

Supplementary Information

Visible-Light- Induced Synthesis of Amides from Aldehydes and O-Benzoyl Hydroxylamines Catalyzed by Copper

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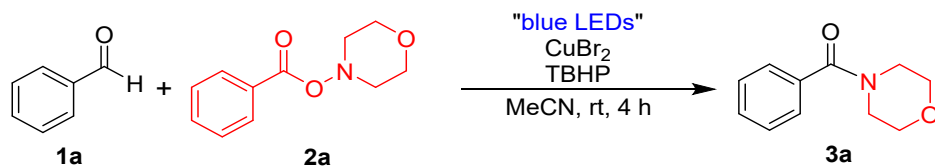
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1. General Information

All chemicals and solvents were purchased from Sigma-Aldrich (St. Louis, MO, USA), Tokyo Chemical Industry (Tokyo, Japan), and Alfa Aesar (Ward Hill, MA, USA) and were used without any purification. Reaction progress was analyzed by thin-layer chromatography (TLC) using silica gel 60 F₂₅₄ pre-coated aluminum plate from Merck and TLC spots were observed under UV light (254 nm) exposure. Flash chromatography was carried out using 230–400 mesh silica gel and analytical grade solvents. Stuart SMP10 Melting Point Apparatus was used to record melting points of products. Structure elucidation by NMR (¹H and ¹³C NMR) was performed on a Bruker AVANCE III HD-400 MHz Fourier transform NMR spectrometer at the Future Energy Convergence Core Center (FECC). The chemical shifts were reported in δ units (ppm) relative to the residual protonated solvent resonance, the coupling constants (*J*) quoted in Hz, and multiplicity of signals was abbreviated as follows: singlet (s); doublet (d); doublet of doublet (dd); triplet (t); multiplet (m).

2. Screening of reaction conditions for the synthesis of amides.

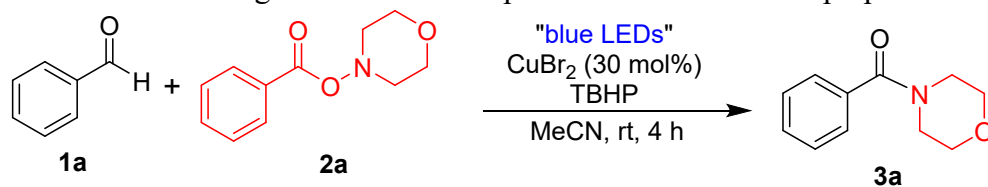
Table S1. Screening of amount of CuBr₂ for the preparation of amides.



Entry	CuBr ₂	Yield ^b (%)
1	CuBr ₂ (0.01 equiv.)	29
2	CuBr ₂ (0.05 equiv.)	44
3	CuBr ₂ (0.1 equiv.)	67
4	CuBr ₂ (0.2 equiv.)	84
5	CuBr ₂ (0.3 equiv.)	90
6	CuBr ₂ (0.5 equiv.)	90

^a Reaction conditions: compound **1a** (1.0 mmol), compound **2a** (0.5 mmol), CuBr₂, TBHP (1.5 mmol), MeCN (2 mL), irradiation by blue LEDs (5W x 2 bulbs), 4 h, open air. ^b Isolated yield after purification by flash column chromatography.

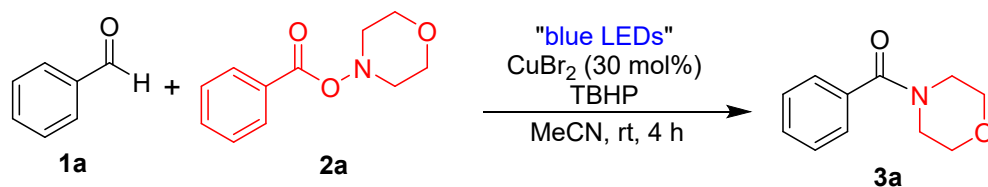
Table S2. Screening of amount of morpholino benzoate for the preparation of amides.



Entry	Morpholino benzoate	Yield ^b (%)
1	Morpholino benzoate (2.0 equiv)	45
2	Morpholino benzoate (1.5 equiv)	52
3	Morpholino benzoate (1.25 equiv)	59
4	Morpholino benzoate (1.0 equiv)	73
5	Morpholino benzoate (0.8 equiv)	81
6	Morpholino benzoate (0.67 equiv.)	85
7	Morpholino benzoate (0.5 equiv.)	90
8	Morpholino benzoate (0.33 equiv.)	90

^a Reaction conditions: compound **1a** (1.0 mmol), compound **2a**, CuBr₂ (0.15 mmol), TBHP (1.5 mmol), MeCN (2 mL), 4 h, open air. ^b Isolated yield after purification by flash column chromatography.

Table S3. Screening of amount of *tert*-butyl hydroperoxide for the preparation of amides.



Entry	<i>tert</i> -Butyl hydroperoxide (TBHP)	Yield ^b (%)
1	<i>tert</i> -Butyl hydroperoxide (0.5 equiv)	62
2	<i>tert</i> -Butyl hydroperoxide (1.0 equiv)	72
3	<i>tert</i> -Butyl hydroperoxide (1.2 equiv)	75
4	<i>tert</i> -Butyl hydroperoxide (1.5 equiv)	81
5	<i>tert</i> -Butyl hydroperoxide (2.0 equiv)	88
6	<i>tert</i> -Butyl hydroperoxide (3.0 equiv)	90
7	<i>tert</i> -Butyl hydroperoxide (4.0 equiv.)	90

^a Reaction conditions: compound **1a** (1.0 mmol), compound **2a** (0.5 mmol), CuBr₂ (0.15 mmol), TBHP, MeCN (2 mL), 4 h, open air. ^b Isolated yield after purification by flash column chromatography.

3. General procedure

Preparation of O-Benzoyl Hydroxylamines^[S1]

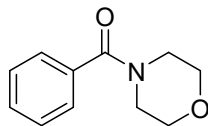
To a round-bottomed flask was added benzoyl peroxide (2.90 g, 12 mmol), K₂HPO₄ (3.13 g, 18 mmol), and DMF (25 mL). The suspension was stirred for 15 min. Morpholine (1.25 g, 14.4 mmol) was added to the reaction mixture in one portion and the suspension was stirred for 2 h at room temperature. After the reaction finished, deionized water (25 mL) was added to the mixture and stirred for 2 min to dissolve all solids. The reaction mixture was then extracted with EtOAc (25 mL). The organic layer was collected and washed with saturated NaHCO₃ (25 mL x 2), DI water (100 mL), and brine (25 mL), dried over anhydrous Na₂SO₄, and concentrated under reduced pressure. The obtained crude products were purified with column chromatography using *n*-hexane-EtOAc (10:1) as the eluent.

Preparation of amides

Benzaldehyde **1a** (106 mg, 1.0 mmol), morpholino benzoate **2a** (103 mg, 0.5 mmol), and CuBr₂ (33 mg, 0.15 mmol) were added to acetonitrile (2 mL) and were stirred for 5 min. Then, TBHP solution (70% wt. in H₂O) (193 mg, 1.5 mmol) was added to the reaction mixture. The mixture was stirred under blue LEDs irradiation (5 W ×2) at room temperature for 4 h. The product mixture was quenched in water (25 mL) and extracted with EtOAc (25 mL). The organic layer was collected and dried over anhydrous Na₂SO₄, and the solvent was concentrated under reduced pressure. The crude mixture was purified using flash column chromatography on silica gel with *n*-hexane-EtOAc as eluent to afford the desired product **4a** (86 mg, 90%).

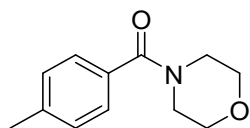
4. Characterization data for amides

Morpholino(phenyl)methanone (**3a**)^[S2]



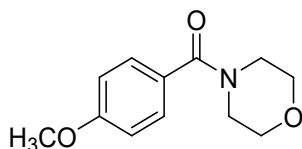
3a was obtained in 90% yield (0.086 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.38 (m, 5H), 3.70-3.45 (m, 8H); ¹³C NMR (100 MHz, CDCl₃) δ 170.6, 135.5, 130.0, 128.7 (2C), 127.2 (2C), 67.0 (2C), 48.5, 42.8; HRMS (ESI) m/z (M+H)⁺ calcd for C₁₁H₁₄NO₂⁺ = 192.1020; found 192.1022.

Morpholino(p-tolyl)methanone (**3b**)^[S2]



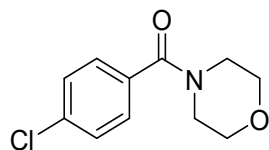
3b was obtained in 73% yield (0.075 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.29 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 3.66 (br s, 8H), 2.35 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.7, 140.1, 132.4, 129.2 (2C), 127.3 (2C), 66.9 (2C), 48.4, 42.8, 21.4; HRMS (ESI) m/z (M+H)⁺ calcd for C₁₂H₁₆NO₂⁺ = 206.1176; found 206.1179.

(4-Methoxyphenyl)(morpholino)methanone (**3c**)^[S2]



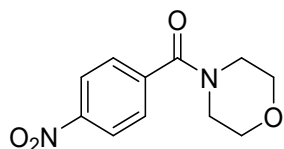
3c was obtained in 79% yield (0.087 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.37 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H), 3.69-3.64 (m, 8H); ¹³C NMR (100 MHz, CDCl₃) δ 170.5, 160.9, 129.3 (2C), 127.4, 113.9 (2C), 66.9 (2C), 55.4, 47.4, 43.6; HRMS (ESI) m/z (M+H)⁺ calcd for C₁₂H₁₆NO₃⁺ = 222.1125; found 222.1126.

(4-Chlorophenyl)(morpholino)methanone (**3d**)^[S2]



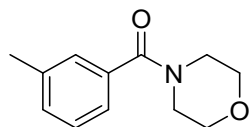
3d was obtained in 88% yield (0.099 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 73-75 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.38-7.32 (m, 4H), 3.67-3.43 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.4, 136.1, 133.7, 128.9 (2C), 128.7 (2C), 66.9 (2C), 48.4, 42.5; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{13}\text{ClNO}_2^+ = 226.0630$; found 226.0633.

Morpholino(4-nitrophenyl)methanone (3e)^[S3]



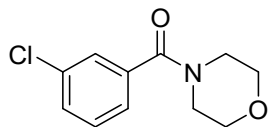
3e was obtained in 92% yield (0.109 g) according to the general procedure (Hexan/EtOAc, 1:2); white solid, m.p. 101-102 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.29 (d, $J = 8.4$ Hz, 2H), 7.59 (d, $J = 8.4$ Hz, 2H), 3.79-3.38 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.1, 148.6, 141.5, 128.3 (2C), 124.0 (2C), 66.8 (2C), 48.1, 42.7; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{13}\text{N}_2\text{O}_4^+ = 237.0870$; found 237.0874.

Morpholino(m-tolyl)methanone (3f)^[S4]



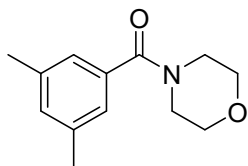
3f was obtained in 75% yield (0.077 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.29-7.25 (m, 1H); 7.22-7.20 (m, 2H); 7.17-7.15 (m, 1H); 3.69-3.45 (m, 8H); 2.36 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.7, 138.6, 135.4, 130.7, 128.5, 127.8, 124.1, 67.0 (2C), 48.3, 42.6, 21.5; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{12}\text{H}_{16}\text{NO}_2^+ = 206.1176$; found 206.1178.

(3-Chlorophenyl)(morpholino)methanone (3g)^[S5]



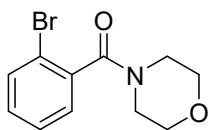
3g was obtained in 93% yield (0.105 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.39 (m, 2H), 7.37-7.33 (m, 1H), 7.28 (d, J = 7.6 Hz, 1H), 3.74-3.43 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.9, 137.2, 134.8, 130.2, 130.1, 127.4, 125.3, 66.9 (2C), 48.4, 42.8; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{13}\text{ClNO}_2^+$ = 226.0630; found 226.0631.

(3,5-Dimethylphenyl)(morpholino)methanone (3h)^[S6]



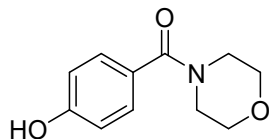
3h was obtained in 68% yield (0.075 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.01 (s, 1H), 6.97 (s, 2H), 3.70-3.42 (m, 8H), 2.99 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.8, 138.3 (2C), 135.3, 131.4, 124.6 (2C), 66.9 (2C), 48.3, 42.5, 21.3 (2C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{13}\text{H}_{18}\text{NO}_2^+$ = 220.1333; found 220.1336.

(2-Bromophenyl)(morpholino)methanone (3i)^[S7]



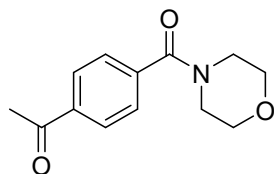
3i was obtained in 84% yield (0.113 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 92-93 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.59-7.57 (m, 1H), 7.40-7.34 (m, 1H), 7.27-7.23 (m, 2H), 3.87-3.80 (m, 1H), 3.78-3.69 (m, 4H), 3.60-3.55 (m, 1H), 3.31-3.18 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.9, 137.7, 132.9, 130.6, 127.9, 127.8, 119.3, 66.9, 66.8, 47.3, 42.2; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{13}\text{BrNO}_2^+$ = 270.0125; found 270.0129.

(4-hydroxyphenyl)(morpholino)methanone (3j)^[S8]



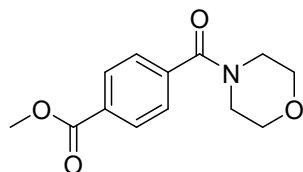
3j was obtained in 75% yield (0.077 g) according to the general procedure (Hexan/EtOAc, 1:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 8.44 (s, 1H), 7.24 (d, J = 8.8 Hz, 2H), 6.74 (d, J = 8.8 Hz, 2H), 3.81-3.56 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.6, 158.9, 129.3 (2C), 125.6, 115.7 (2C), 66.9 (2C), 49.1, 43.2; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{14}\text{NO}_3^+ = 208.0974$; found 208.0971.

1-(4-(morpholine-4-carbonyl)phenyl)ethan-1-one (**3k**)^[S9]



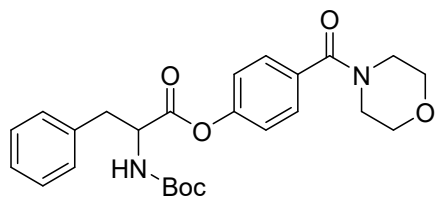
3k was obtained in 83% yield (0.099 g) according to the general procedure (Hexan/EtOAc, 1:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, J = 8.4 Hz, 2H), 7.49 (d, J = 8.4 Hz, 2H), 3.77-3.38 (m, 8H), 2.60 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.3, 169.4, 139.8, 138.0, 128.7 (2C), 127.4 (2C), 66.9 (2C), 48.2, 42.6, 26.8; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{13}\text{H}_{16}\text{NO}_3^+ = 234.1130$; found 234.1128.

