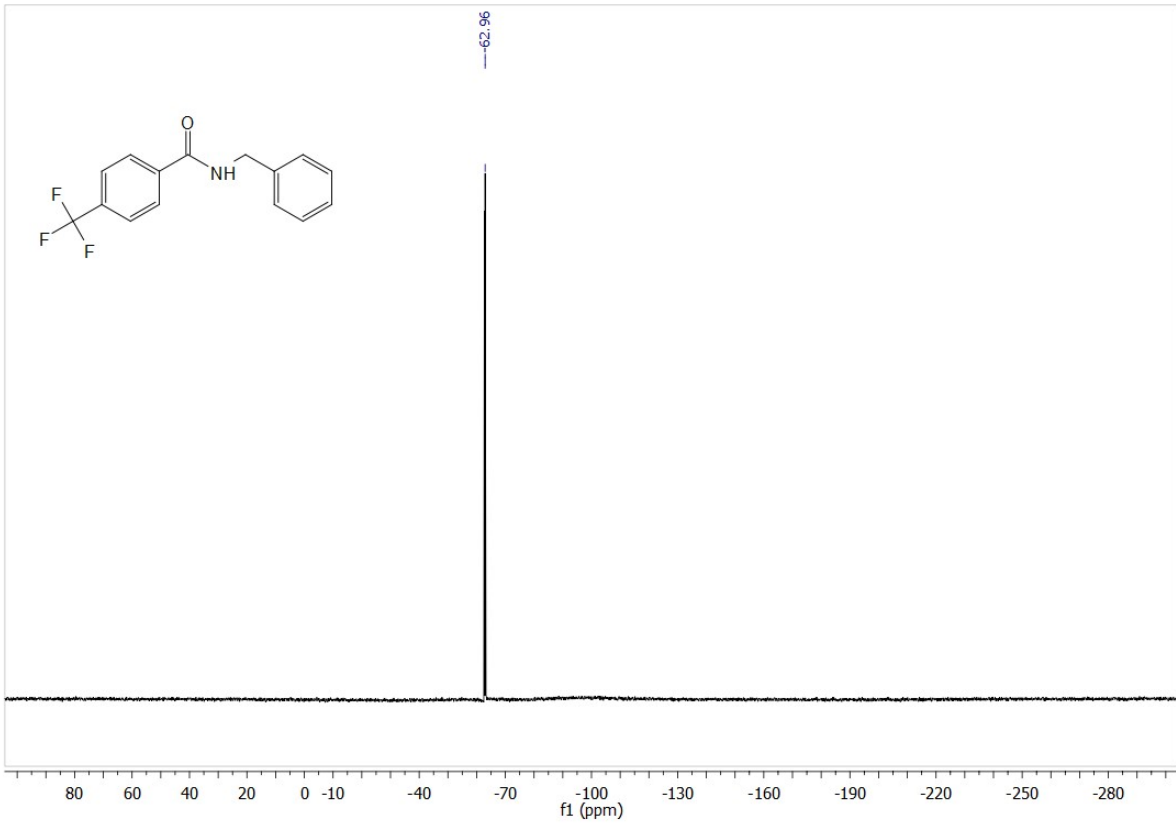


¹H and ¹³C NMR Spectra of *N*-benzyl-4-nitrobenzamide (3u)

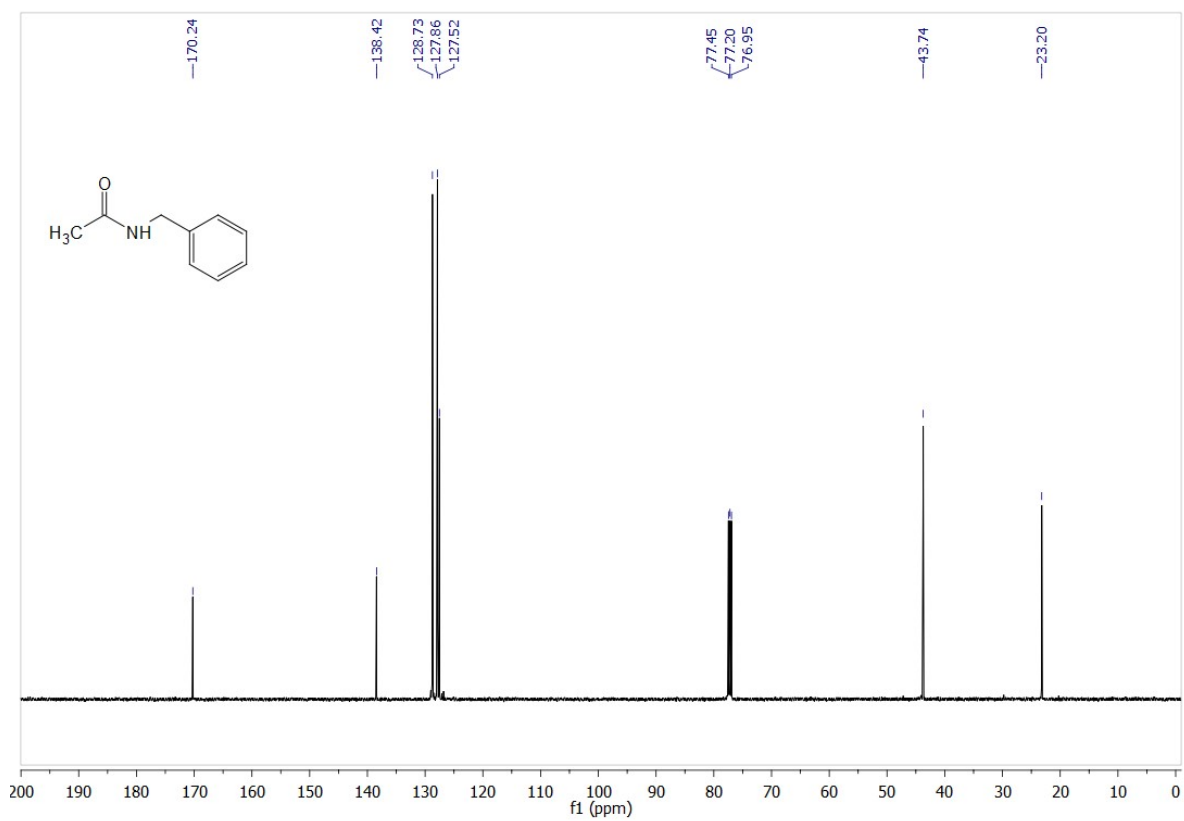
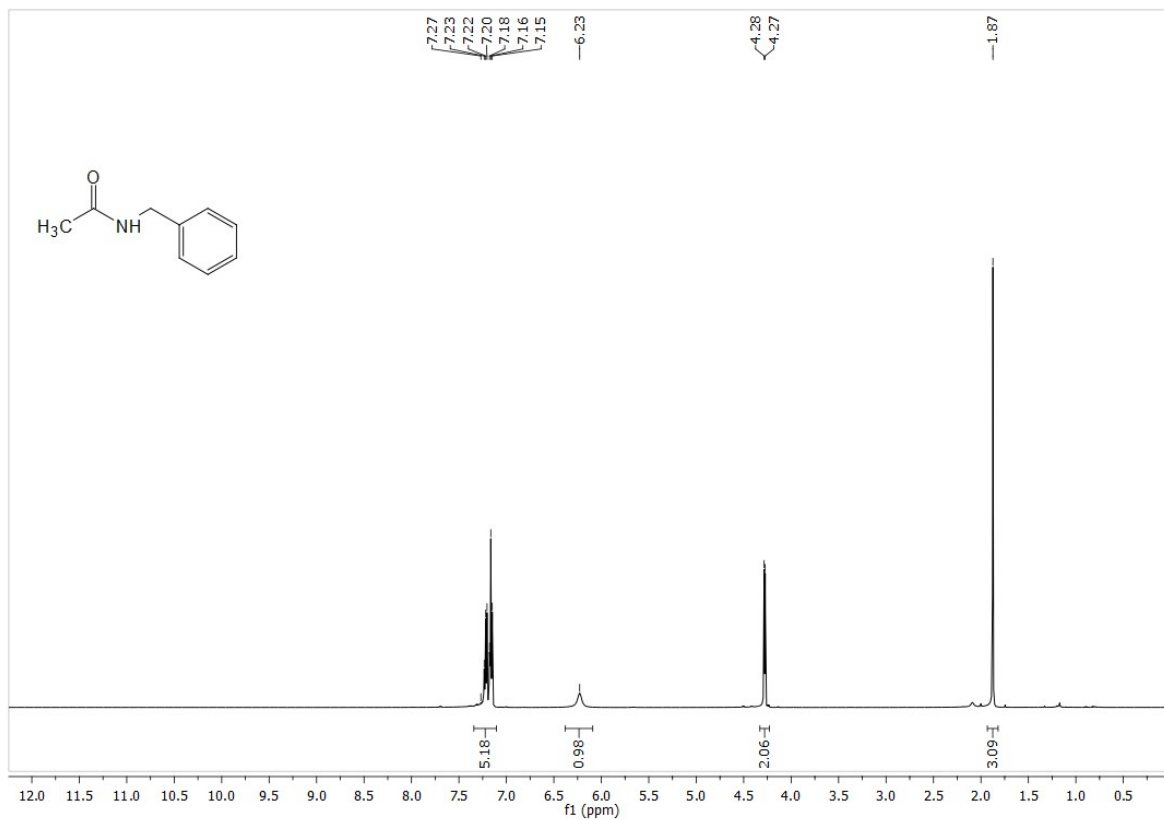


^1H , ^{13}C and ^{19}F NMR Spectra of *N*-benzyl-4-fluorobenzamide (3v)