Methyl 4-(morpholine-4-carbonyl)benzoate (**3l**)^[S10]



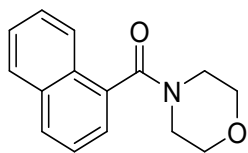
3l was obtained in 81% yield (0.101 g) according to the general procedure (Hexan/EtOAc, 1:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 8.09 (d, J = 8.8 Hz, 2H), 7.47 (d, J = 8.8 Hz, 2H), 3.92 (s, 3H), 3.82-3.39 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.5, 166.4, 139.7, 131.5, 130.0 (2C), 127.2 (2C), 66.9 (2C), 52.5, 48.2, 42.6; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{16}\text{H}_{16}\text{NO}_4^+ = 250.1079$; found 250.1078.

4-(morpholine-4-carbonyl)phenyl (tert-butoxycarbonyl)phenylalaninate (**3m**)



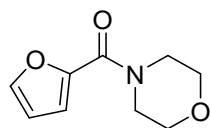
3m was obtained in 72% yield (0.163 g) according to the general procedure (Hexan/EtOAc, 1:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42 (d, $J = 8.4$ Hz, 2H), 7.35-7.28 (m, 3H), 7.23 (d, $J = 7.2$ Hz, 2H), 7.04 (d, $J = 8.4$ Hz, 2H), 5.11 (dd, $J = 7.6$ Hz, $J = 124.4$ Hz, 1H), 3.68-3.46 (m, 8H), 3.22 (d, $J = 6.4$ Hz, 2H), 1.43 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.5, 169.6, 155.2, 151.5, 135.7, 133.1, 129.5 (2C), 128.9 (2C), 128.7 (2C), 127.4, 121.7, 80.4, 66.9 (2C), 54.8, 48.3, 42.9, 38.4, 28.4 (3C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{25}\text{H}_{31}\text{N}_2\text{O}_6^+ = 455.2182$; found 455.2179.

Morpholino(naphthalen-1-yl)methanone (**3n**)^[S2]



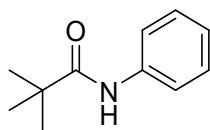
3j was obtained in 82% yield (0.099 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.89-7.84 (m, 3H), 7.56-7.43 (m, 3H), 7.41 (dd, $J = 7.2, 1.6$ Hz, 1H), 4.00-3.83 (m, 4H), 3.54-3.49 (m, 2H), 3.22-3.18 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 133.8, 133.6, 129.7, 129.5, 128.6, 127.3, 126.7, 125.3, 124.7, 124.0, 67.2, 67.1, 47.7, 42.3; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{15}\text{H}_{16}\text{NO}_2^+ = 242.1176$; found 242.1177.

Furan-2-yl(morpholino)methanone (**3o**)^[S11]



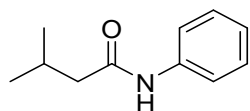
3k was obtained in 89% yield (0.081 g) according to the general procedure (Hexan/EtOAc, 2:1); green oil; ^1H NMR (400 MHz, CDCl_3) δ 7.48-7.47 (m, 1H), 7.03-7.02 (m, 1H), 6.49-6.47 (m, 1H), 3.81-3.73 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.2, 147.9, 143.9, 116.9, 111.5, 67.1 (2C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_9\text{H}_{12}\text{NO}_3^+ = 182.0812$; found 182.0815.

***N*-Phenylpivalamide (4a)** ^[S12]



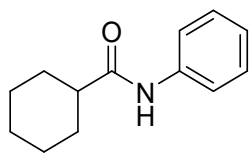
4a was obtained in 91% yield (0.081 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 132-136 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.55-7.52 (m, 2H), 7.33-7.29 (m, 2H), 7.11-7.08 (m, 1H), 1.32 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 176.7, 138.2, 129.1 (2C), 124.32, 120.1 (2C), 39.7, 27.8 (3C); HRMS (ESI) m/z (M+H)⁺ calcd for C₁₁H₁₆NO⁺ = 178.1227; found 178.1230.

3-Methyl-*N*-phenylbutanamide (4b) ^[S13]



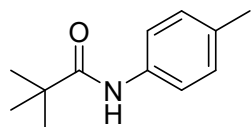
4b was obtained in 53% yield (0.047 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 109-110 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.53 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.10 (t, *J* = 7.6 Hz, 1H), 2.24-2.20 (m, 2H), 1.04-0.95 (m, 7H); ¹³C NMR (100 MHz, CDCl₃) δ 170.9, 138.1, 129.1 (2C), 124.3, 119.9 (2C), 47.2, 26.4, 22.6 (2C); HRMS (ESI) m/z (M+H)⁺ calcd for C₁₁H₁₆NO⁺ = 178.1227; found 178.1229.

***N*-Phenylcyclohexanecarboxamide (4c)** ^[S4]



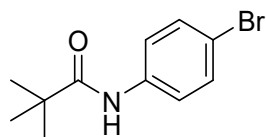
4c was obtained in 78% yield (0.079 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 144-145 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.54 (d, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.09 (t, *J* = 7.6 Hz, 1H), 2.26-2.20 (m, 1H), 1.97 (d, *J* = 12.8 Hz, 2H), 1.85 (d, *J* = 12.8 Hz, 2H), 1.72-1.69 (m, 1H), 1.59-1.50 (m, 2H), 1.35-1.23 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 174.5, 138.2, 129.1 (2C), 124.2, 119.9, 119.8, 46.7, 29.8 (2C), 25.8 (3C); HRMS (ESI) m/z (M+H)⁺ calcd for C₁₃H₁₈NO⁺ = 204.1383; found 204.1384.

***N*-(*p*-tolyl)pivalamide (4d)** ^[S14]



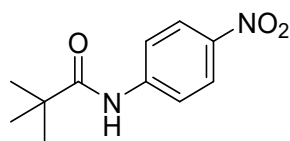
4d was obtained in 90% yield (0.086 g) according to the general procedure (Hexan/EtOAc, 4:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.40 (d, J = 8.4 Hz, 2H), 7.31 (s, 1H), 7.11 (d, J = 8.0 Hz, 2H), 2.30 (s, 3H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.6, 136.6, 133.8, 129.5 (2C), 120.2 (2C), 39.6, 27.7 (3C), 20.9; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{12}\text{H}_{18}\text{NO}^+$ = 192.1388; found 192.1387.

N-(4-bromophenyl)pivalamide (4e) ^[S14]



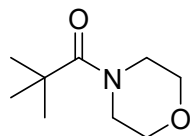
4e was obtained in 84% yield (0.107 g) according to the general procedure (Hexan/EtOAc, 4:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.45-7.39 (m, 4H), 7.32 (s, 1H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.7, 137.2, 132.0 (2C), 121.7 (2C), 116.9, 39.8, 27.7 (3C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{15}\text{BrNO}^+$ = 256.0337; found 256.0335.

N-(4-nitrophenyl)pivalamide (4f) ^[S14]



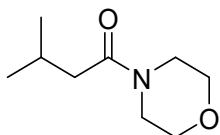
4f was obtained in 80% yield (0.089 g) according to the general procedure (Hexan/EtOAc, 4:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 8.18 (d, J = 9.2 Hz, 2H), 7.74-7.72 (m, 3H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 177.2, 144.1, 143.5, 125.1 (2C), 119.4 (2C), 40.1, 27.5 (3C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{15}\text{N}_2\text{O}^+$ = 223.1083; found 223.1080.

2,2-dimethyl-1-morpholinopropan-1-one (4g) ^[S15]



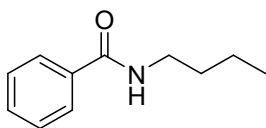
4g was obtained in 85% yield (0.073 g) according to the general procedure (Hexan/EtOAc, 4:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 3.66-3.62 (m, 8H), 1.27 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.6, 67.0 (2C), 45.9 (2C), 38.7, 28.4 (3C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_9\text{H}_{18}\text{NO}_2^+ = 172.1338$; found 172.1337.

3-Methyl-1-morpholinobutan-1-one (**4h**) ^[S16]



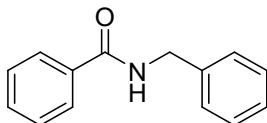
4h was obtained in 86% yield (0.074 g) according to the general procedure (Hexan/EtOAc, 4:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 3.57-3.38 (m, 8H), 2.11-1.99 (m, 3H), 0.90-0.86 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.1, 66.9, 66.6, 46.2, 41.8, 41.7, 25.6, 22.6 (2C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_9\text{H}_{18}\text{NO}_2^+ = 172.1338$; found 172.1335.

N-Butylbenzamide (**5a**) ^[S17]



5b was obtained in 61% yield (0.054 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.76-7.74 (m, 2H), 7.48 (t, $J = 7.2$ Hz, 1H), 7.43-7.40 (m, 2H), 6.21 (br s, 1H), 3.46 (dd, $J = 12.8, 7.2$ Hz, 2H), 1.64-1.56 (m, 2H), 1.46-1.36 (m, 2H), 0.95 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.7, 134.9, 131.4, 128.7 (2C), 126.9 (2C), 39.9, 31.9, 20.3, 13.9; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{16}\text{NO}^+ = 178.1227$; found 178.1230.

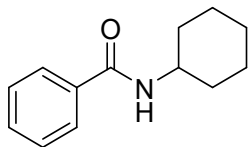
N-Benzylbenzamide (**5b**) ^[S18]



5b was obtained in 67% yield (0.071 mg) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 104-105 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.80-7.78 (m, 2H), 7.52-7.48 (m, 1H), 7.44-7.40 (m, 2H), 7.36-7.29 (m, 5H), 6.50 (br s, 1H), 4.65 (d, $J = 5.6$ Hz, 2H); ^{13}C

NMR (100 MHz, CDCl₃) δ 167.5, 138.3, 134.5, 131.7, 128.9 (2C), 128.7 (2C), 128.0 (2C), 127.7, 127.1 (2C), 44.3; HRMS (ESI) m/z (M+H)⁺ calcd for C₁₄H₁₄NO⁺ = 212.1070; found 212.1072.

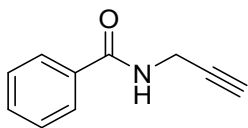
***N*-Cyclohexylbenzamide (5c)** ^[S18]



Exact Mass: 203.1310

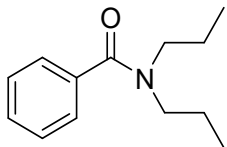
5c was obtained in 50% yield (0.051 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 153-154 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, J = 7.2 Hz, 2H), 7.48 (t, J = 7.2 Hz, 1H), 7.39 (t, J = 7.2 Hz, 2H), 6.11 (br s, 1H), 3.99-3.93 (m, 1H), 2.03-1.99 (m, 2H), 1.76-1.71 (m, 2H), 1.65-1.61 (m, 1H), 1.44-1.36 (m, 2H), 1.28-1.17 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 166.8, 135.2, 131.3, 128.6 (2C), 126.9 (2C), 48.8, 33.4 (2C), 25.7, 25.0 (2C); HRMS (ESI) m/z (M+H)⁺ calcd for C₁₃H₁₈NO = 204.1383; found 204.1386.

***N*-(Prop-2-yn-1-yl)benzamide (5d)** ^[S12]



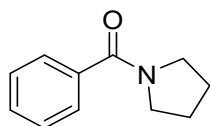
5d was obtained in 51% yield (0.040 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 111-112 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.80-7.77 (m, 2H), 7.53-7.50 (m, 1H), 7.45-7.42 (m, 2H), 6.36 (br s, 1H), 4.26 (dd, J = 5.2, 2.8 Hz, 2H), 2.28 (t, J = 2.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 167.2, 133.9, 131.9, 128.8 (2C), 127.2 (2C), 79.6, 72.1, 29.9; HRMS (ESI) m/z (M+H)⁺ calcd for C₁₀H₁₀NO⁺ = 160.0757; found 160.0759.

***N,N*-Dipropylbenzamide (5e)** ^[S19]



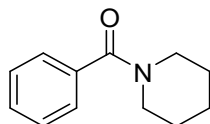
5e was obtained in 70% yield (0.072 g) according to the general procedure (Hexan/EtOAc, 2:1); colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.37-7.31 (m, 5H), 3.44 (s, 2H), 3.14 (s, 2H), 1.66-1.51 (m, 4H), 0.96 (s, 3H), 0.72 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.8, 137.5, 129.1, 128.6 (2C), 126.5 (2C), 50.8, 46.3, 21.9, 20.8, 11.5, 11.1; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{13}\text{H}_{20}\text{NO}^+$ = 206.1540; found 206.1544.

Phenyl(pyrrolidin-1-yl)methanone (5f) ^[S120]



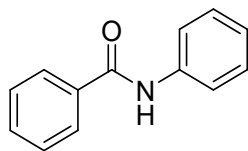
5f was obtained in 73% yield (0.064 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 46-47 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.51-7.49 (m, 2H), 7.40-7.36 (m, 3H), 3.63-3.41 (m, 4H), 1.93-1.87 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.8, 137.3, 129.9, 128.3 (2C), 127.2 (2C), 49.7, 46.3, 26.5, 24.6; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{11}\text{H}_{14}\text{NO}^+$ = 176.1070; found 176.1072.

Phenyl(piperidin-1-yl)methanone (5g) ^[S21]



5g was obtained in 81% yield (0.076 g) according to the general procedure (Hexan/EtOAc, 2:1); yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.38 (s, 5H), 3.70 (br s, 2H), 3.33 (br s, 2H), 1.67-1.51 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.4, 136.6, 129.4, 128.5 (2C), 126.9 (2C), 48.9, 43.2, 26.7, 25.7, 24.7; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{12}\text{H}_{16}\text{NO}^+$ = 190.1227; found 190.1231.

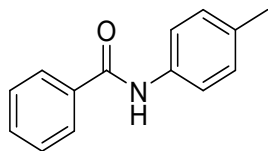
N-Phenylbenzamide (5h) ^[S12]



5h was obtained in 81% yield (0.080 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 166-168 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.88-7.86 (m, 2H), 7.66-7.64 (m,

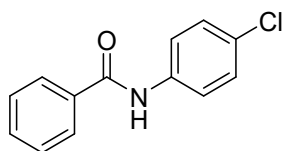
2H), 7.57-7.46 (m, 3H), 7.37 (t, $J = 7.6$ Hz, 2H), 7.16 (t, $J = 7.6$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.9, 138.1, 135.1, 131.9, 129.2 (2C), 128.9 (2C), 127.2 (2C), 124.7, 120.3 (2C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{13}\text{H}_{12}\text{NO}^+ = 198.0914$; found 198.0917.

***N*-(*p*-Tolyl)benzamide (5i)** ^[S21]



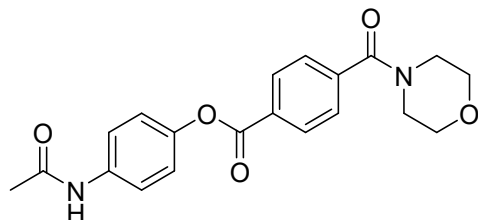
5i was obtained in 82% yield (0.086 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 158-159 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.97 (br s, 1H), 7.86-7.84 (m, 2H), 7.54-7.50 (m, 3H), 7.46-7.42 (m, 2H), 7.16 (d, $J = 8.4$ Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.8, 135.5, 135.2, 134.4, 131.8, 129.7 (2C), 128.9 (2C), 127.1 (2C), 120.4 (2C), 21.0; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{14}\text{H}_{14}\text{NO}^+ = 212.1070$; found 212.1072.

***N*-(4-Chlorophenyl)benzamide (5j)** ^[S22]



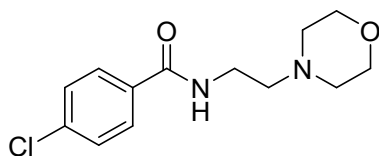
5j was obtained in 68% yield (0.079 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 187 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.87-7.85 (m, 2H), 7.81 (br s, 1H), 7.62-7.55 (m, 3H), 7.52-7.48 (m, 2H), 7.36-7.32 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.8, 136.6, 134.8, 132.2, 129.7, 129.3 (2C), 129.0 (2C), 127.1 (2C), 121.5 (2C); HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{13}\text{H}_{11}\text{ClNO}^+ = 232.0524$; found 232.0528.

4-Acetamidophenyl 4-(morpholine-4-carbonyl)benzoate (6a)



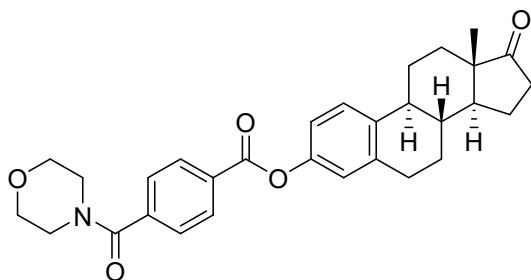
6a was obtained in 78% yield (0.143 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid m.p. 205-206 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.24 (d, J = 8.4 Hz, 2H), 7.54-7.51 (m, 4H), 7.14 (d, J = 8.8 Hz, 2H), 3.81-3.42 (m, 8H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.4, 168.6, 164.6, 146.9, 140.3, 136.1, 130.8, 130.6 (2C), 127.4 (2C), 122.0 (2C), 121.0 (2C), 66.9 (2C), 48.2, 42.7, 24.6; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}_5^+$ = 369.1445; found 369.1449.

Moclobemide (6b)



6b was obtained in 37% yield (0.049 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid, m.p. 136-137 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.73 (d, J = 8.4 Hz, 2H), 7.42 (d, J = 8.8 Hz, 2H), 6.81 (br s, 1H), 3.73 (t, J = 4.4 Hz, 4H), 3.57-3.53 (m, 2H), 2.63-2.52 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.4, 137.8, 133.0, 128.9 (2C), 128.5 (2C), 67.0 (2C), 56.9, 53.4 (2C), 36.1; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{13}\text{H}_{18}\text{ClN}_2\text{O}_2^+$ = 269.1052; found 269.1055.

(8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl 4-(morpholine-4-carbonyl)benzoate (6c)

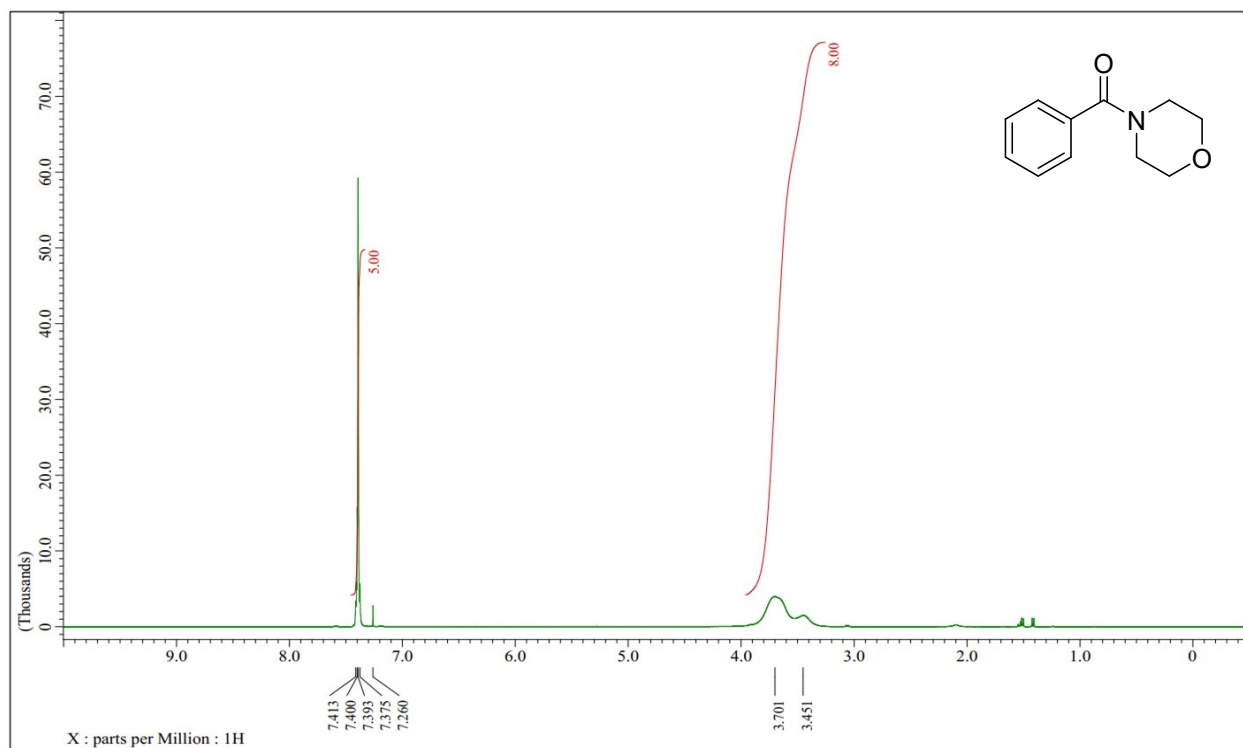


6c was obtained in 32% yield (0.078 g) according to the general procedure (Hexan/EtOAc, 2:1); white solid m.p. 199-201 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.26 (d, J = 8.4 Hz, 2H), 7.55 (d, J = 8.8 Hz, 2H), 7.36 (d, J = 8.4 Hz, 1H), 6.99-6.95 (m, 2H), 3.81-3.42 (m, 8H), 2.95-2.93 (m, 2H), 2.55-2.29 (m, 3H), 2.20-1.96 (m, 4H), 1.69-1.45 (m, 6H), 0.93 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.4, 164.8, 148.8, 140.3, 138.3, 137.8, 131.0, 130.6 (2C), 127.4 (2C), 126.7, 121.7, 118.9, 66.9 (2C), 50.6, 48.1, 44.3, 38.2, 36.0, 31.7, 29.6, 26.5, 25.9, 21.7, 13.9; HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{30}\text{H}_{34}\text{NO}_5^+$ = 488.2432; found 488.2435.

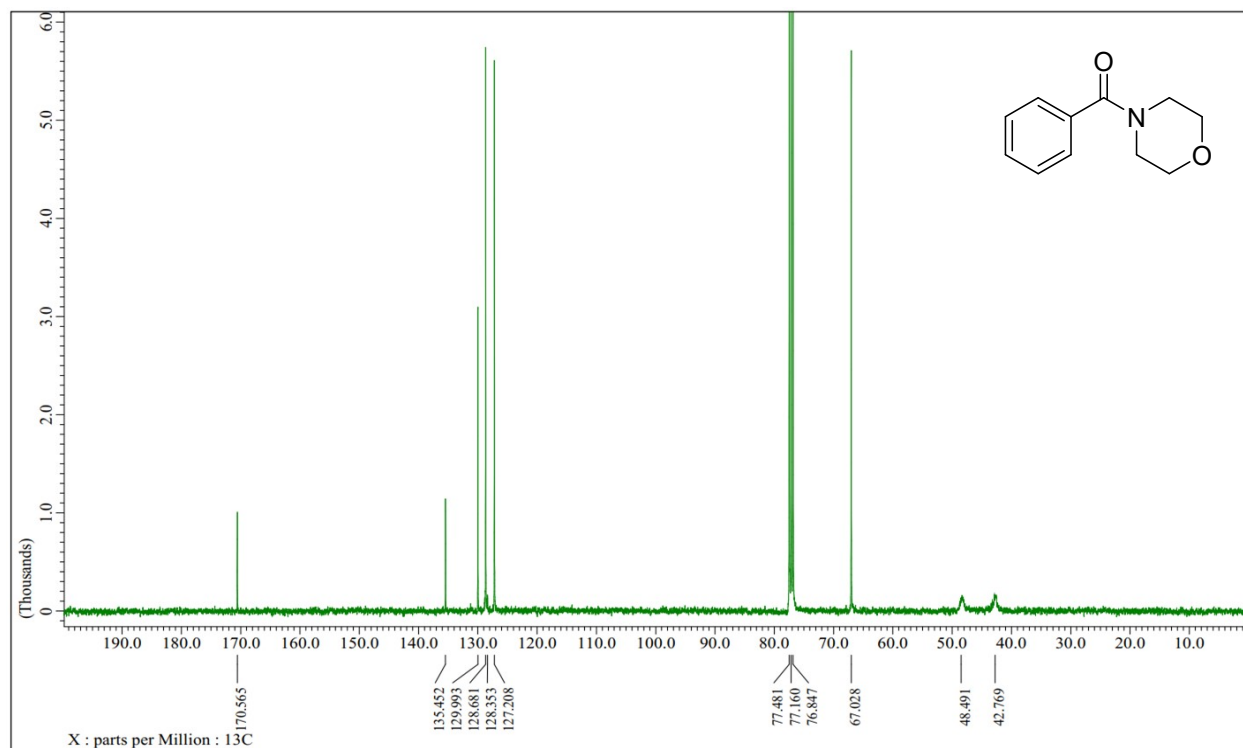
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Morpholino(phenyl)methanone (3a)

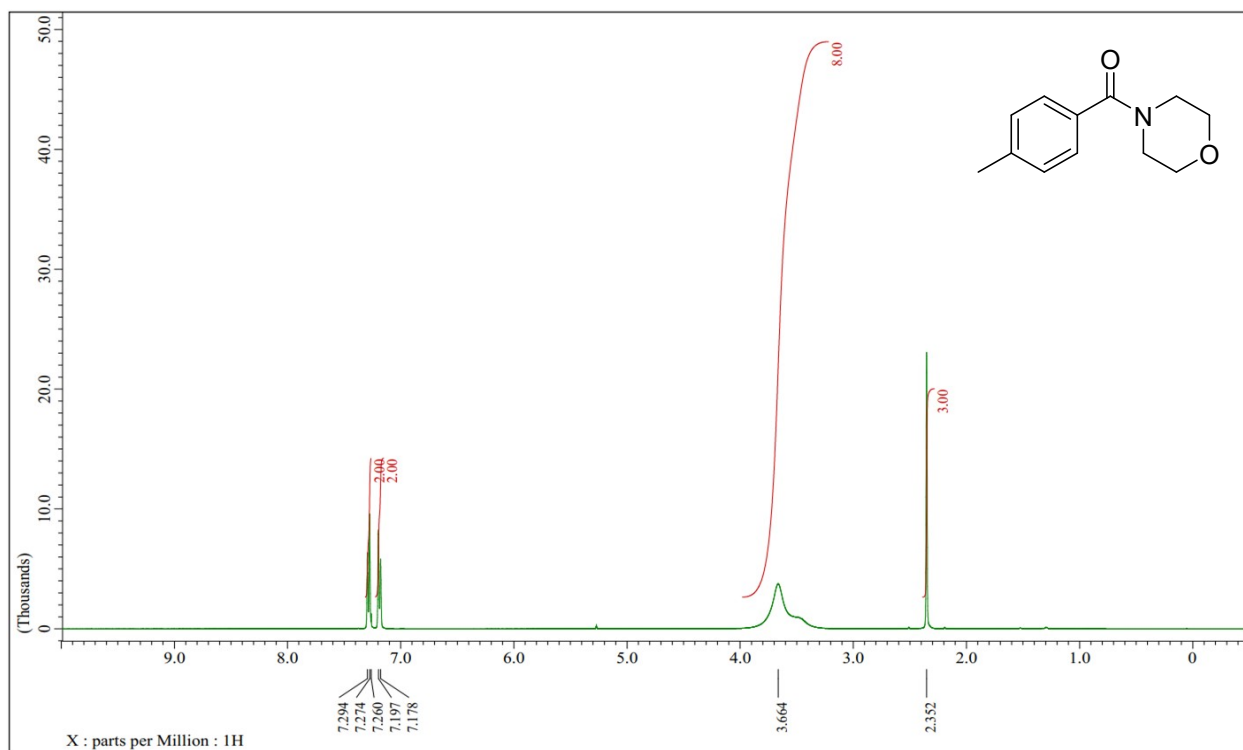


¹H NMR spectrum of morpholino(phenyl)methanone (3a)