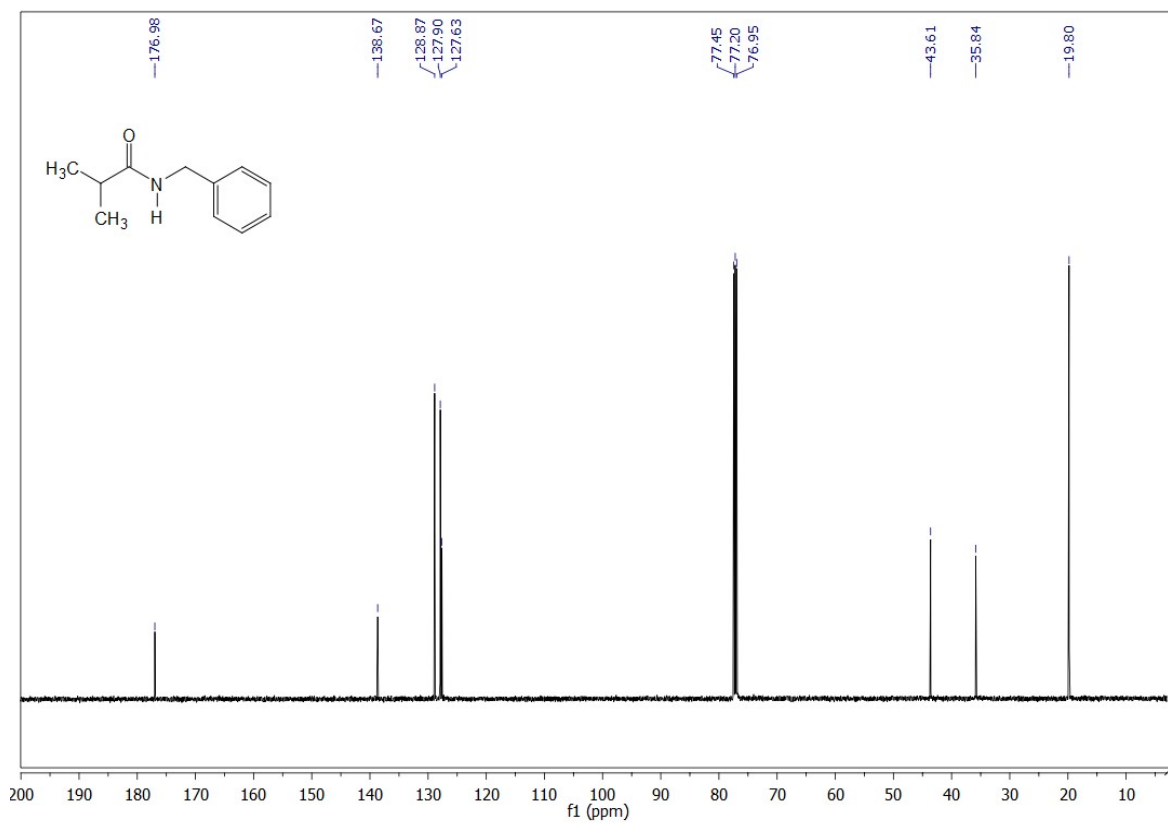




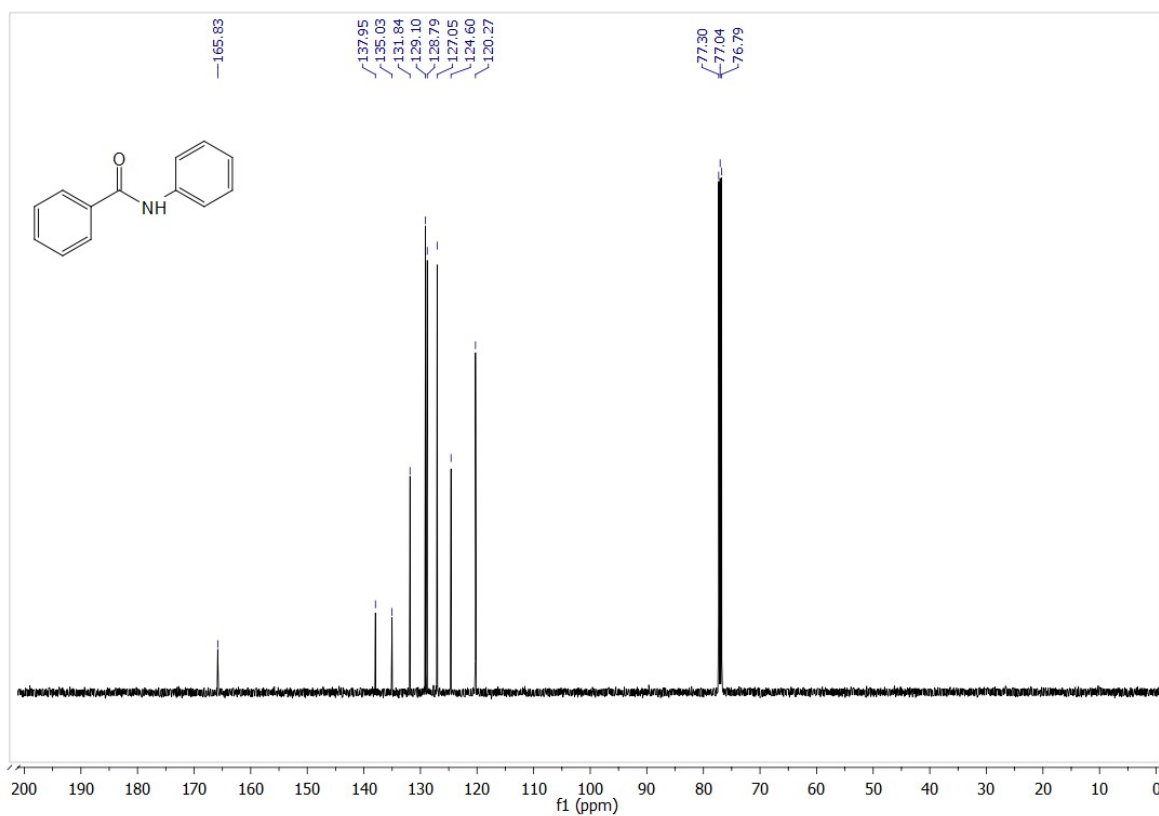
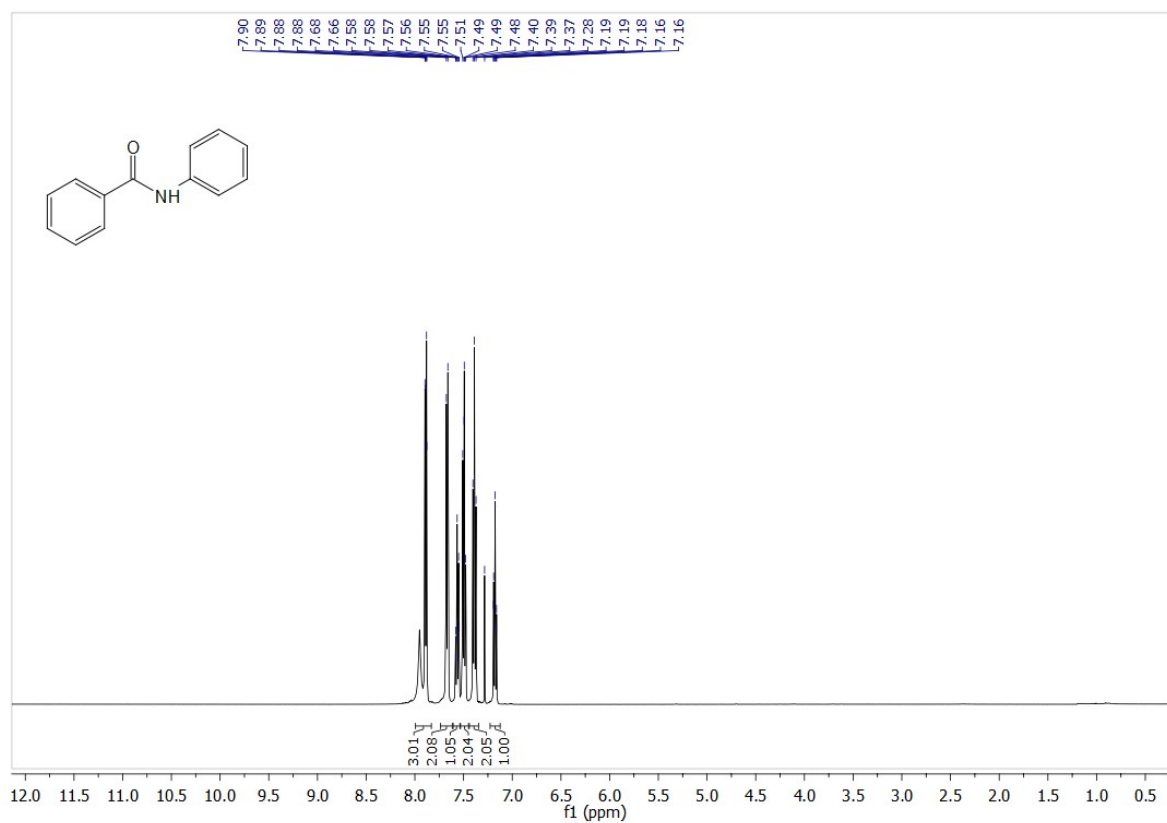
¹H and ¹³C NMR Spectra of *N*-benzylacetamide (3w)



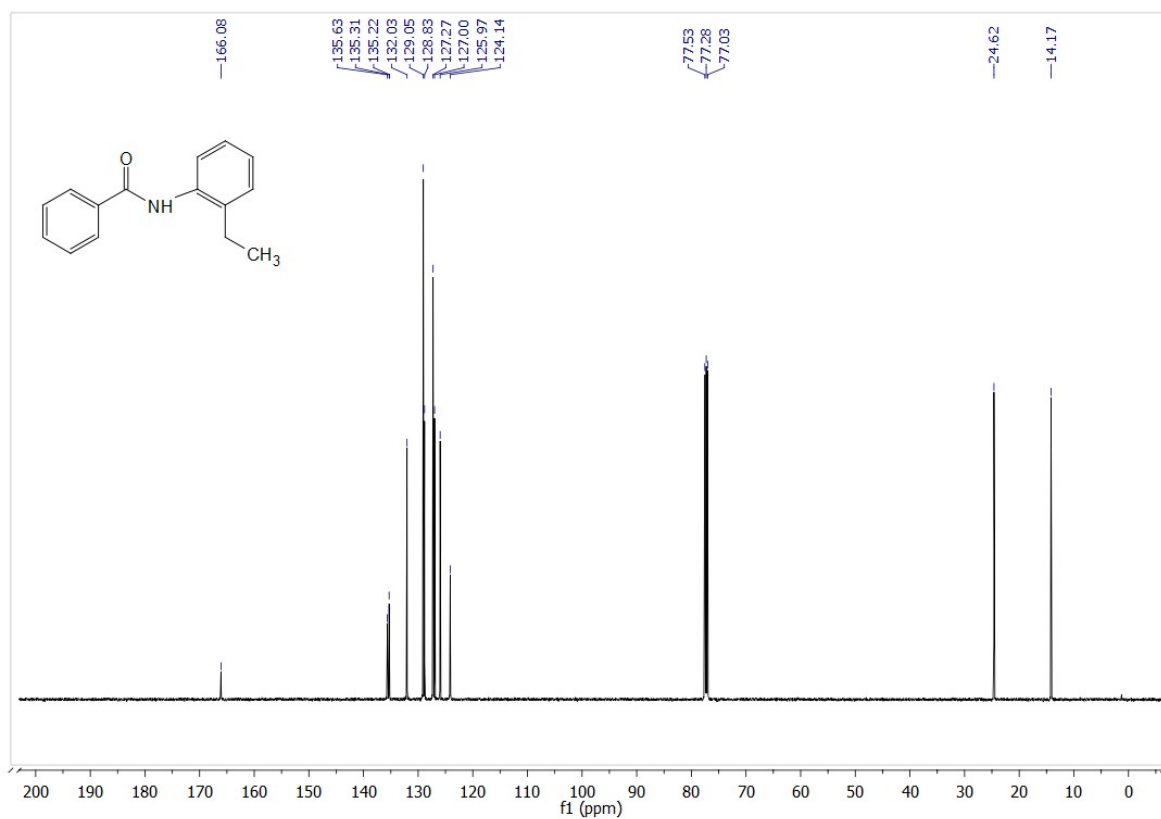
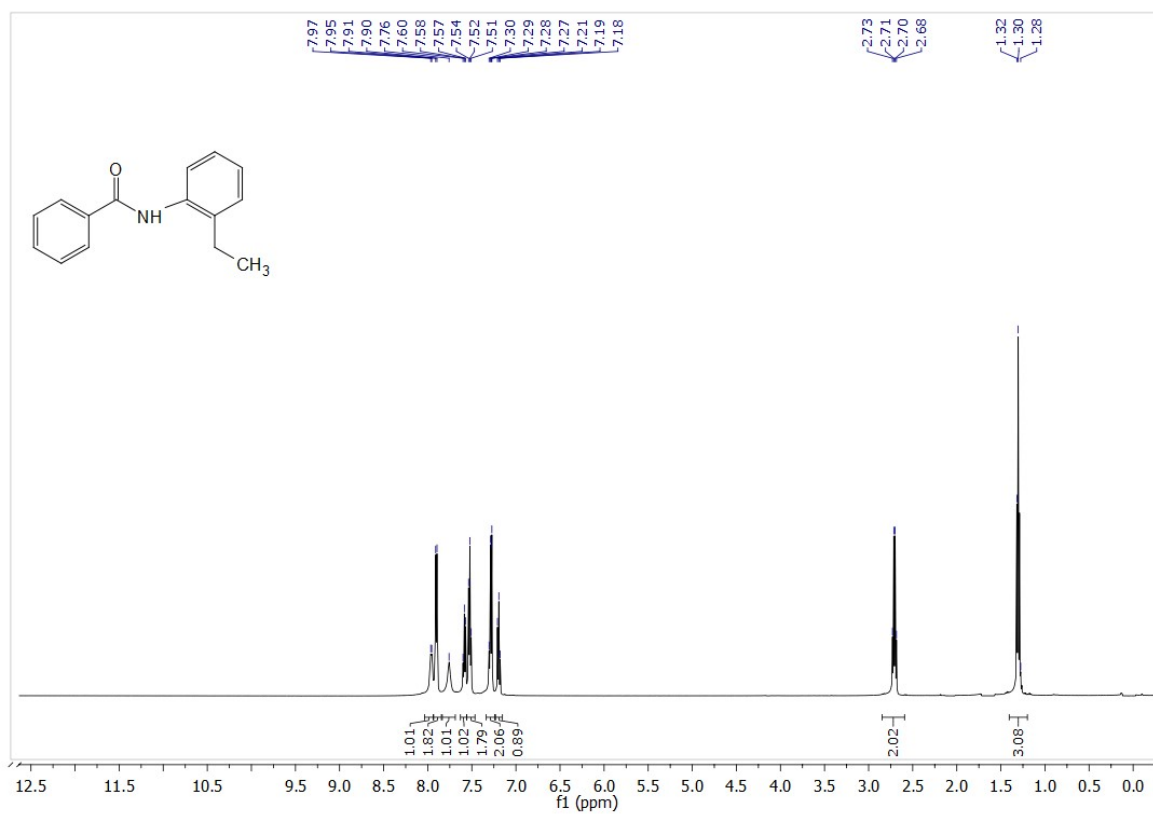
¹H and ¹³C NMR Spectra of *N*-phenylisobutyramide (3x)