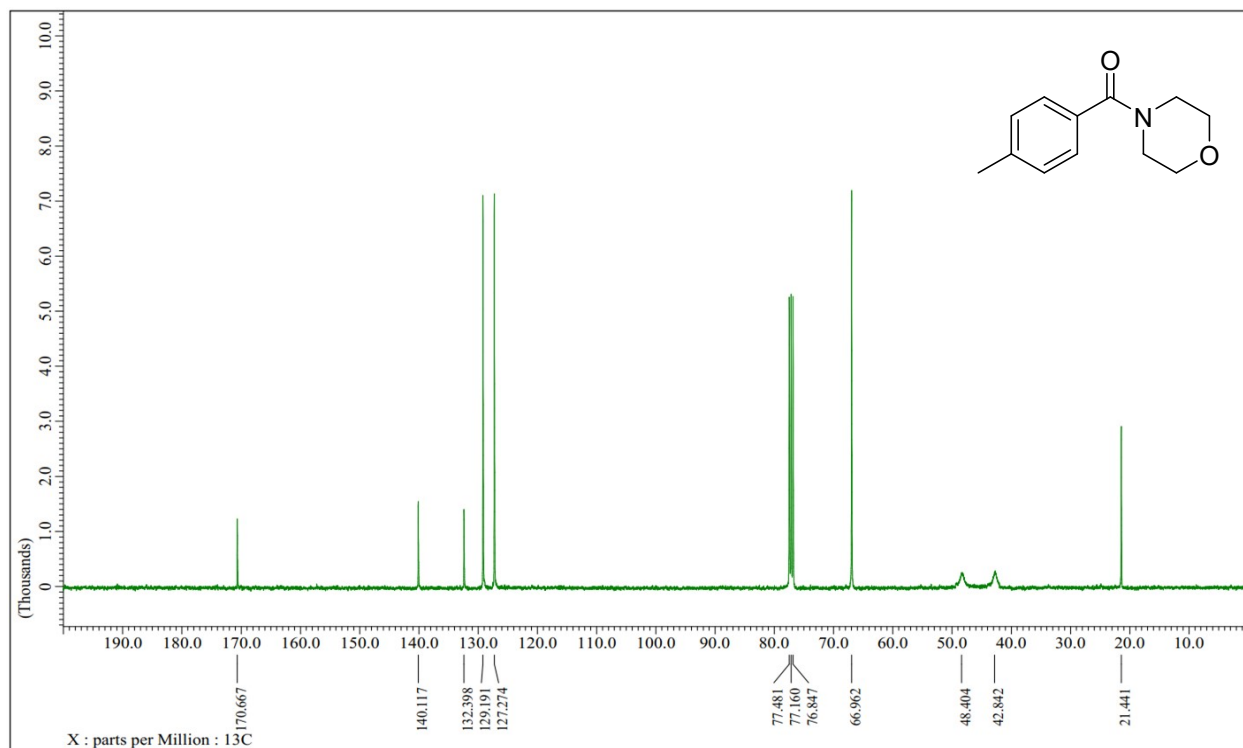


¹³C NMR spectrum of morpholino(phenyl)methanone (3a)

Morpholino(*p*-tolyl)methanone (**3b**)

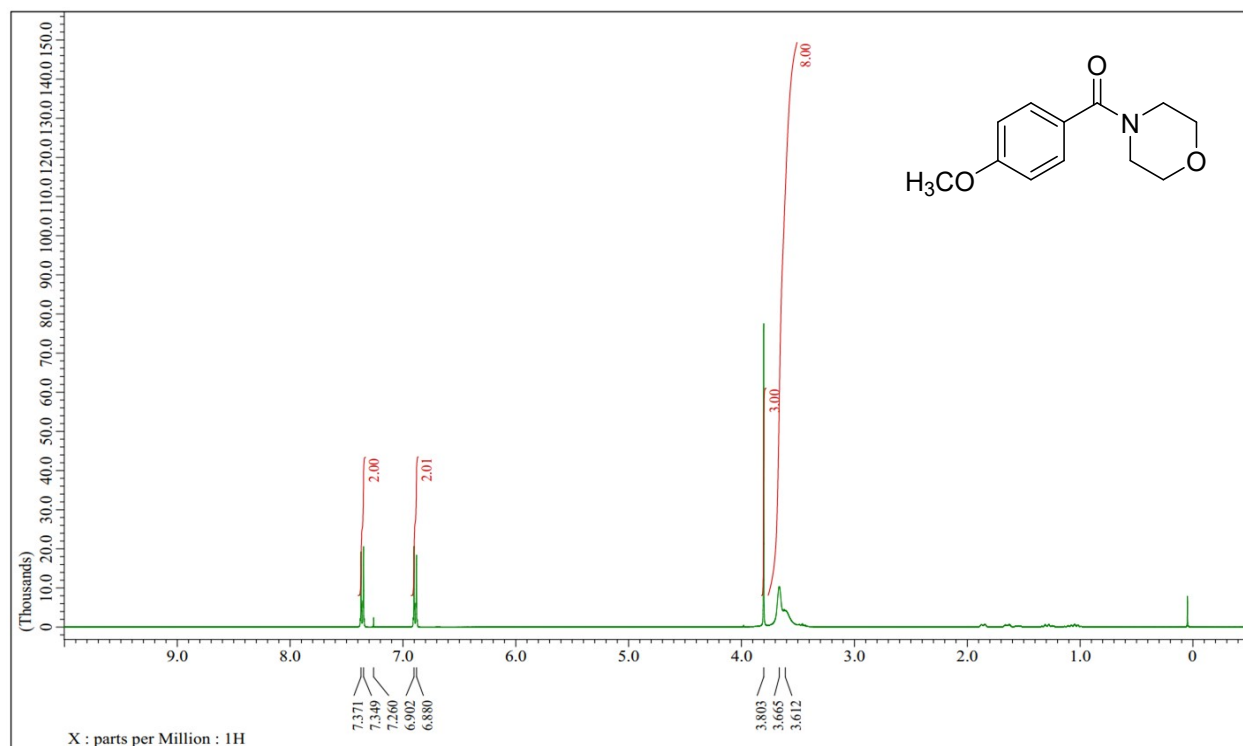


¹H NMR spectrum of morpholino(*p*-tolyl)methanone (**3b**)

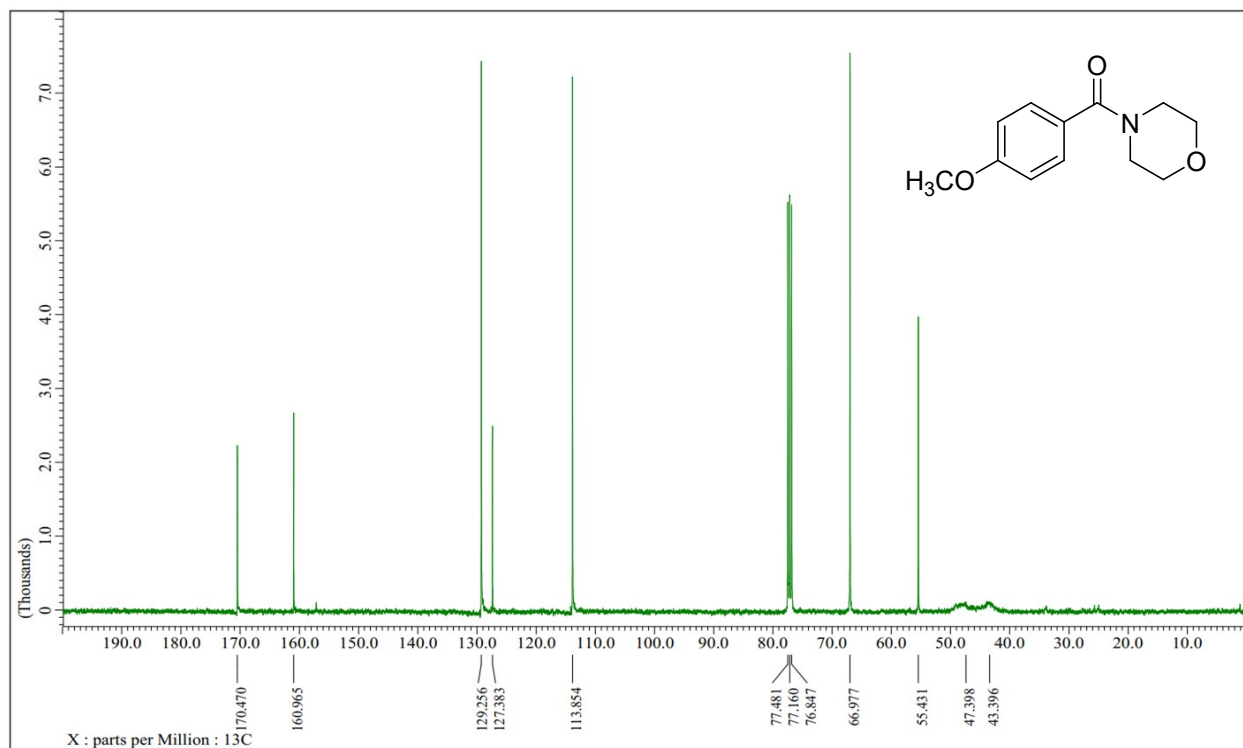


¹³C NMR spectrum of morpholino(*p*-tolyl)methanone (**3b**)

(4-Methoxyphenyl)(morpholino)methanone (3c)

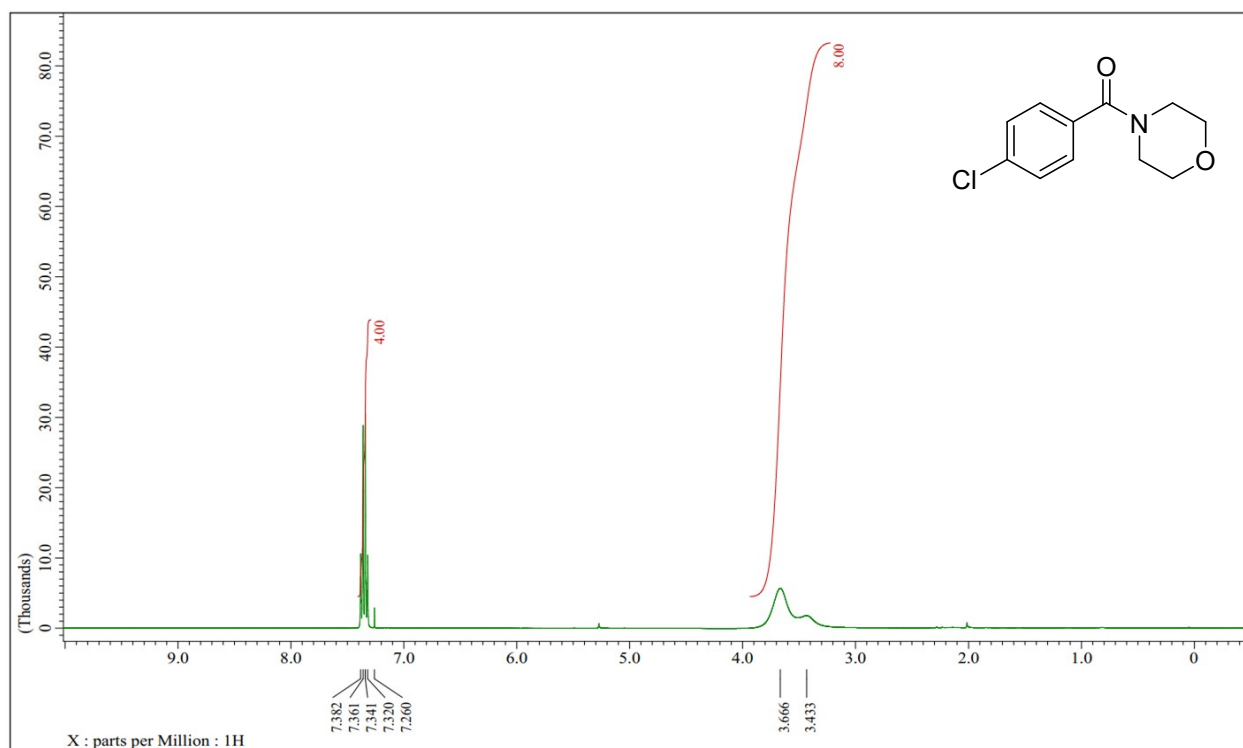


¹H NMR spectrum of (4-methoxyphenyl)(morpholino)methanone (3c)

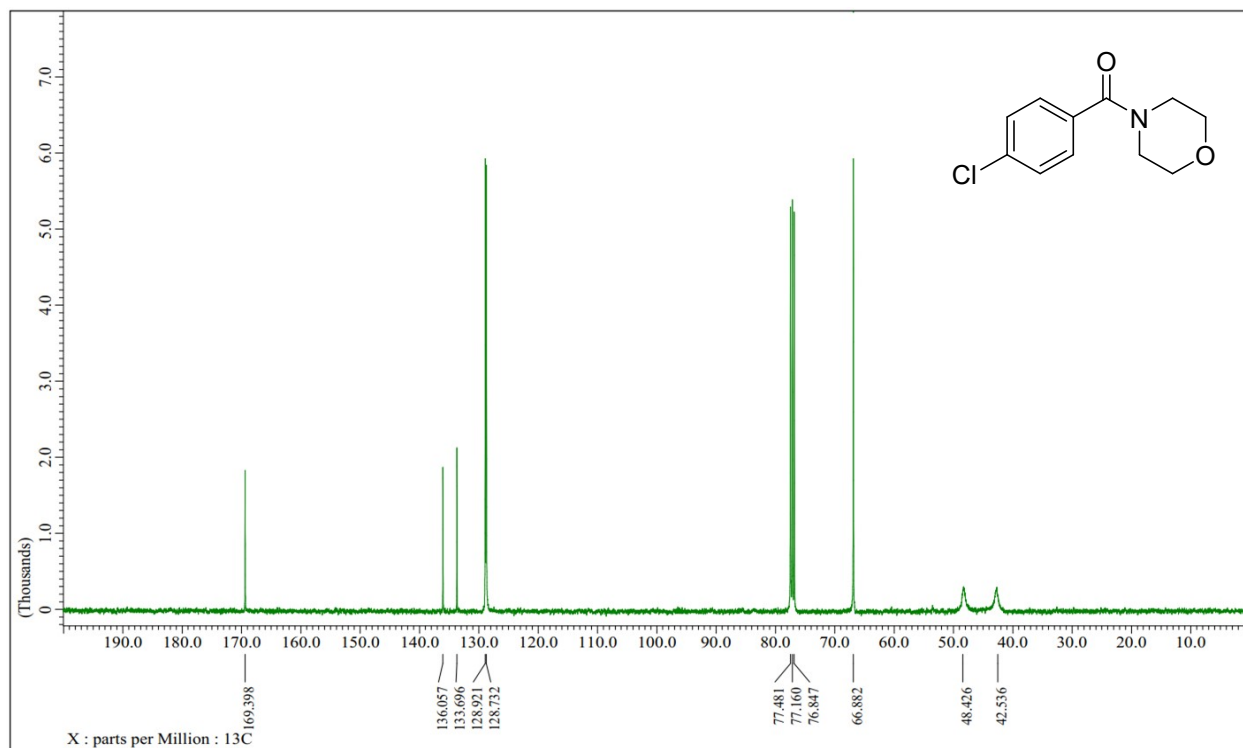


¹³C NMR spectrum of (4-methoxyphenyl)(morpholino)methanone (3c)

(4-Chlorophenyl)(morpholino)methanone (3d)

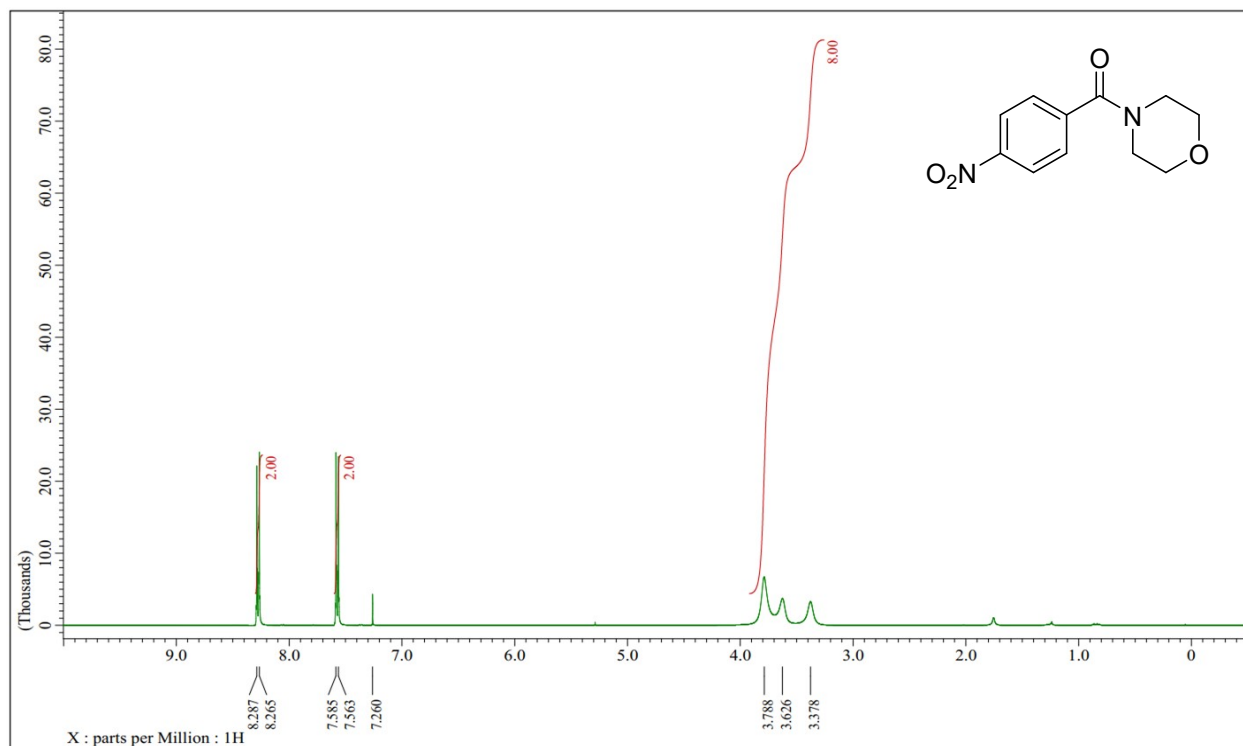


¹H NMR spectrum of (4-chlorophenyl)(morpholino)methanone (3d)

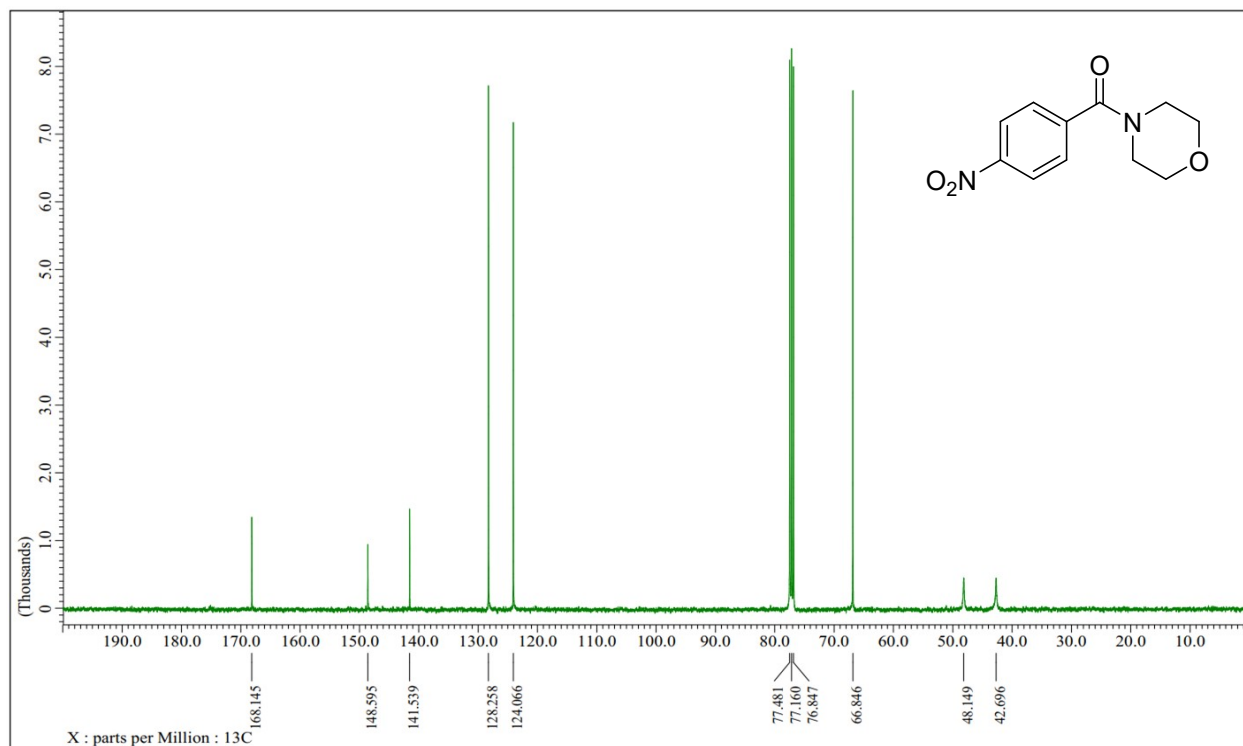


¹³C NMR spectrum of (4-chlorophenyl)(morpholino)methanone (3d)

Morpholino(4-nitrophenyl)methanone (3e)

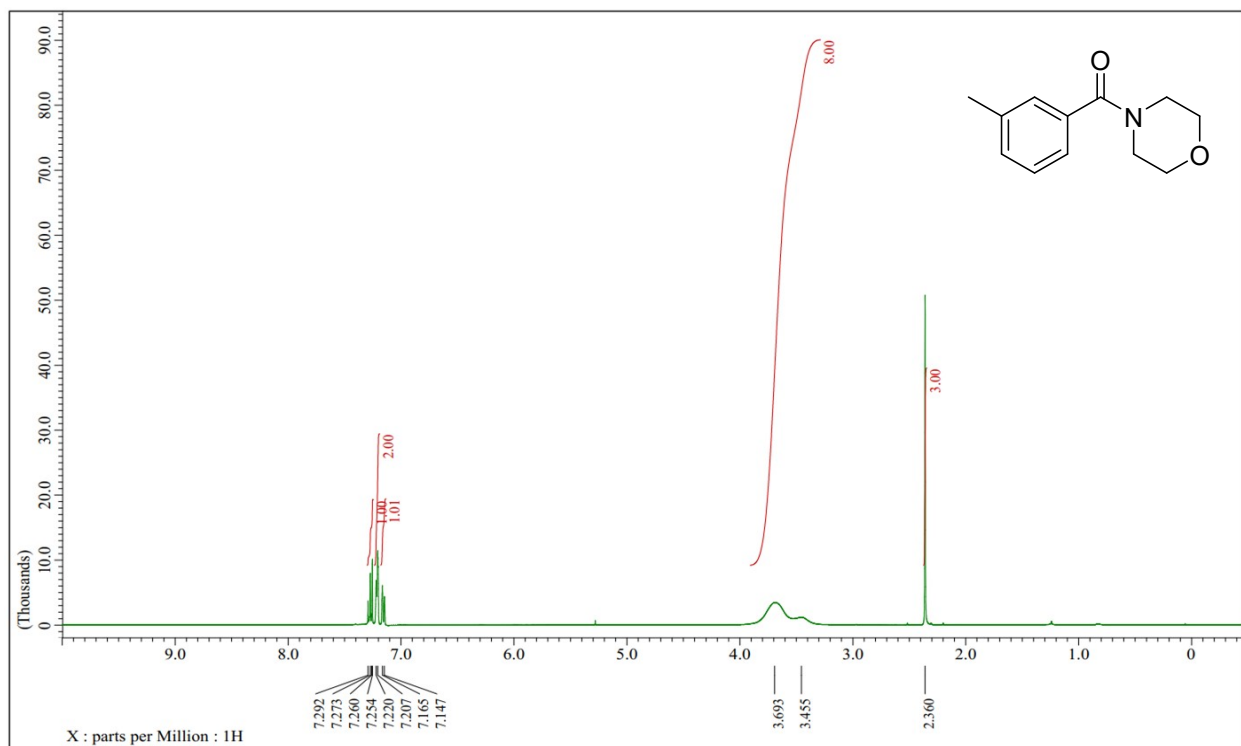


¹H NMR spectrum of morpholino(4-nitrophenyl)methanone (3e)

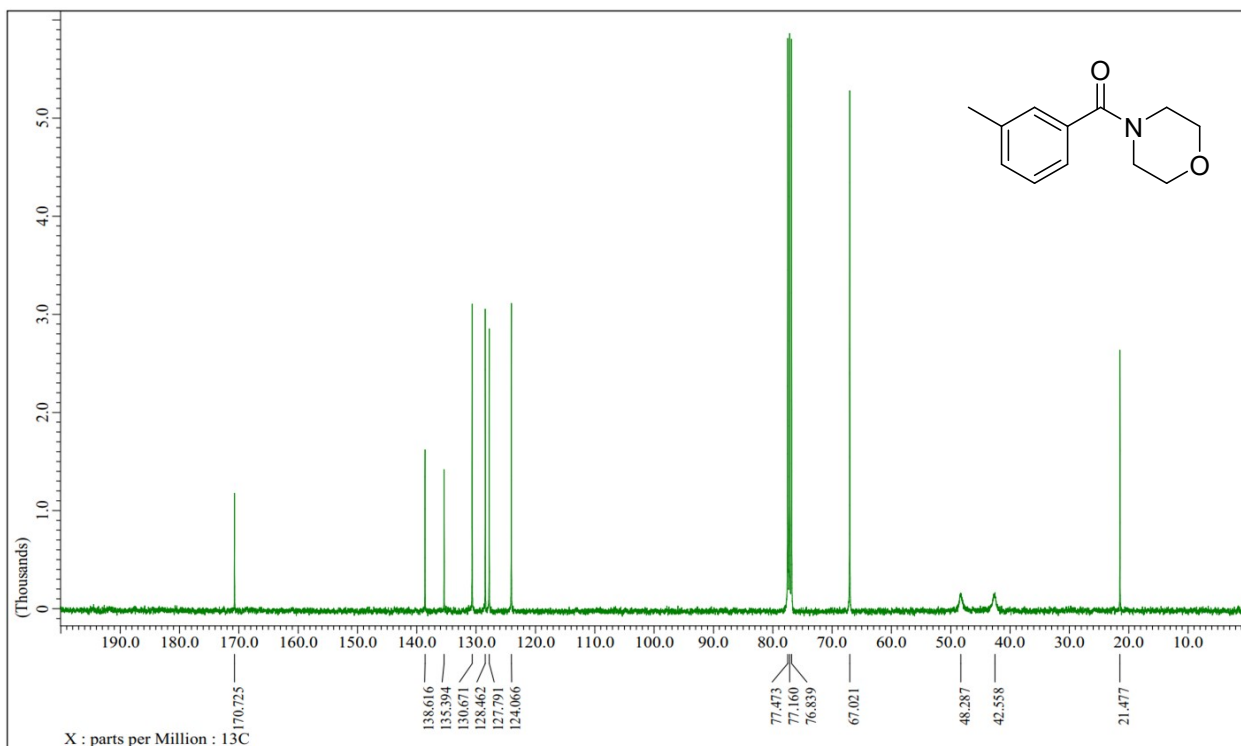


¹³C NMR spectrum of morpholino(4-nitrophenyl)methanone (3e)

Morpholino(*m*-tolyl)methanone (**3f**)

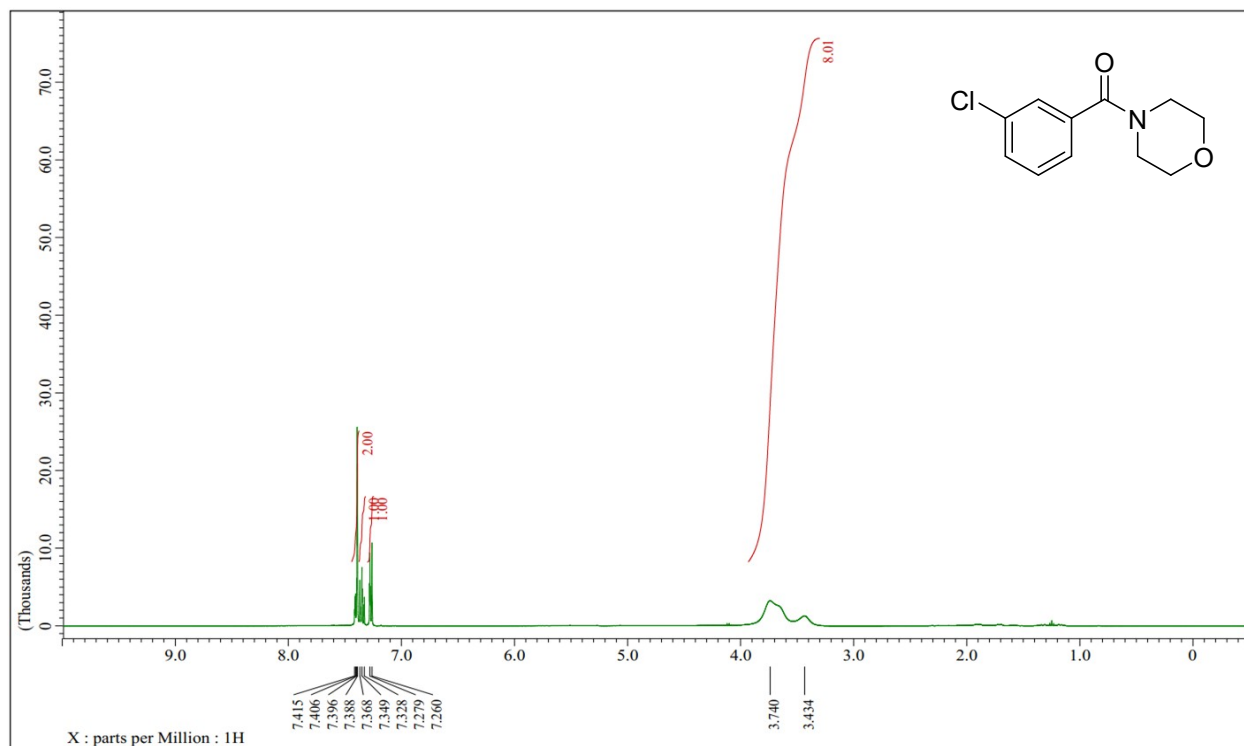


¹H NMR spectrum of morpholino(*m*-tolyl)methanone (**3f**)