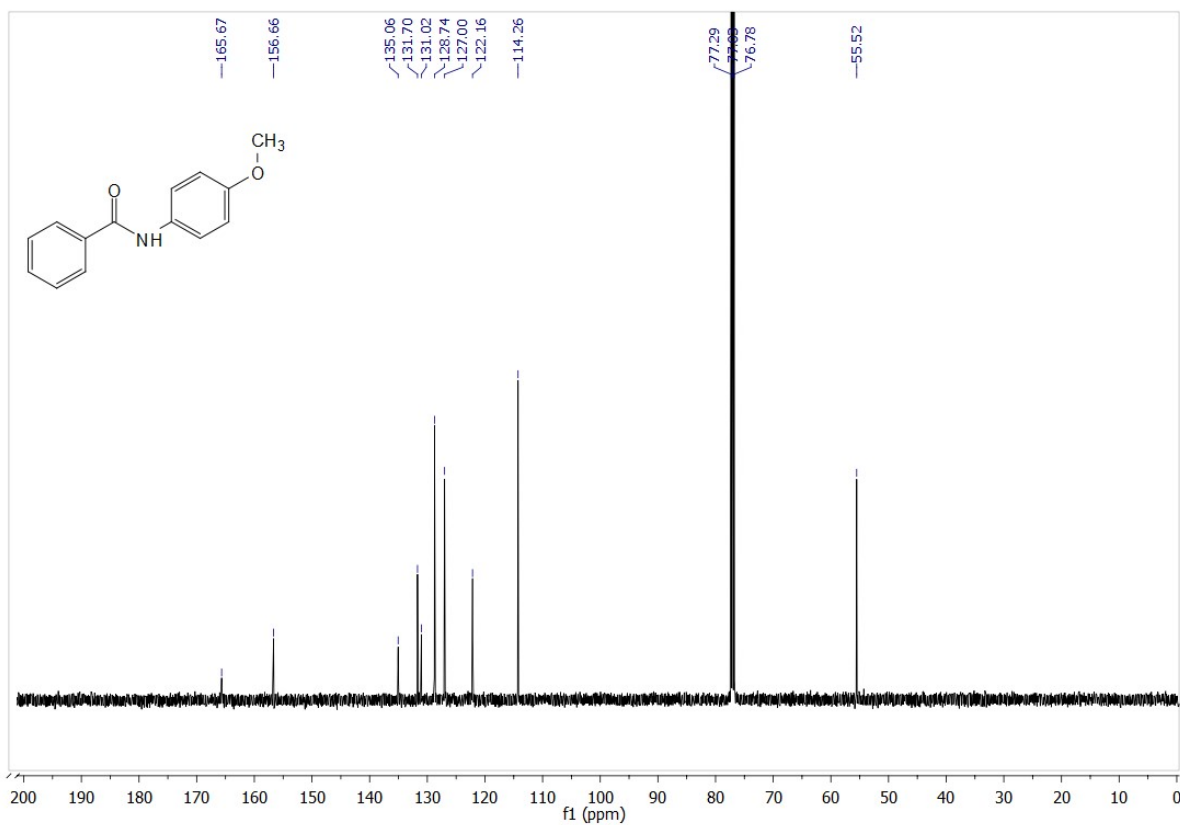
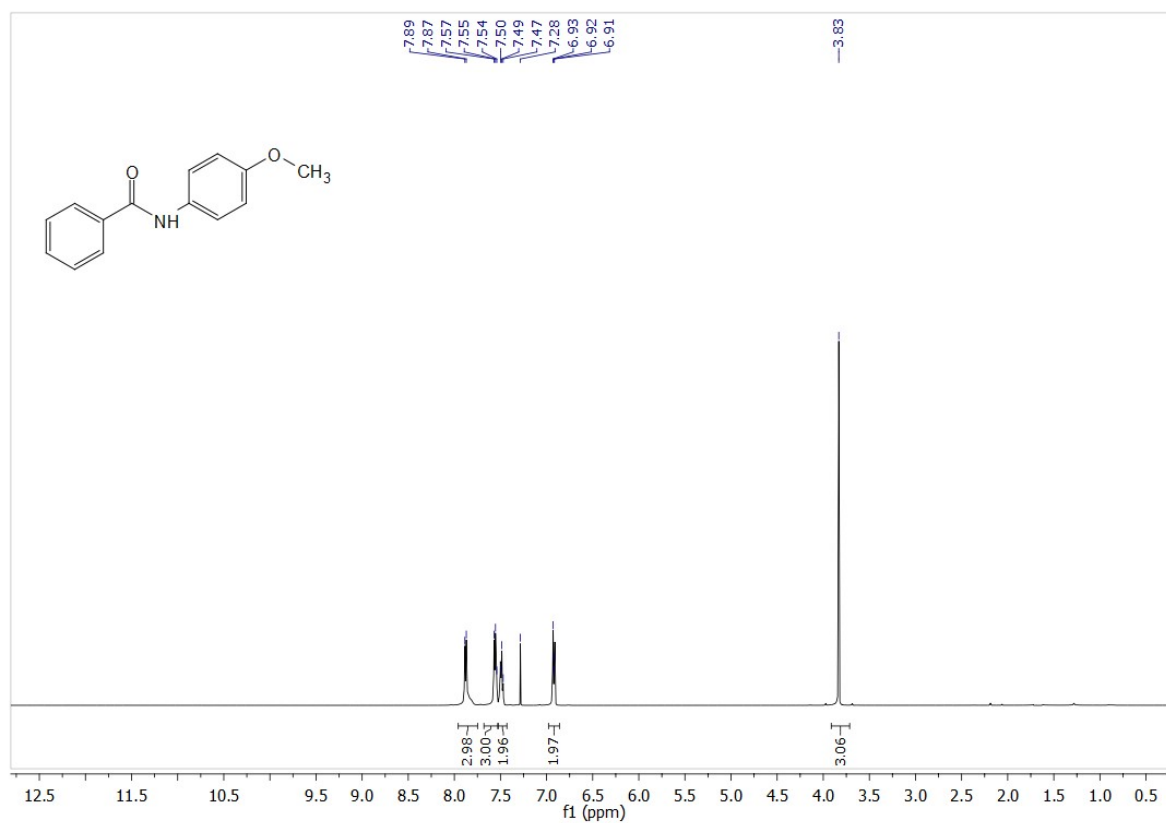
^1H and ^{13}C NMR Spectra of *N*-phenylbenzamide (3y)



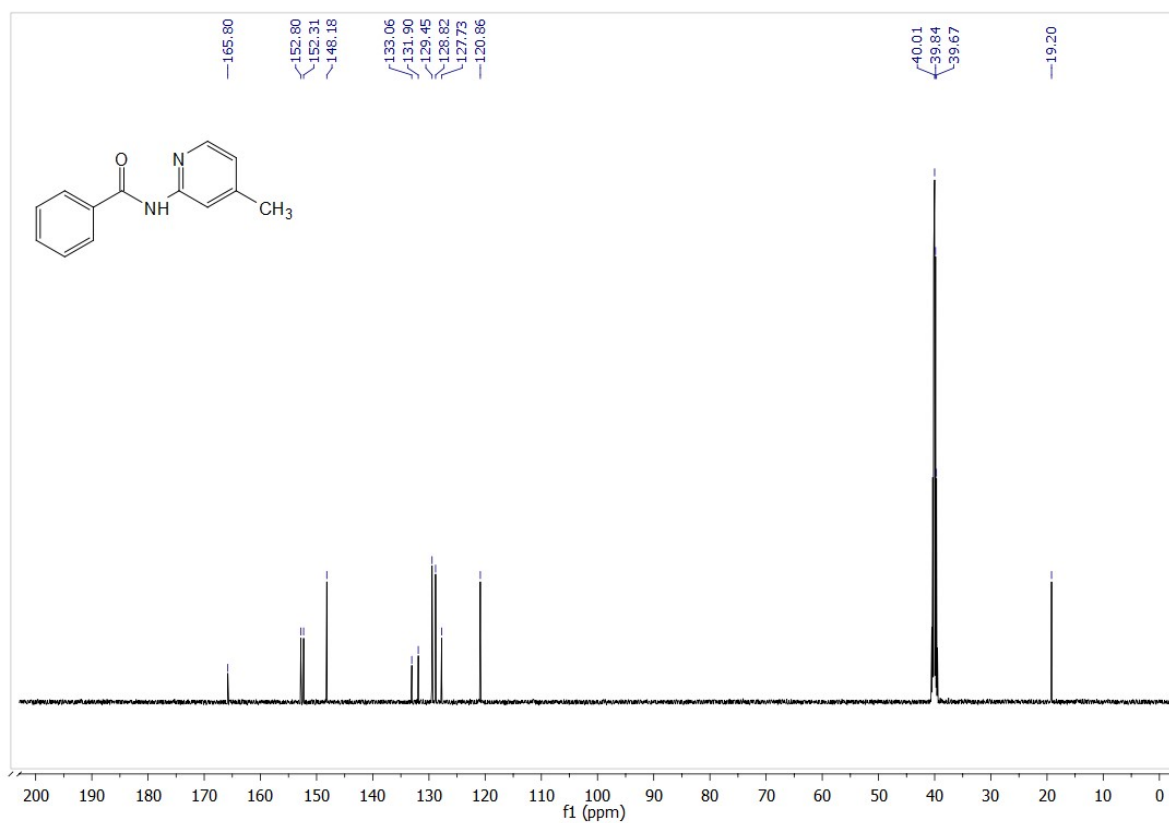
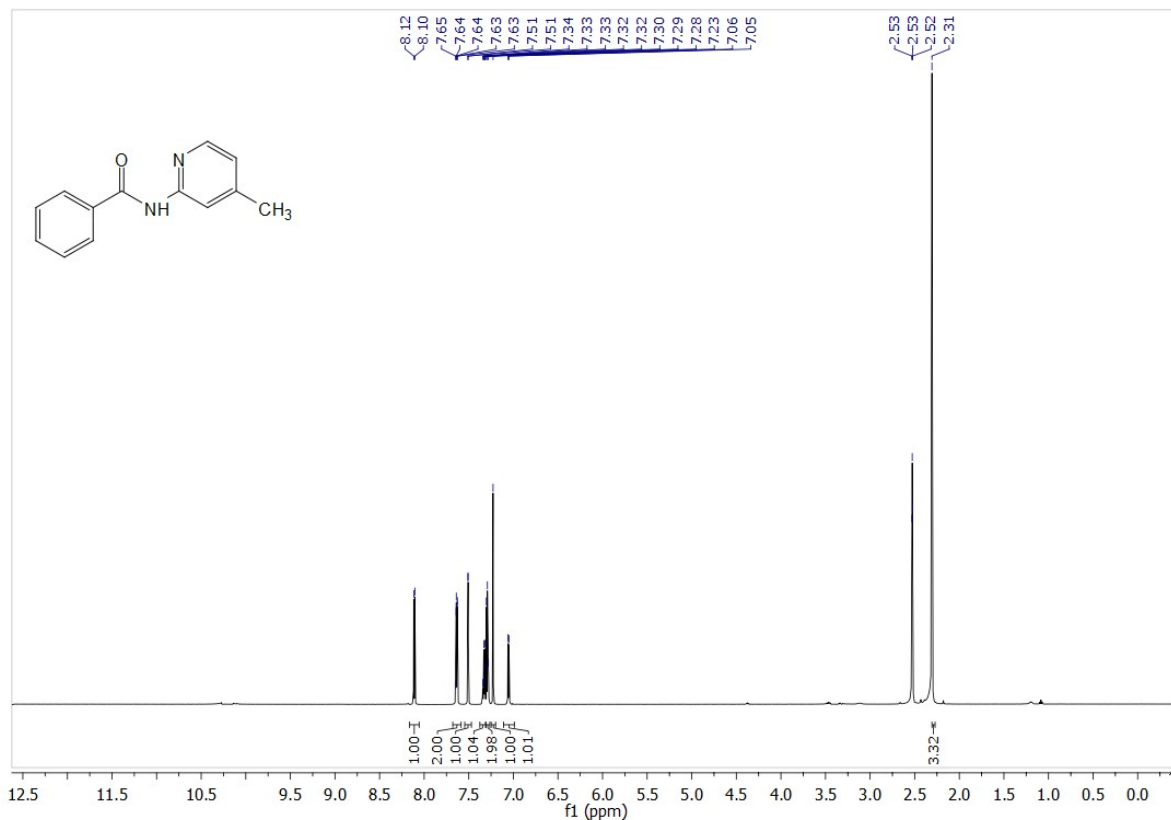
¹H and ¹³C NMR Spectra of *N*-(2-ethylphenyl)benzamide (3z)