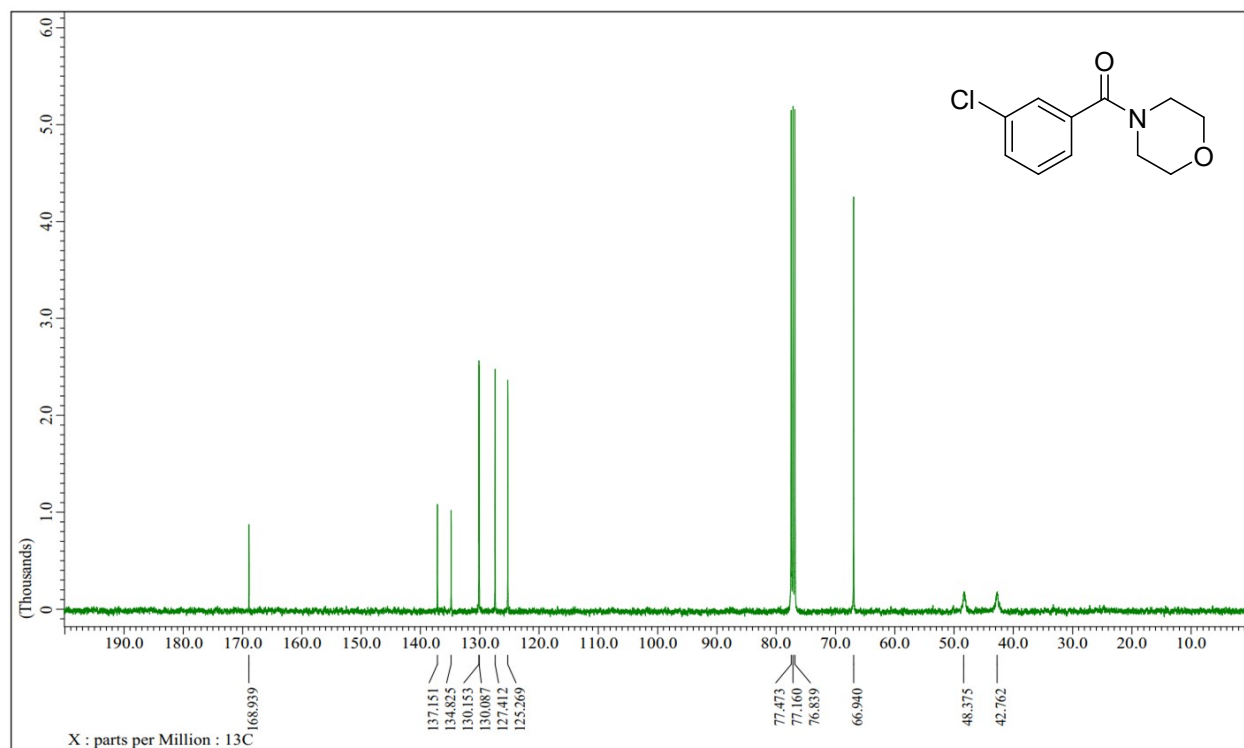


¹³C NMR spectrum of morpholino(*m*-tolyl)methanone (**3f**)

(3-Chlorophenyl)(morpholino)methanone (3g)

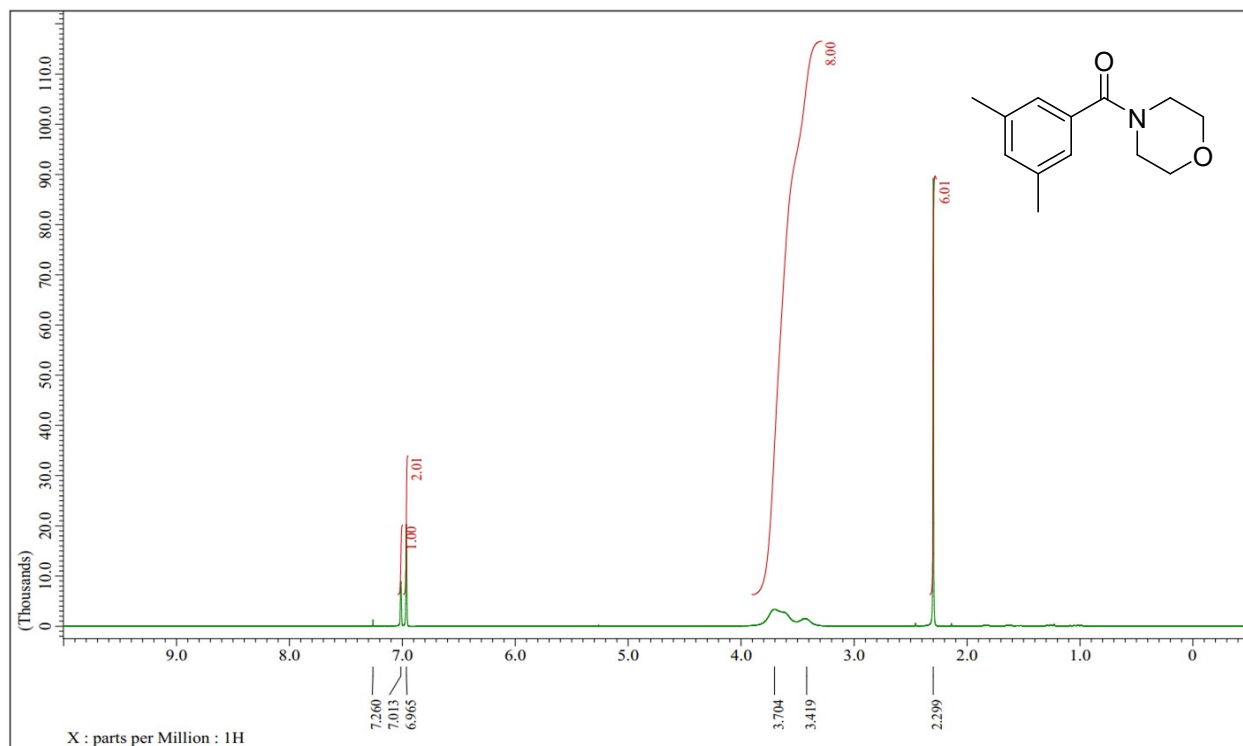


¹H NMR spectrum of (3-chlorophenyl)(morpholino)methanone (3g)

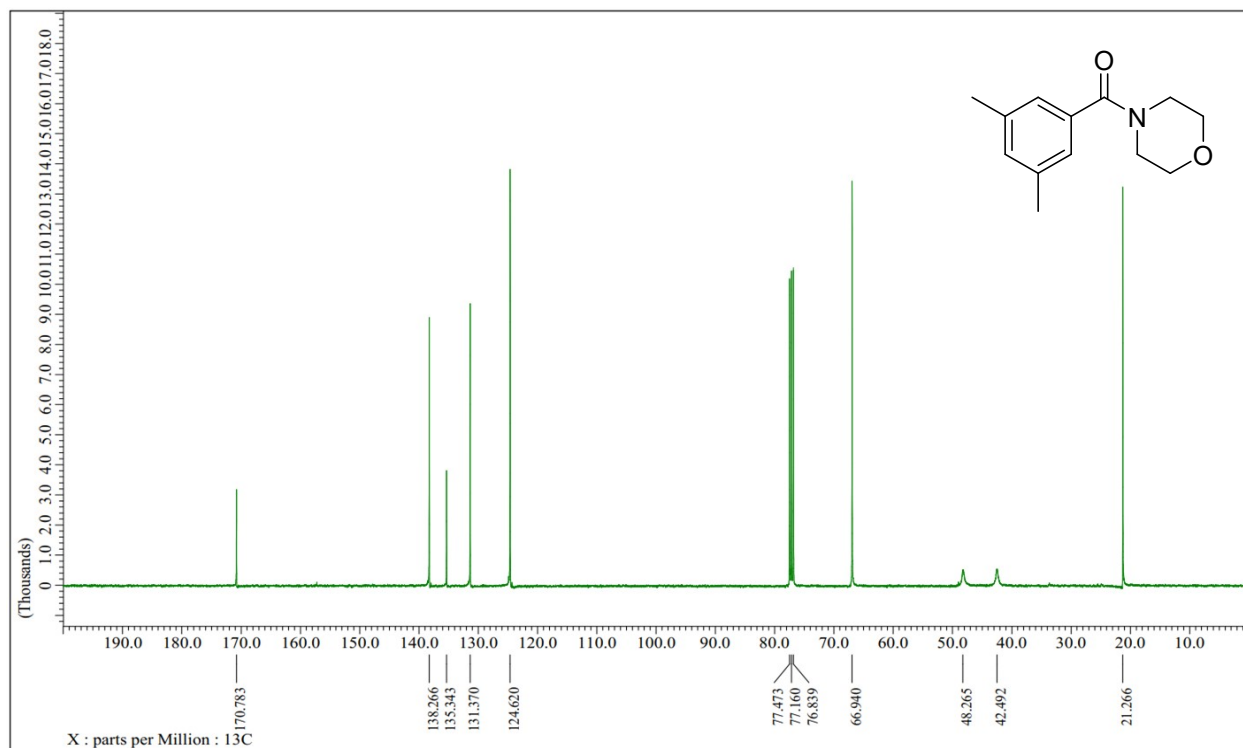


¹³C NMR spectrum of (3-chlorophenyl)(morpholino)methanone (3g)

(3,5-Dimethylphenyl)(morpholino)methanone (3h)

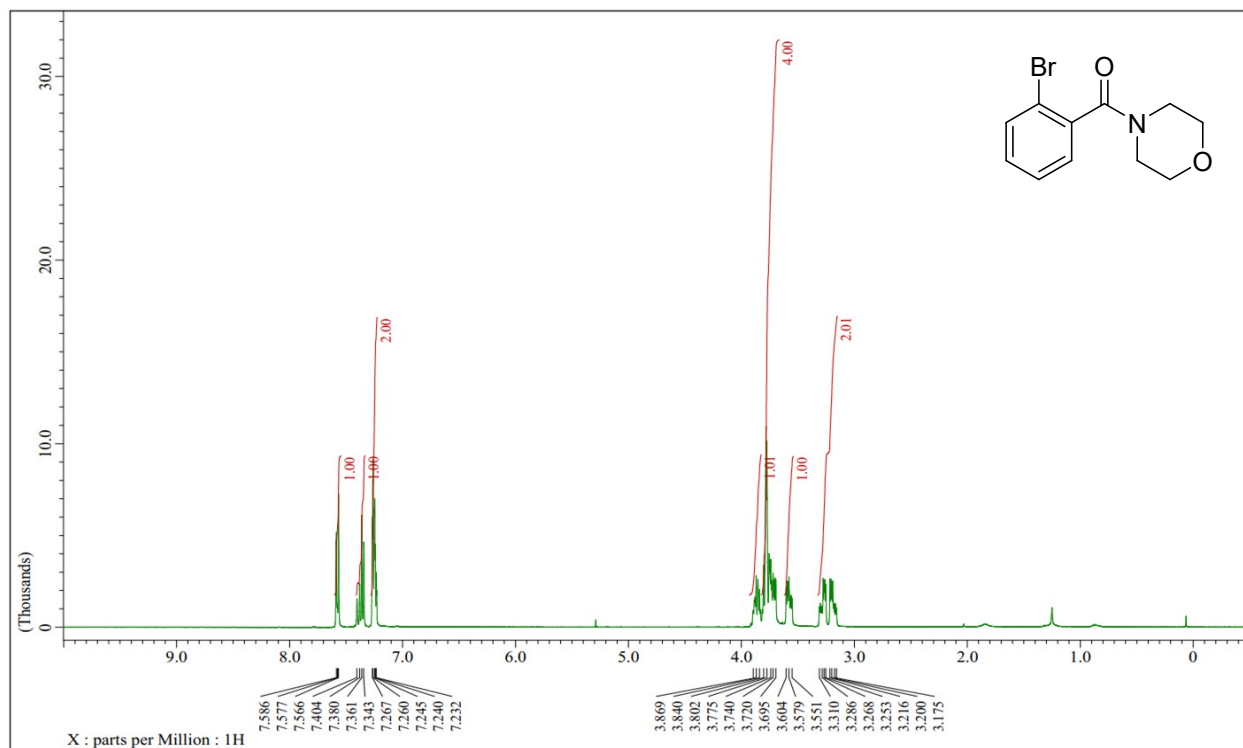


¹H NMR spectrum of (3,5-dimethylphenyl)(morpholino)methanone (3h)

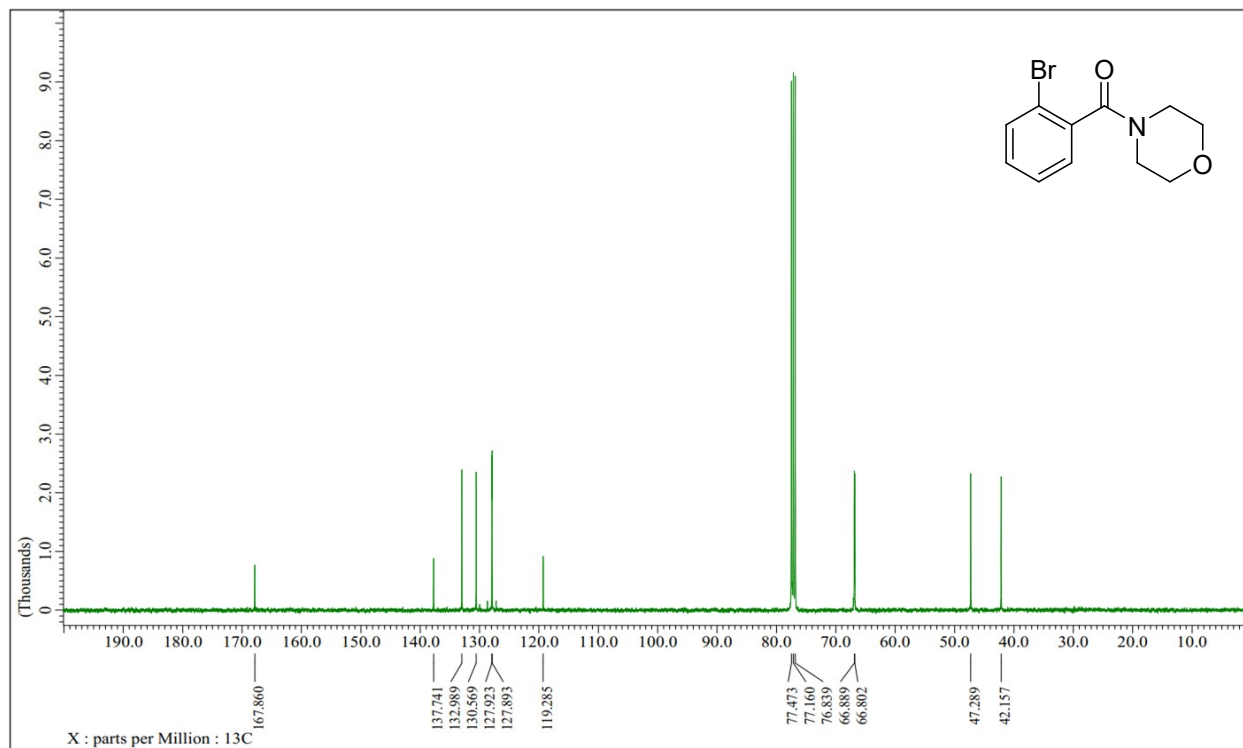


¹³C NMR spectrum of (3,5-dimethylphenyl)(morpholino)methanone (3h)