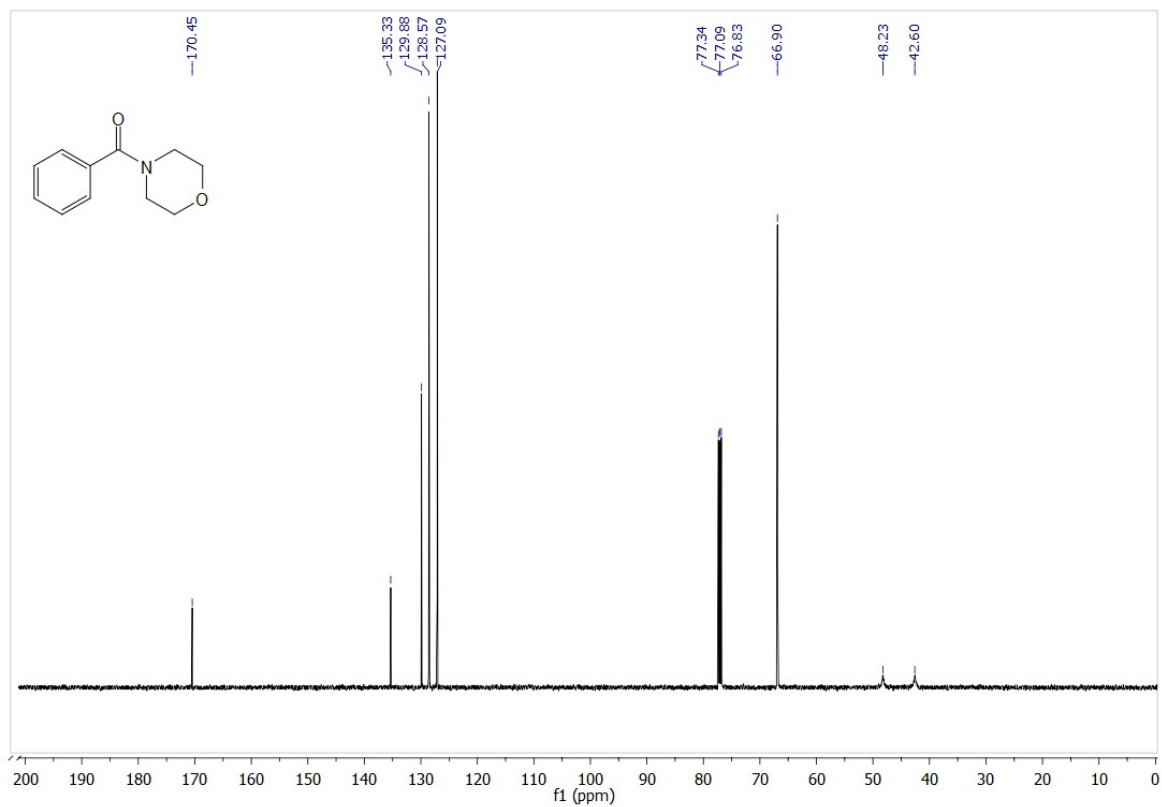
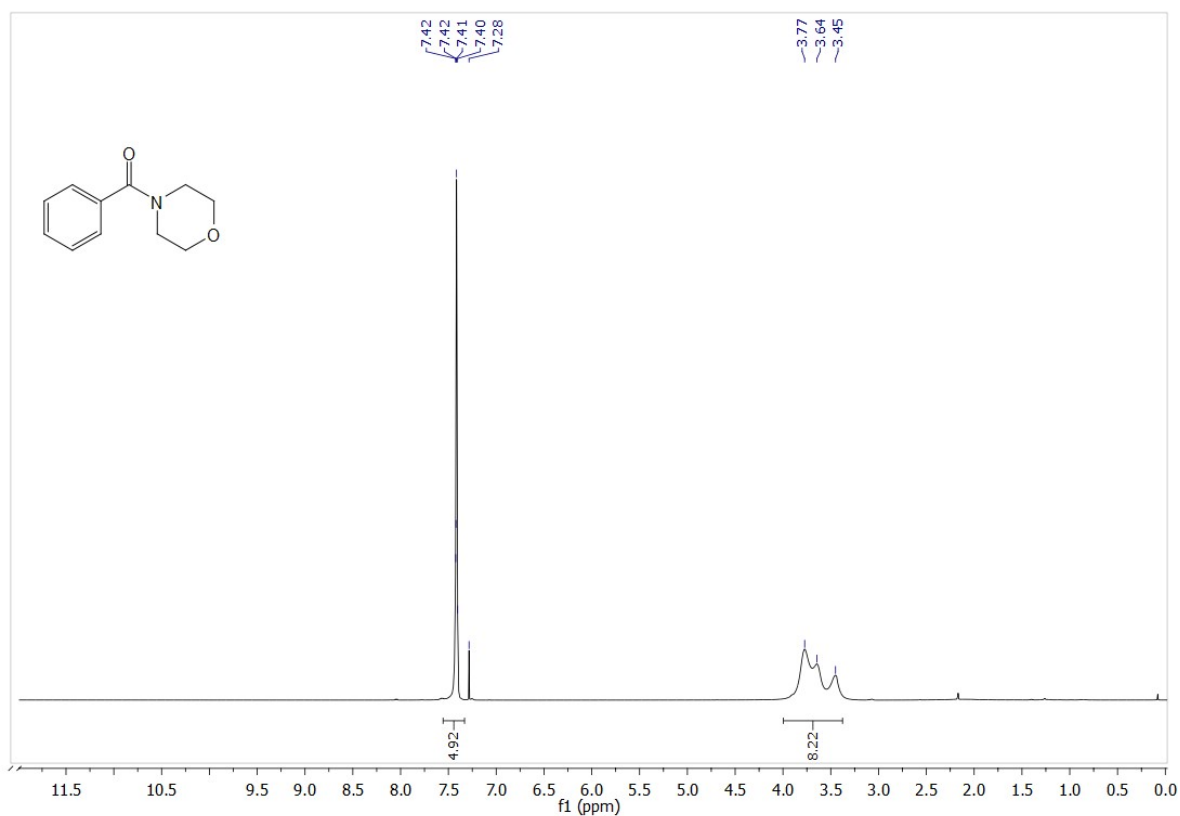
¹H and ¹³C NMR Spectra of *N*-(4-methoxyphenyl)benzamide (3aa)



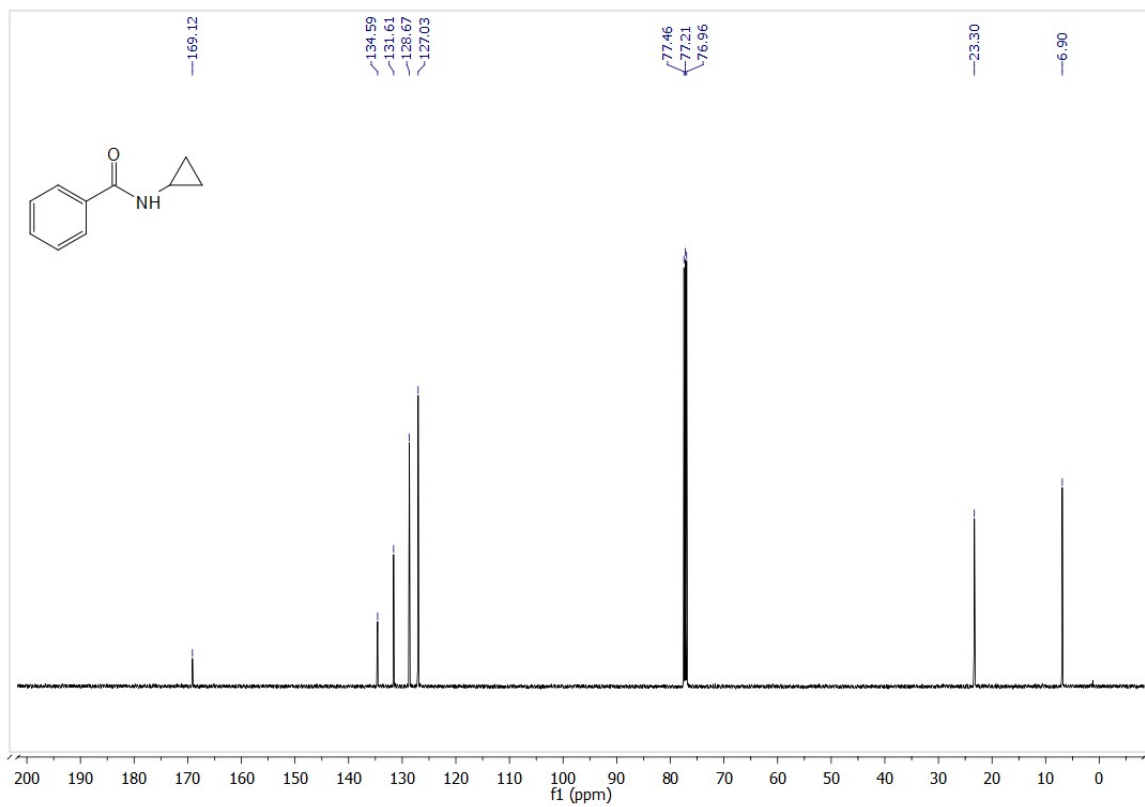
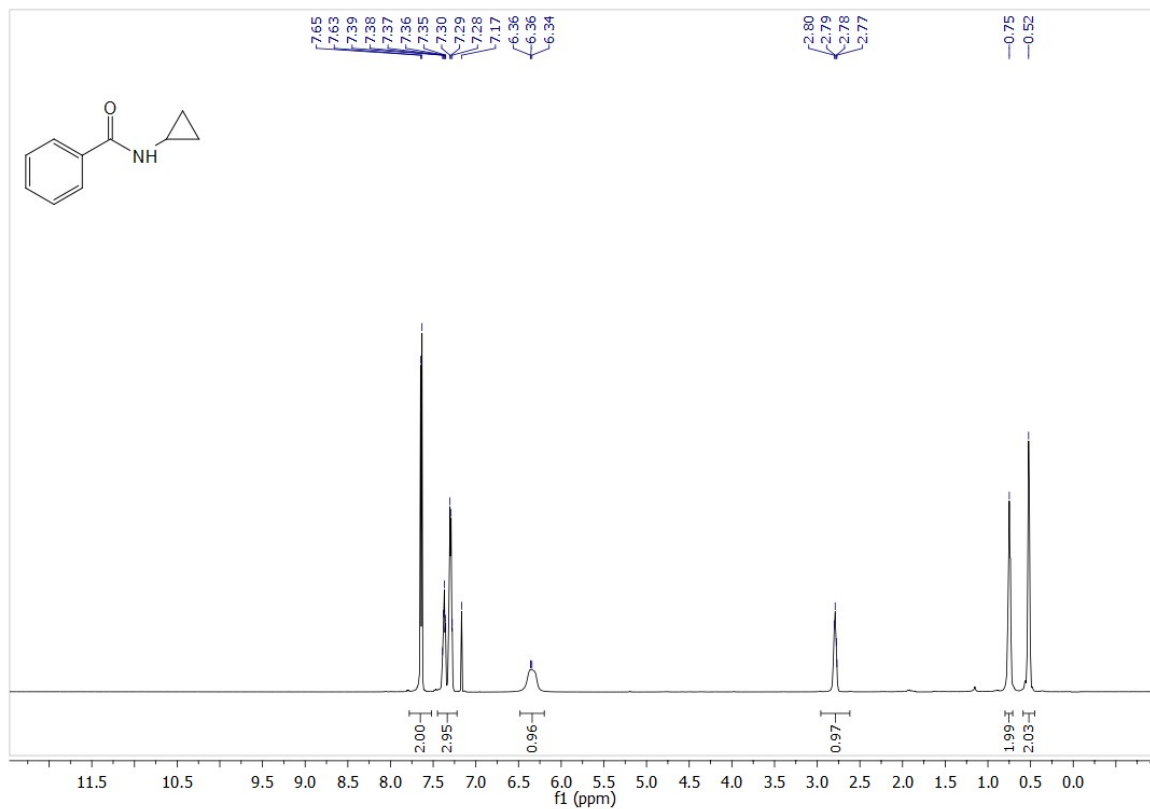
¹H and ¹³C NMR Spectra of *N*-(4-methylpyridin-2-yl)benzamide (3ab)



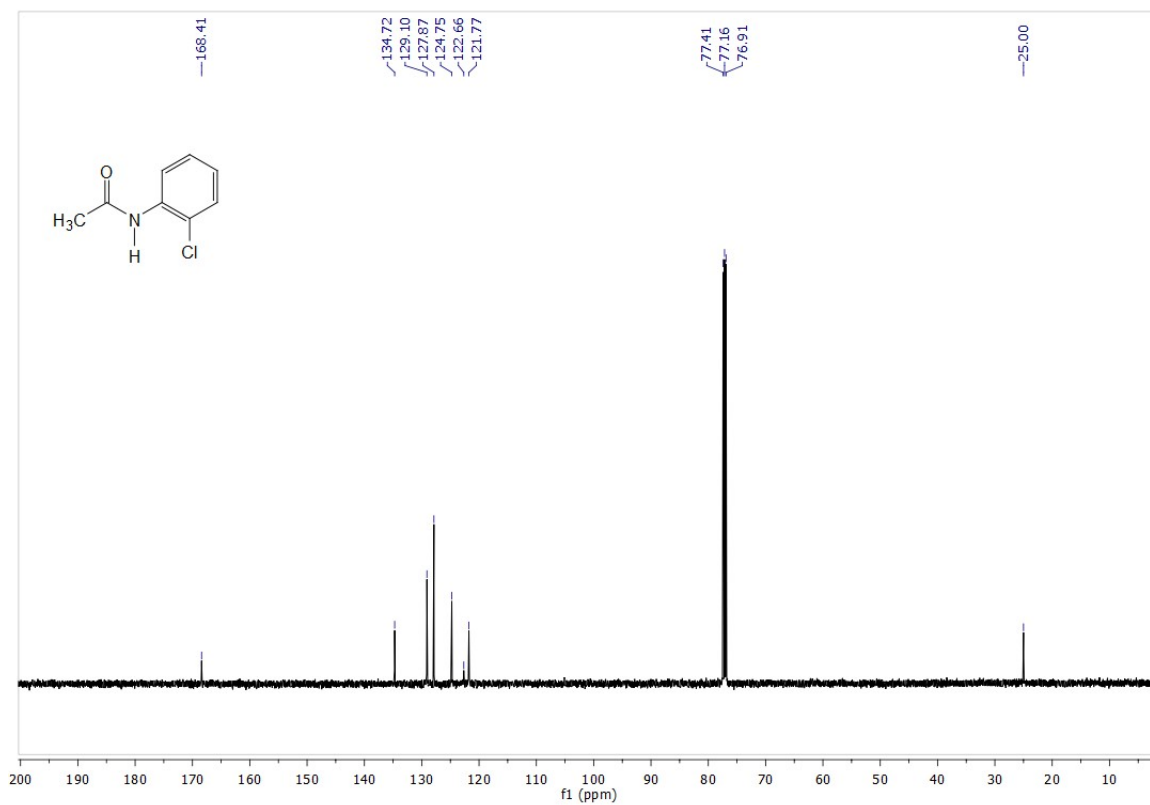
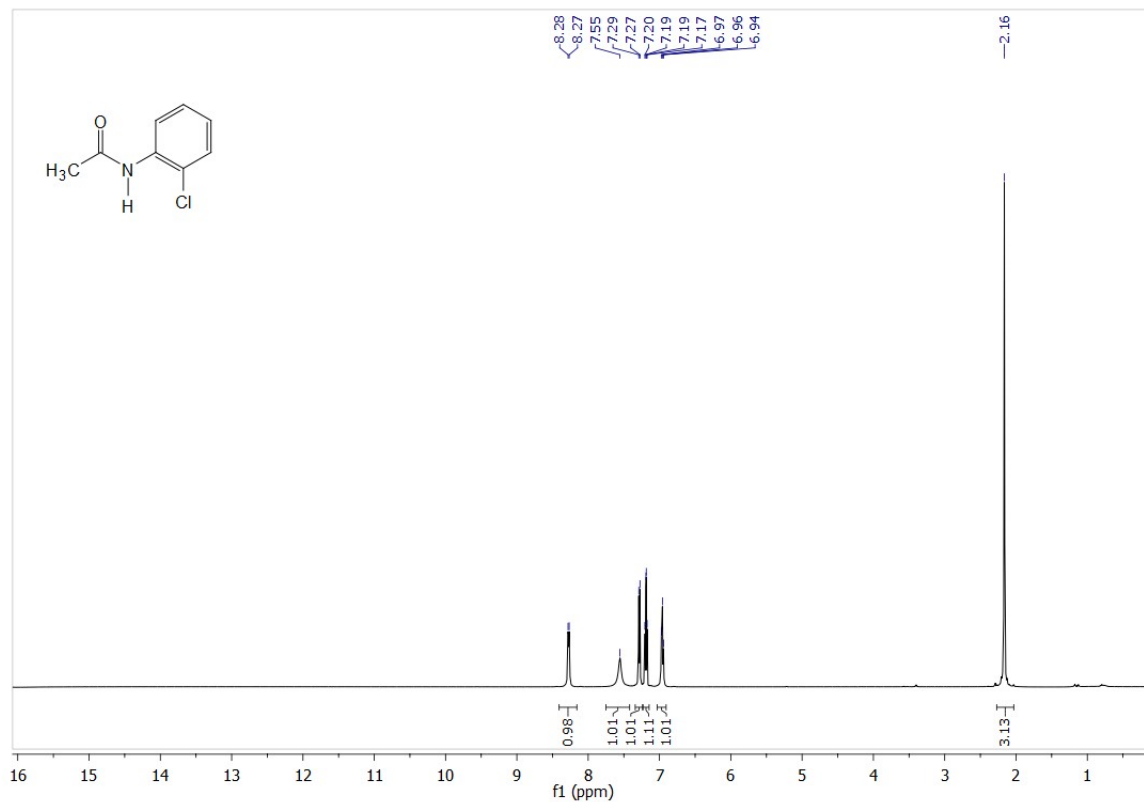
¹H and ¹³C NMR Spectra of morpholino(phenyl)methanone (3ac).