(2-Bromophenyl)(morpholino)methanone (3i)

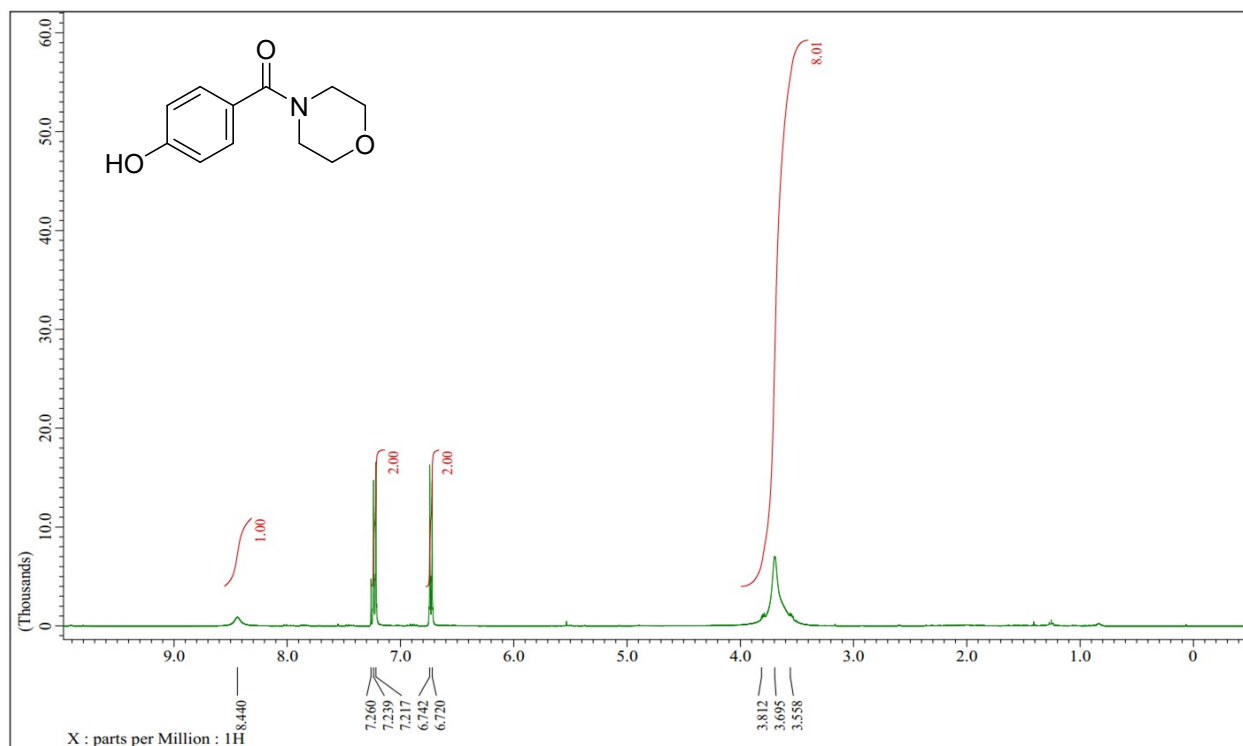


¹H NMR spectrum of (2-bromophenyl)(morpholino)methanone (3i)

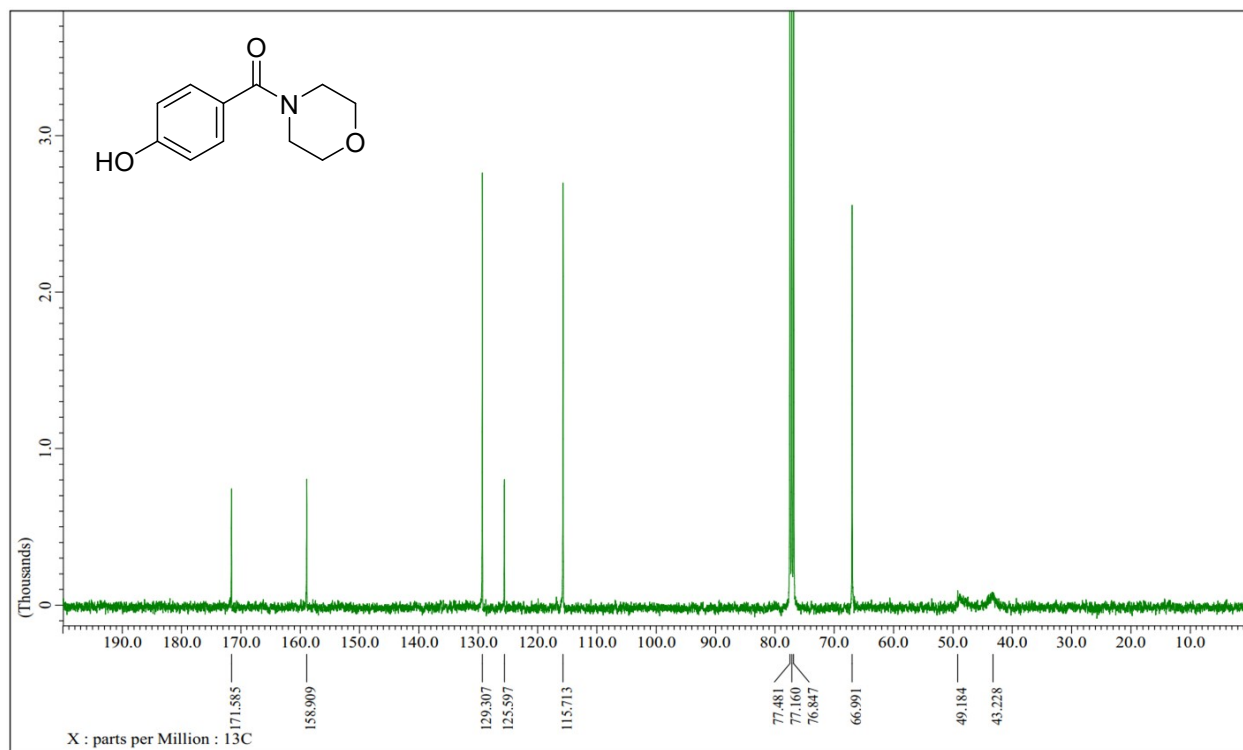


¹³C NMR spectrum of (2-bromophenyl)(morpholino)methanone (3i)

(4-hydroxyphenyl)(morpholino)methanone (3j)

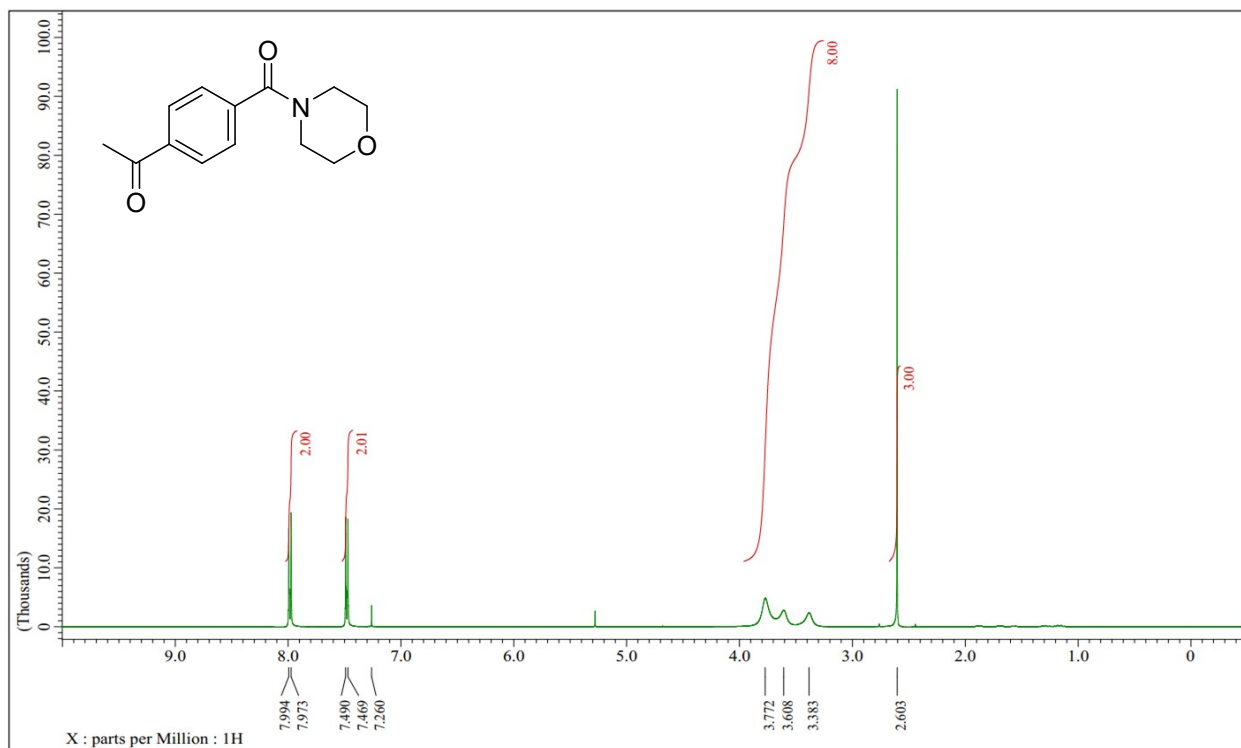


¹H NMR spectrum of (4-hydroxyphenyl)(morpholino)methanone (3j)

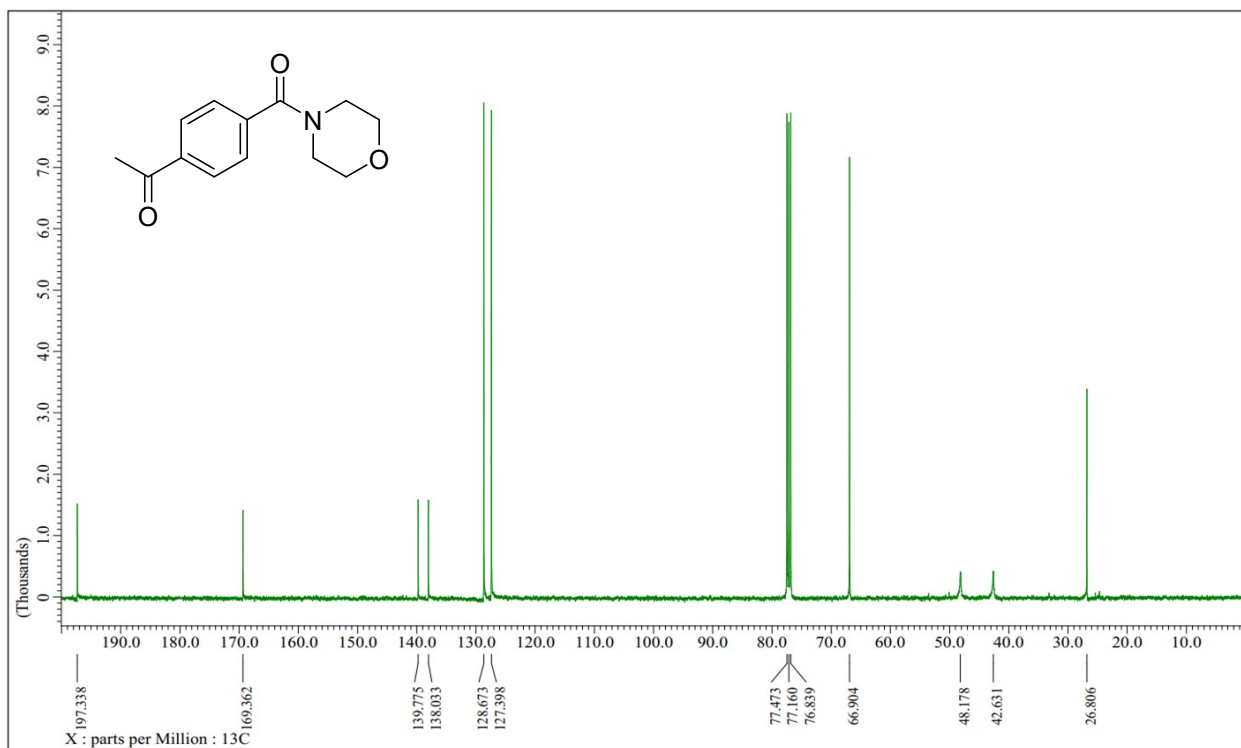


¹³C NMR spectrum of (4-hydroxyphenyl)(morpholino)methanone (3j)

1-(4-(morpholine-4-carbonyl)phenyl)ethan-1-one (3k)