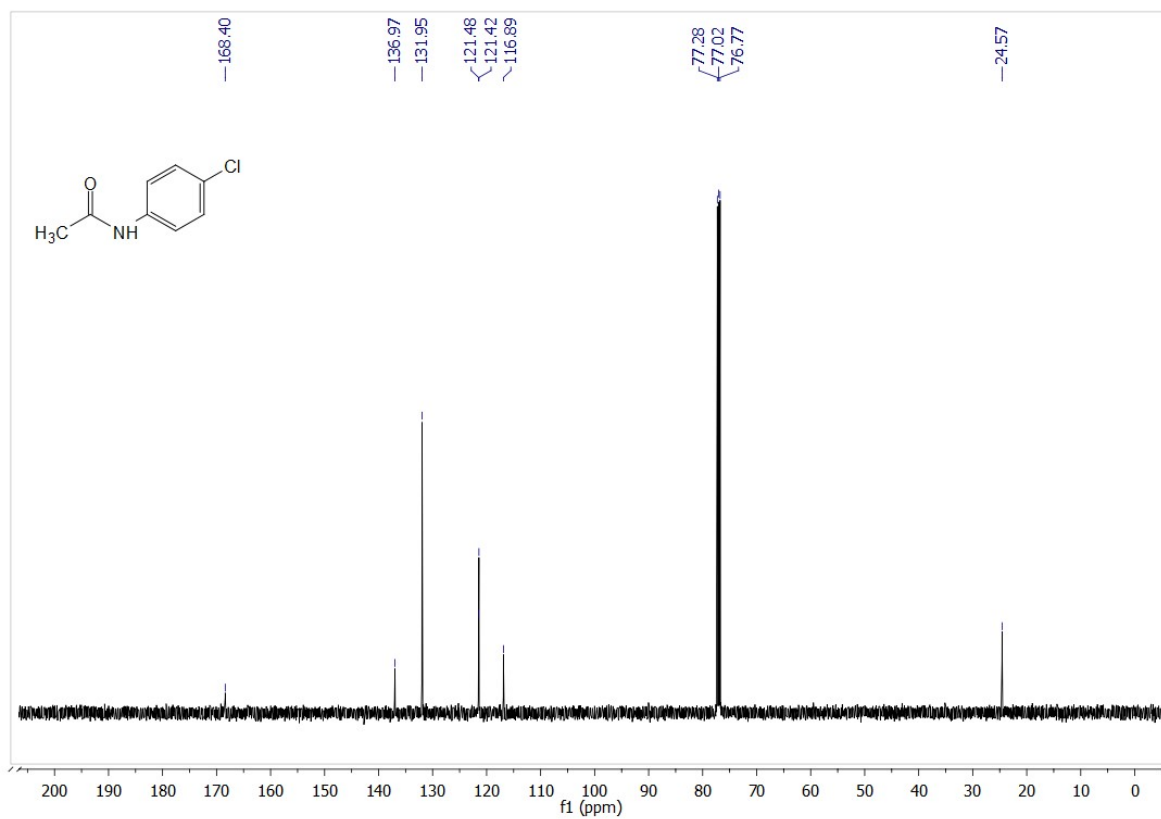
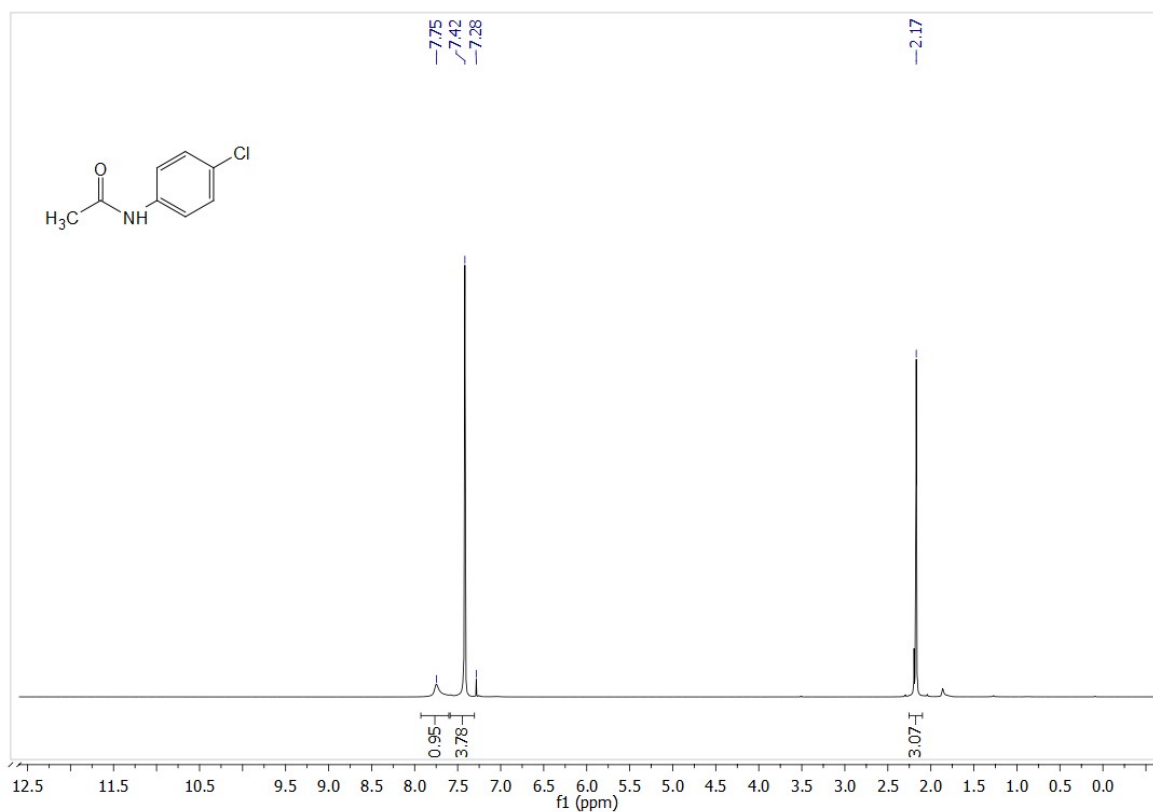
¹H and ¹³C NMR Spectra of *N*-cyclopropylbenzamide (3ad)



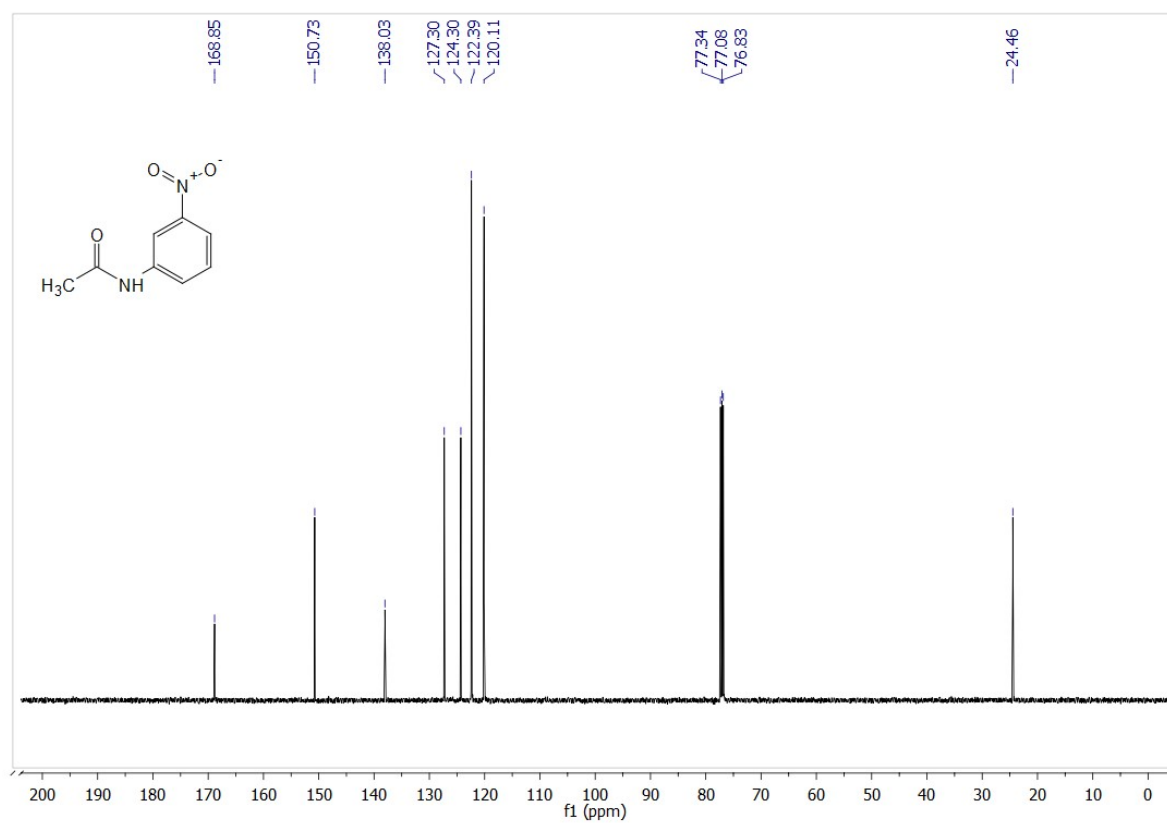
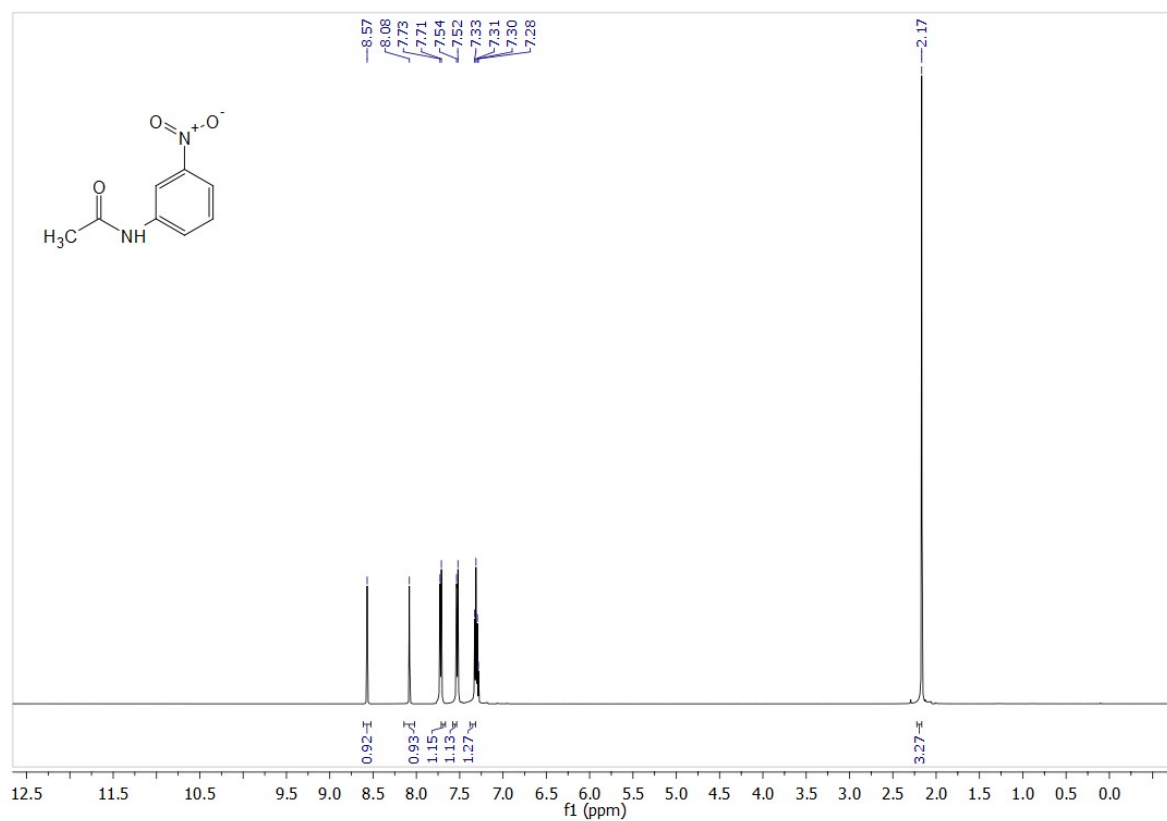
¹H and ¹³C NMR Spectra of *N*-(2-chlorophenyl)acetamide (3ae)



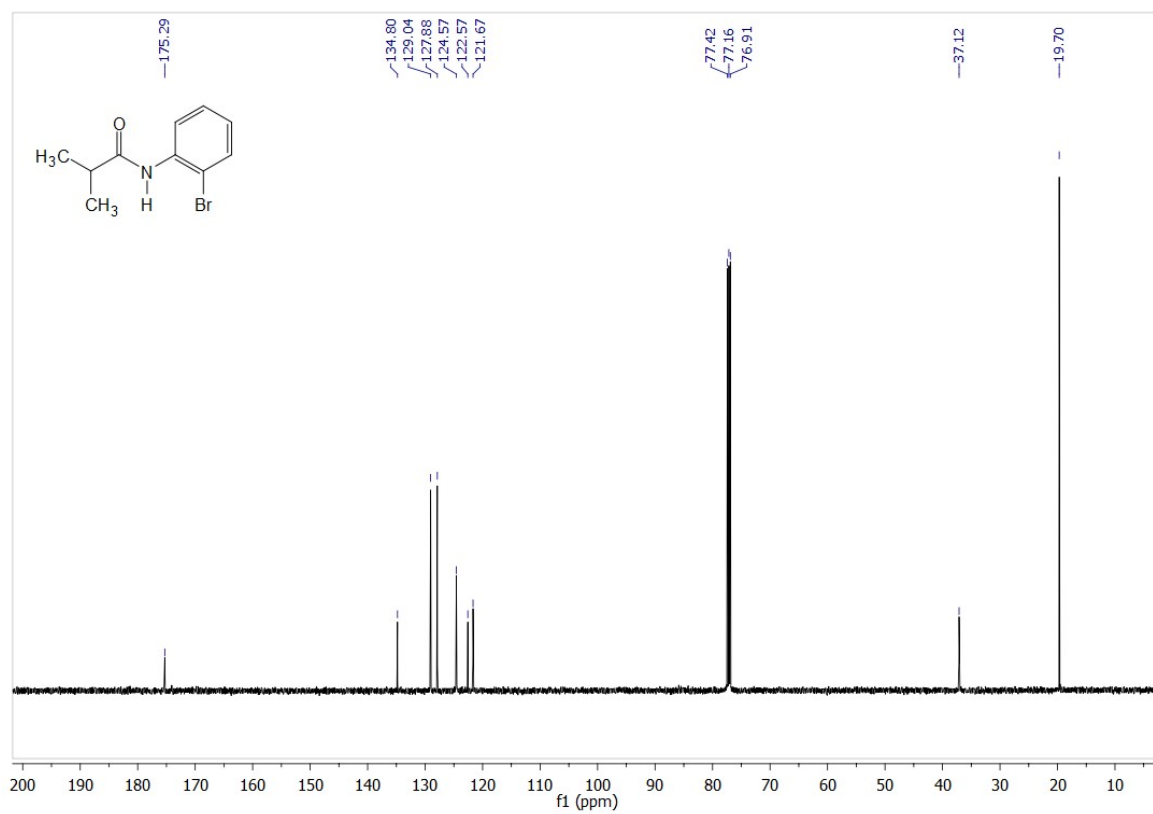
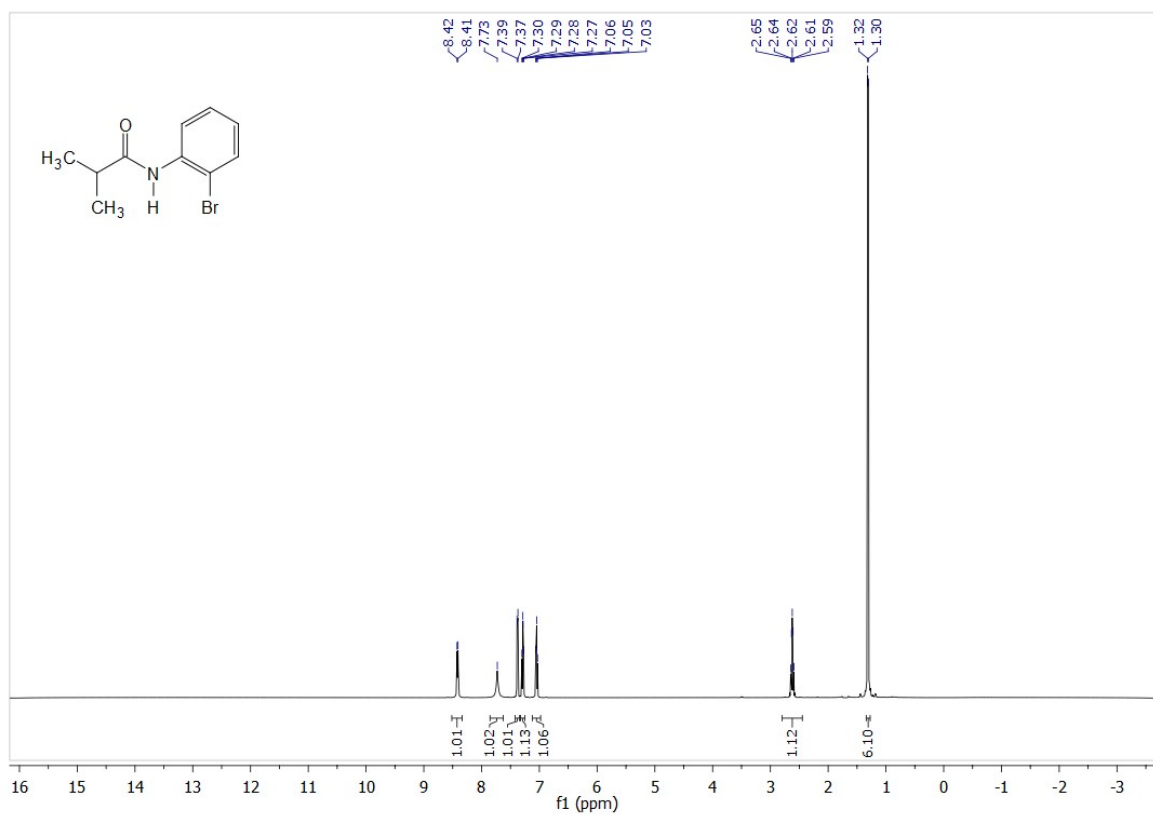
¹H and ¹³C NMR Spectra of *N*-(4-chlorophenyl)acetamide (3af)



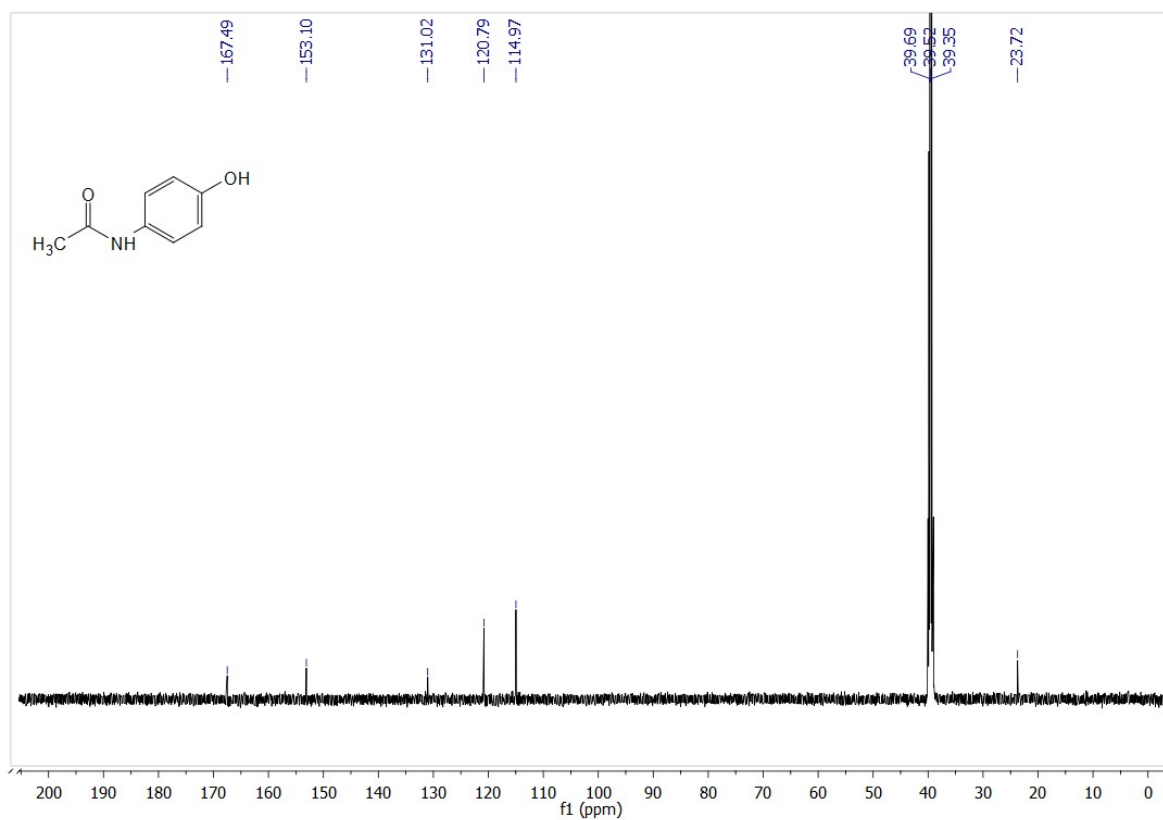
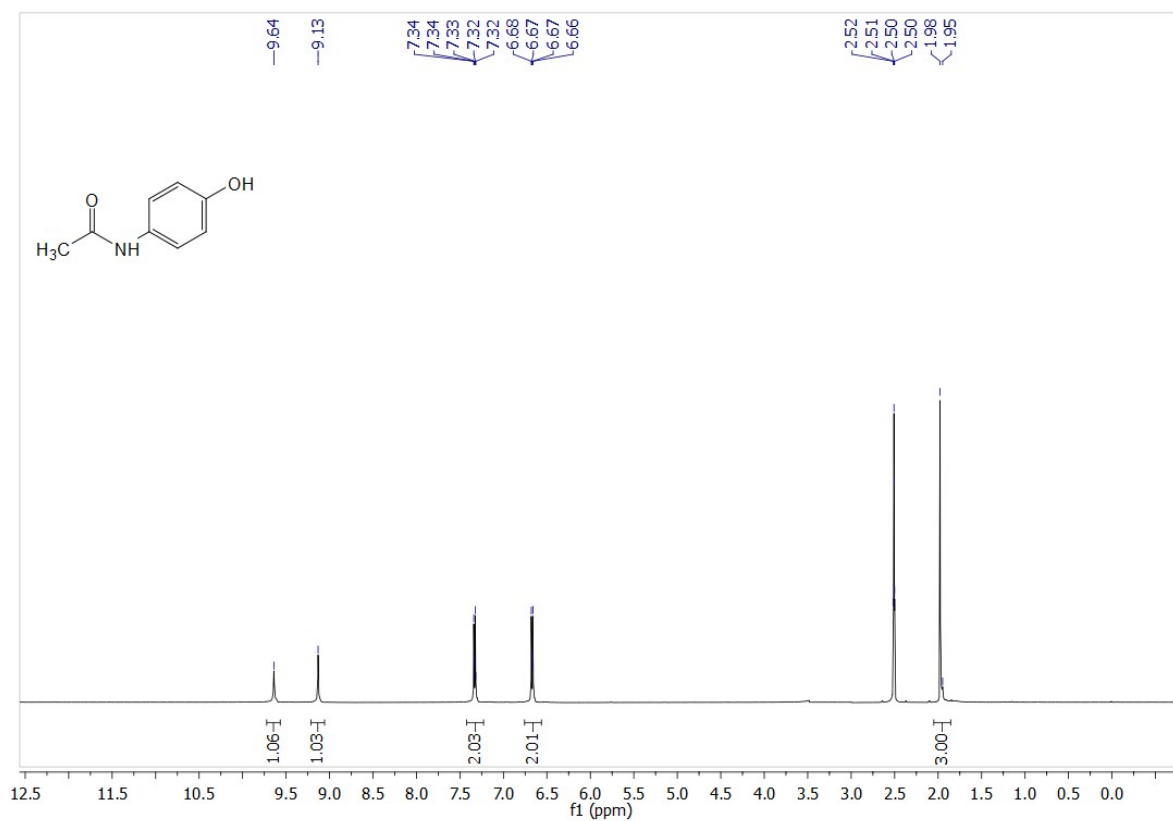
¹H and ¹³C NMR Spectra of *N*-(3-nitrophenyl)acetamide (3ag)



^1H and ^{13}C NMR Spectra of *N*-(2-bromophenyl)isobutyramide (3ah)



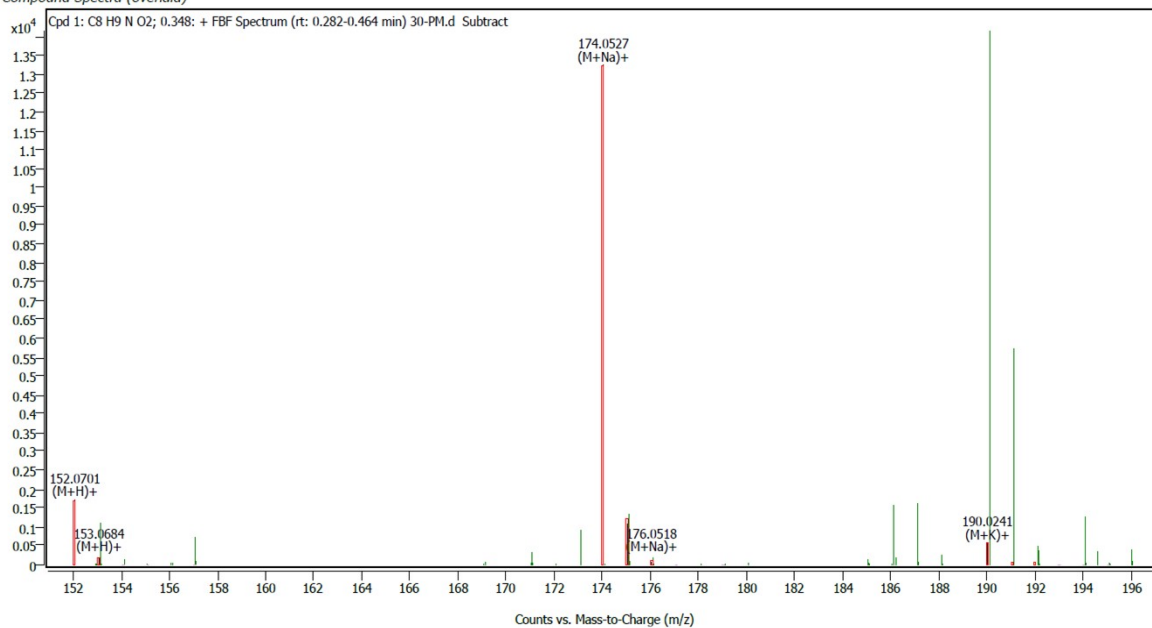
¹H and ¹³C NMR Spectra of *N*-(4-hydroxyphenyl)acetamide (3ai)



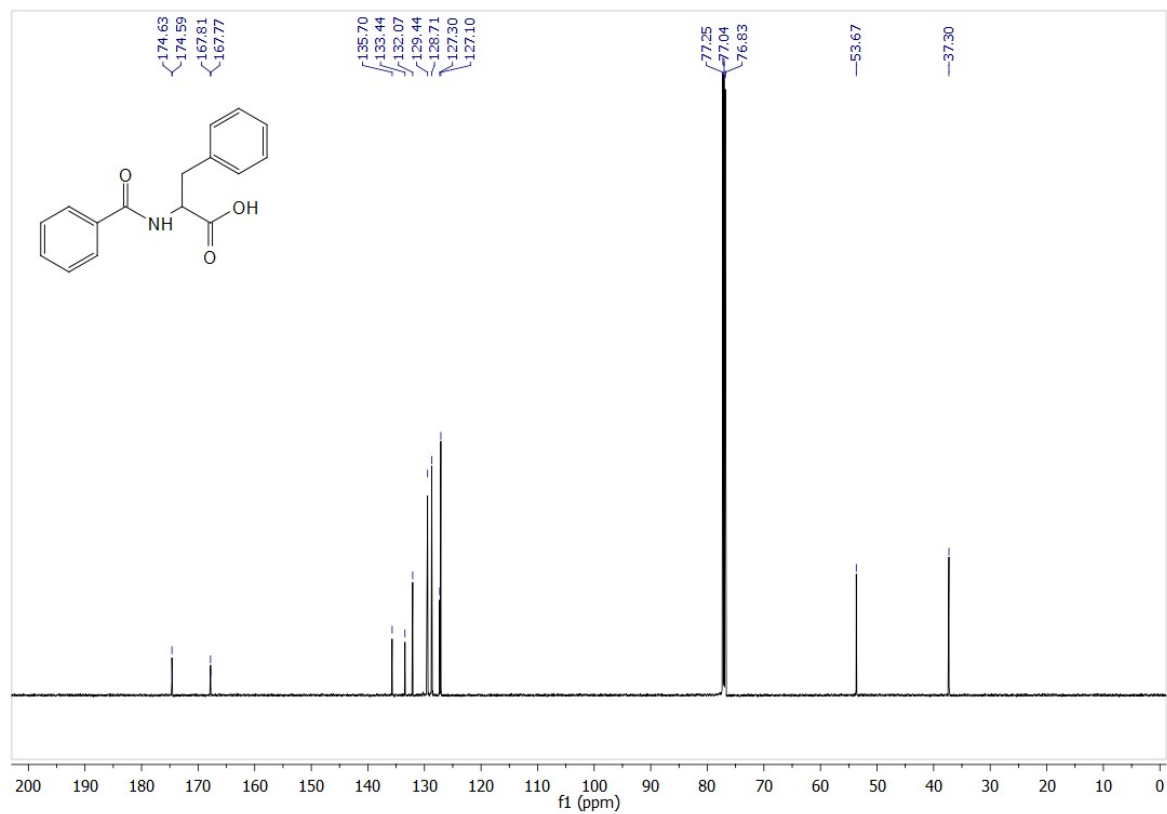
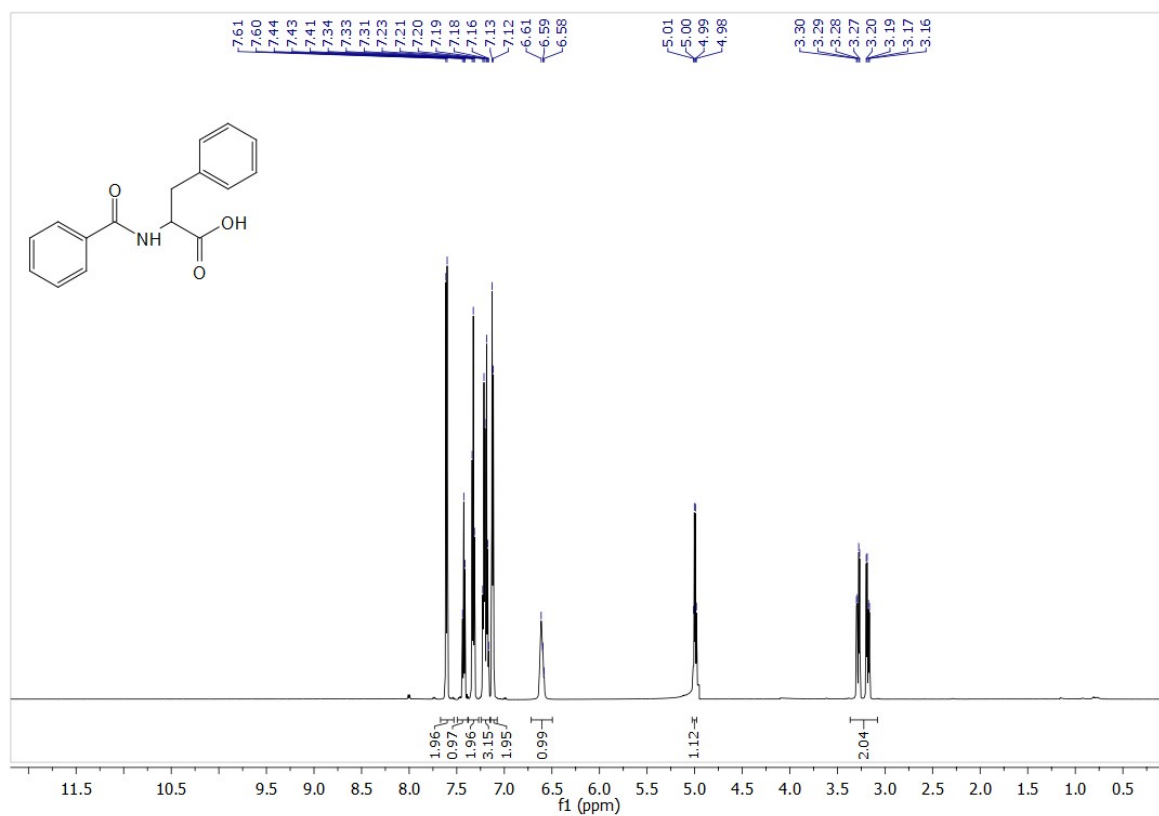
Mass spectra of *N*-(4-hydroxyphenyl)acetamide (3ai)

Cpd. 1: C₈ H₉ N O₂

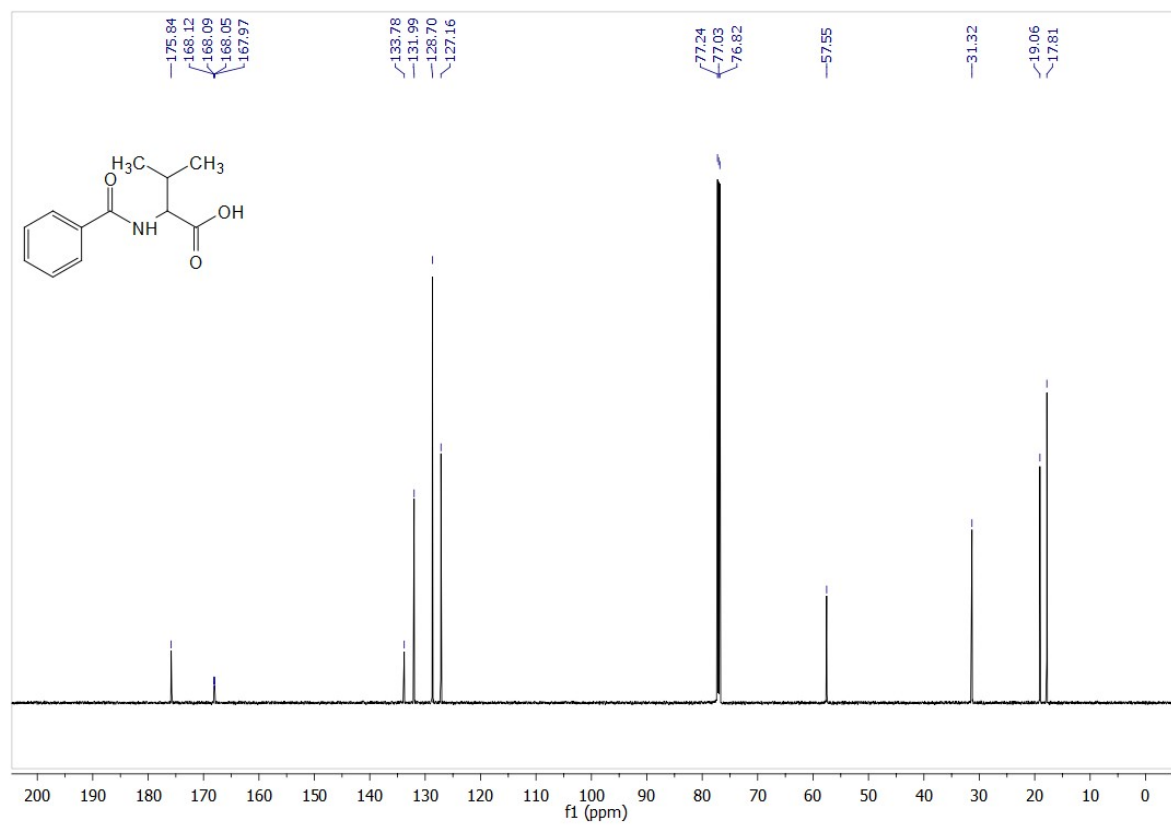
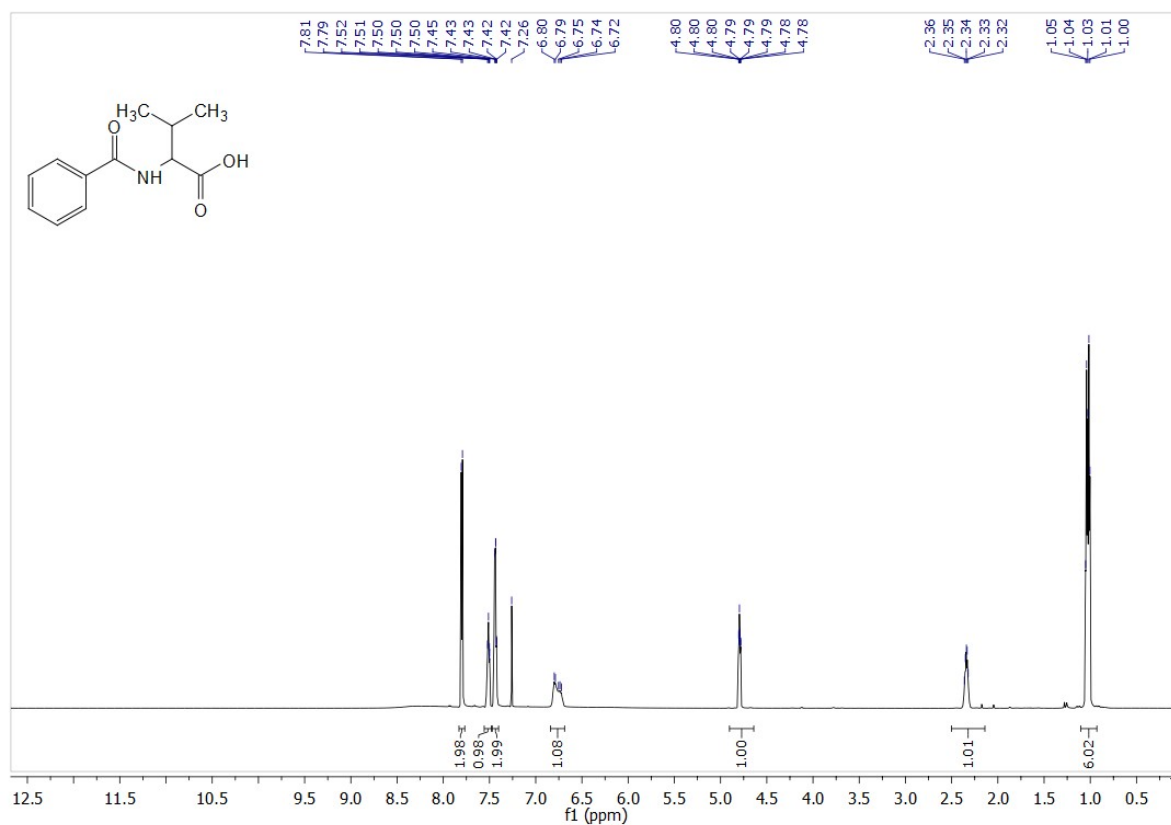
Compound Spectra (overlaid)



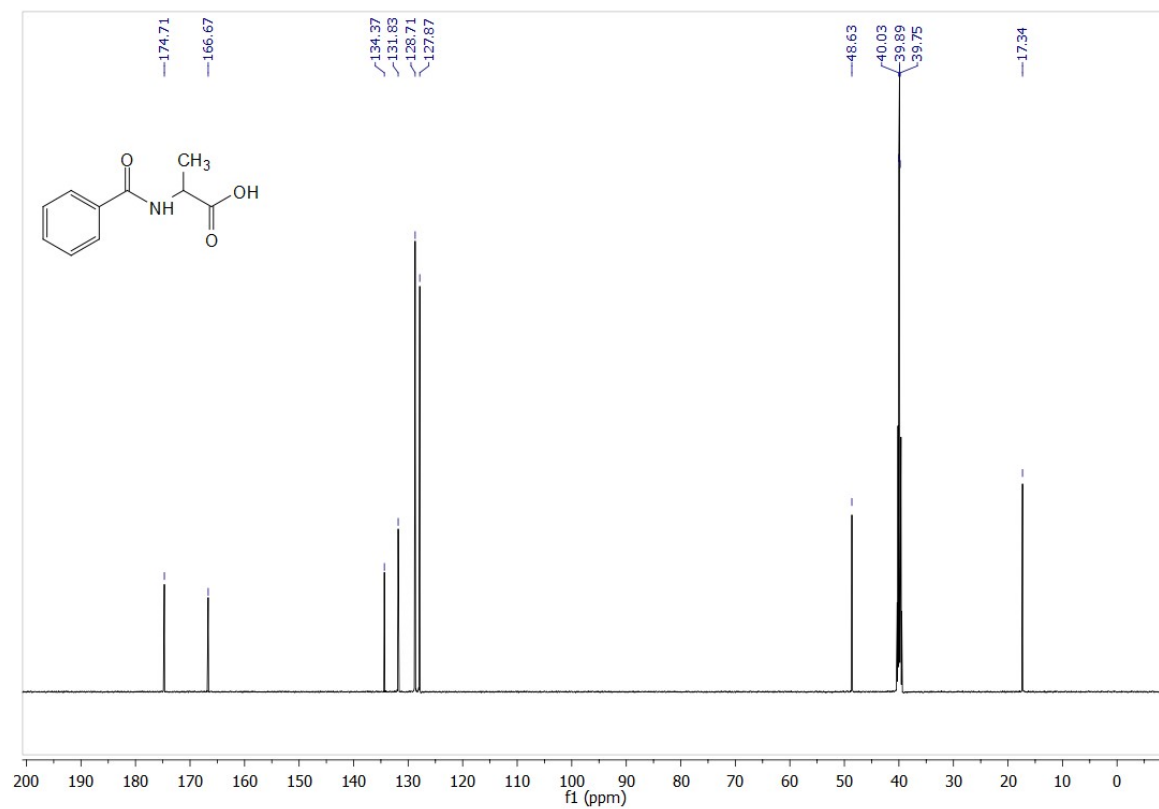
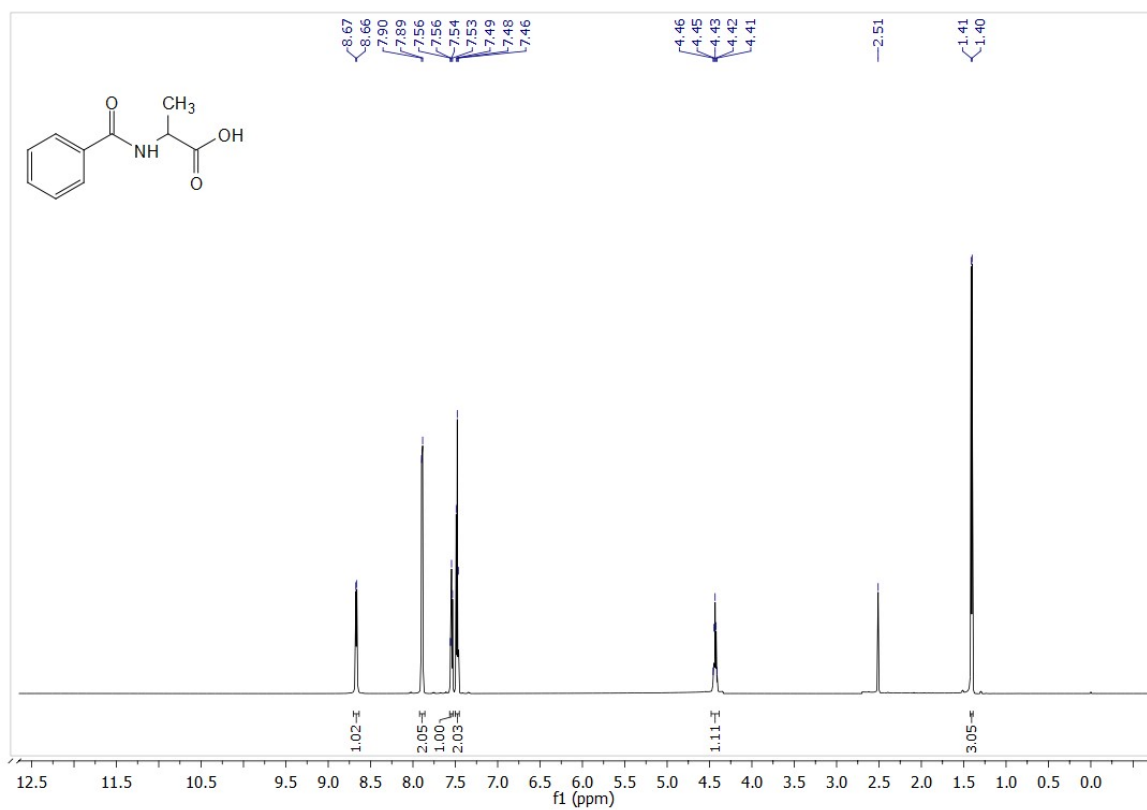
¹H and ¹³C NMR Spectra of benzoylphenylalanine (6a)



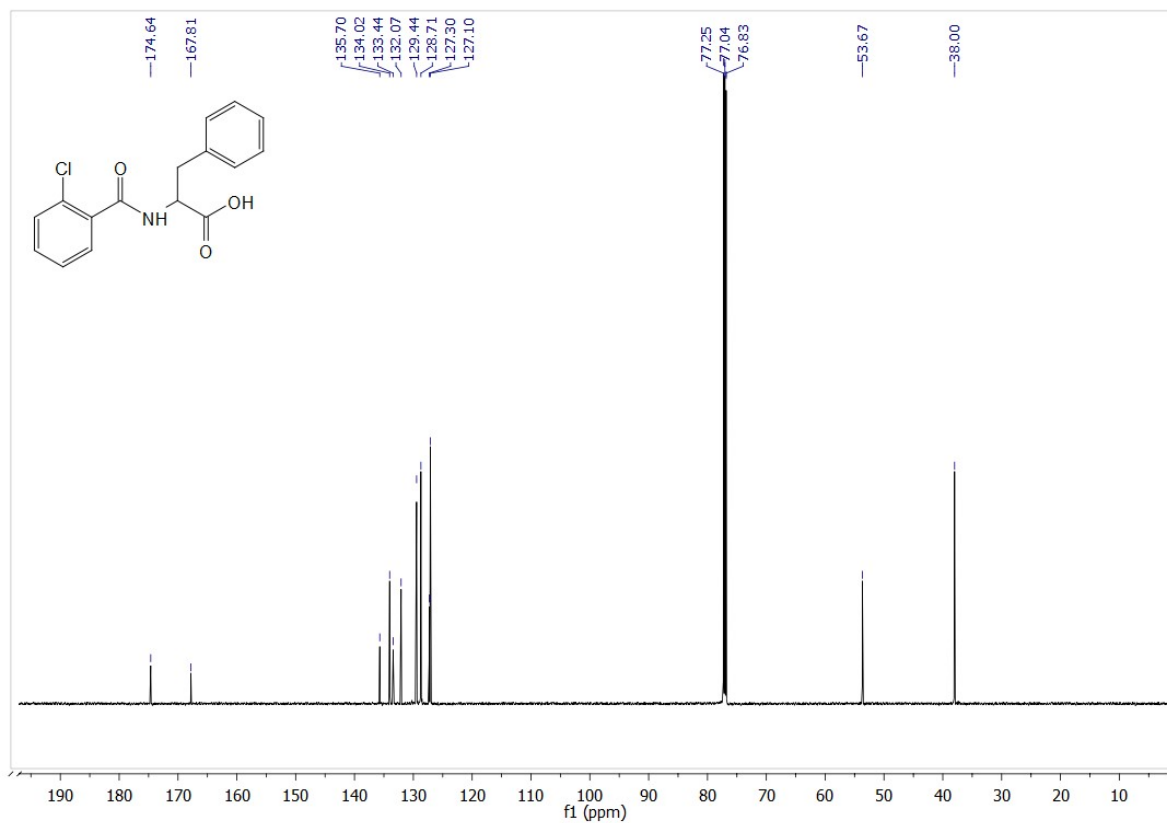
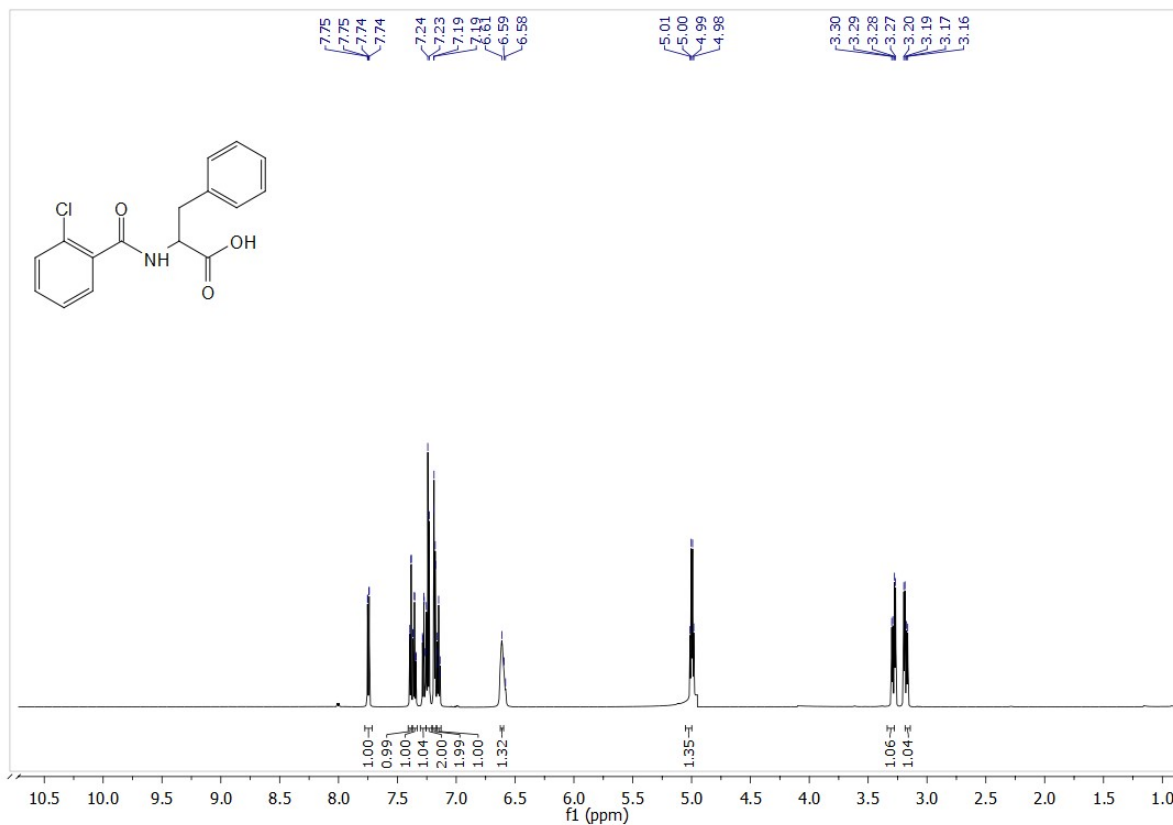
¹H and ¹³C NMR Spectra of benzoylvaline (6b)



¹H and ¹³C NMR Spectra of benzoylalanine (6c)



^1H and ^{13}C NMR Spectra of (2-chlorobenzoyl)phenylalanine (6d)



¹H and ¹³C NMR Spectra of (2-chlorobenzoyl)alanine (6e)