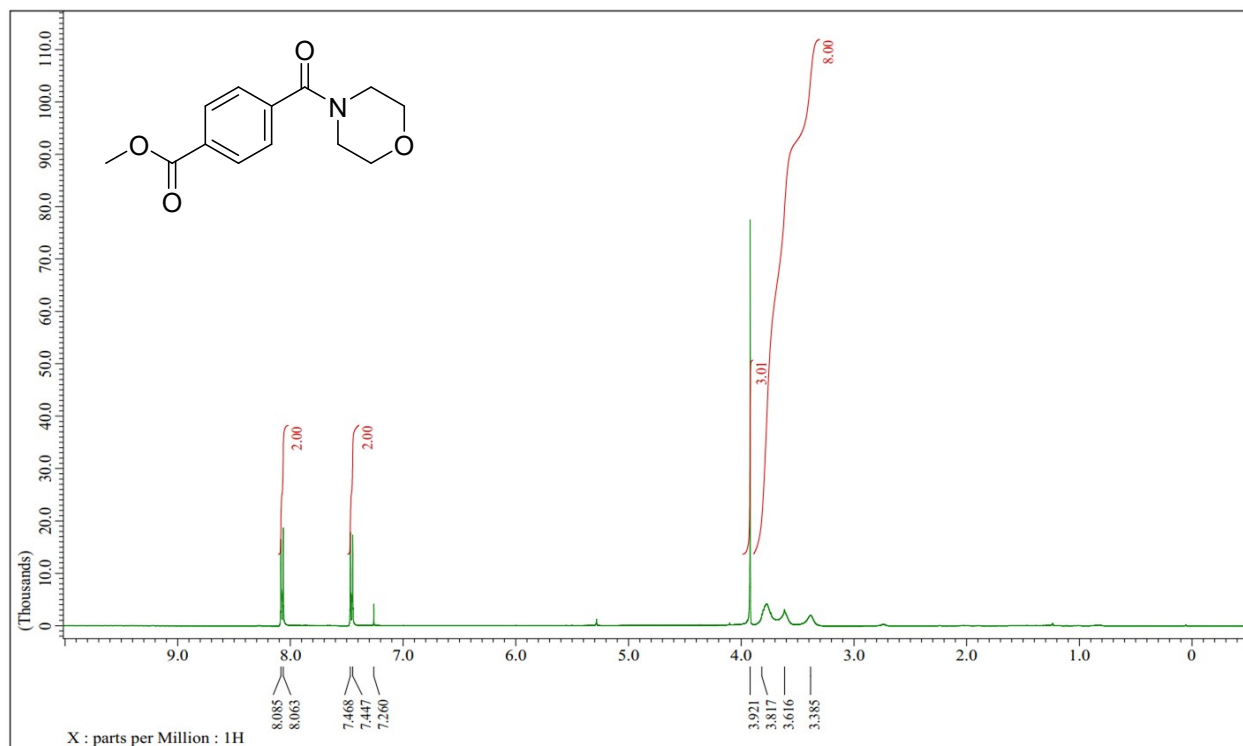


¹H NMR spectrum of 1-(4-(morpholine-4-carbonyl)phenyl)ethan-1-one (3k)

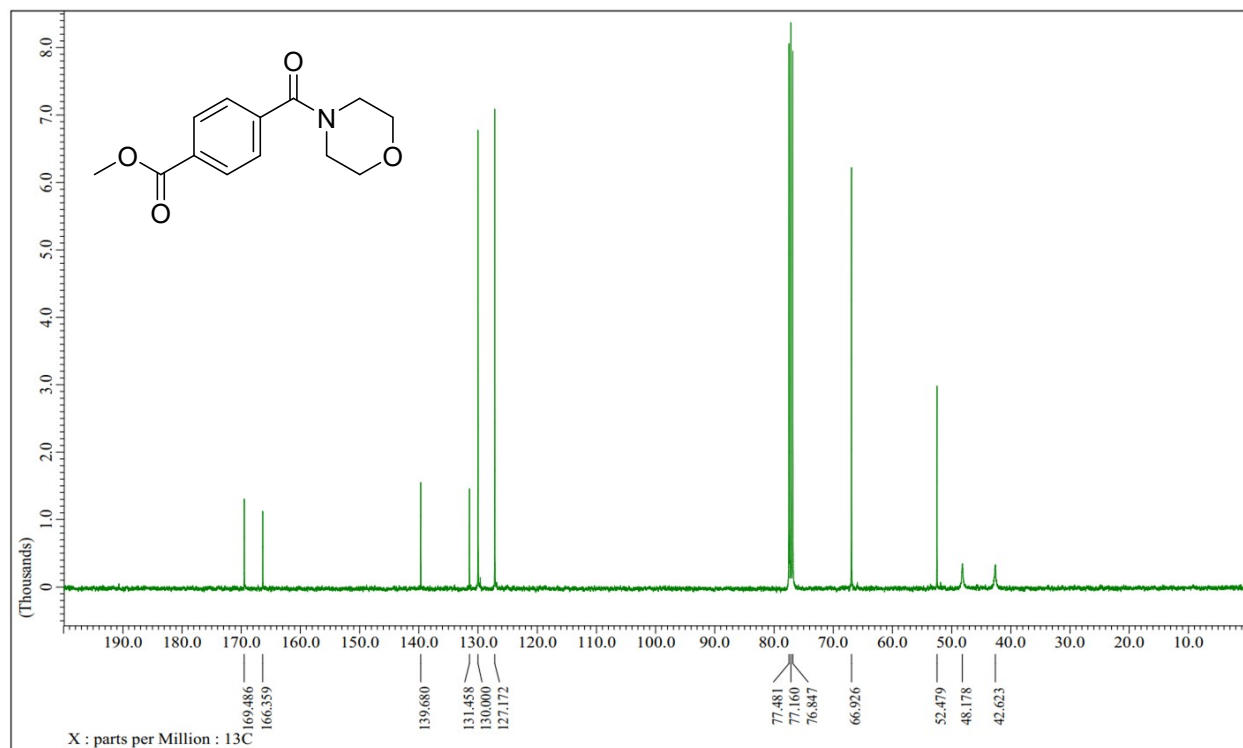


¹³C NMR spectrum of 1-(4-(morpholine-4-carbonyl)phenyl)ethan-1-one (3k)

Methyl 4-(morpholine-4-carbonyl)benzoate (**31**)

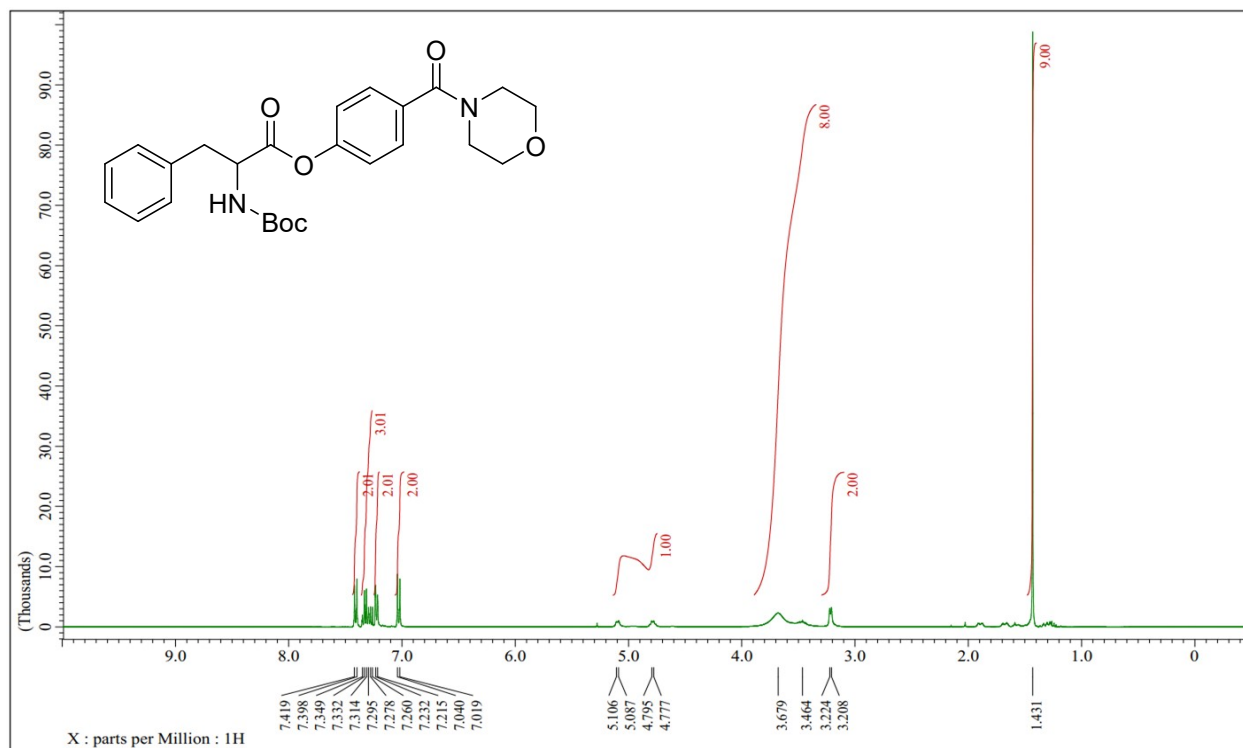


¹H NMR spectrum of methyl 4-(morpholine-4-carbonyl)benzoate (**31**)

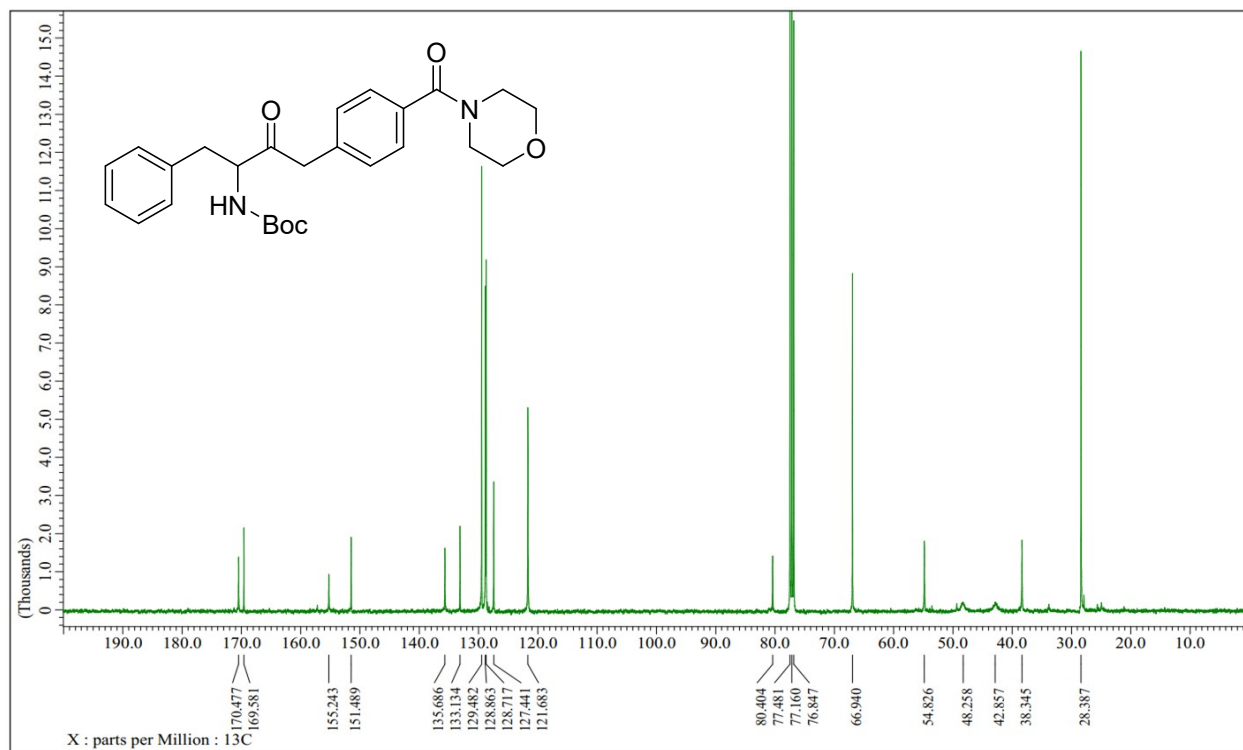


¹³C NMR spectrum of methyl 4-(morpholine-4-carbonyl)benzoate (**31**)

4-(morpholine-4-carbonyl)phenyl (tert-butoxycarbonyl)phenylalaninate (3m)

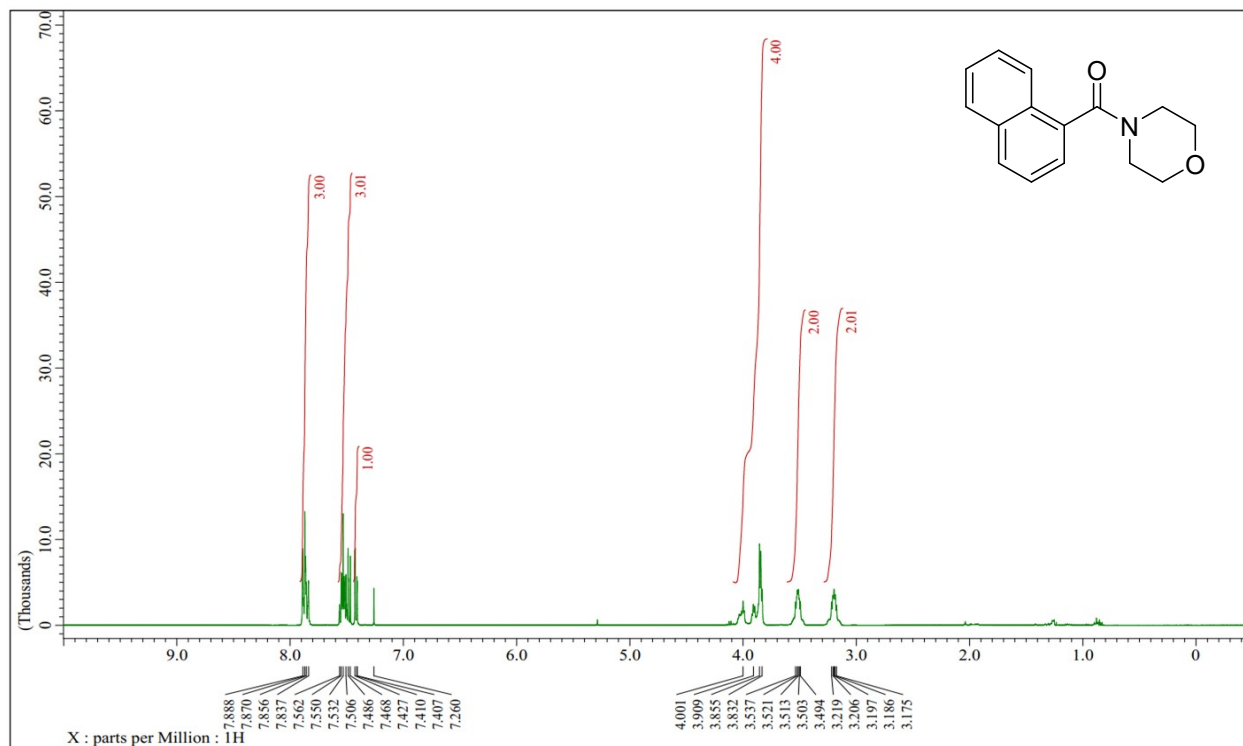


¹H NMR spectrum of 4-(morpholine-4-carbonyl)phenyl (t-butoxycarbonyl)phenylalaninate (3m)

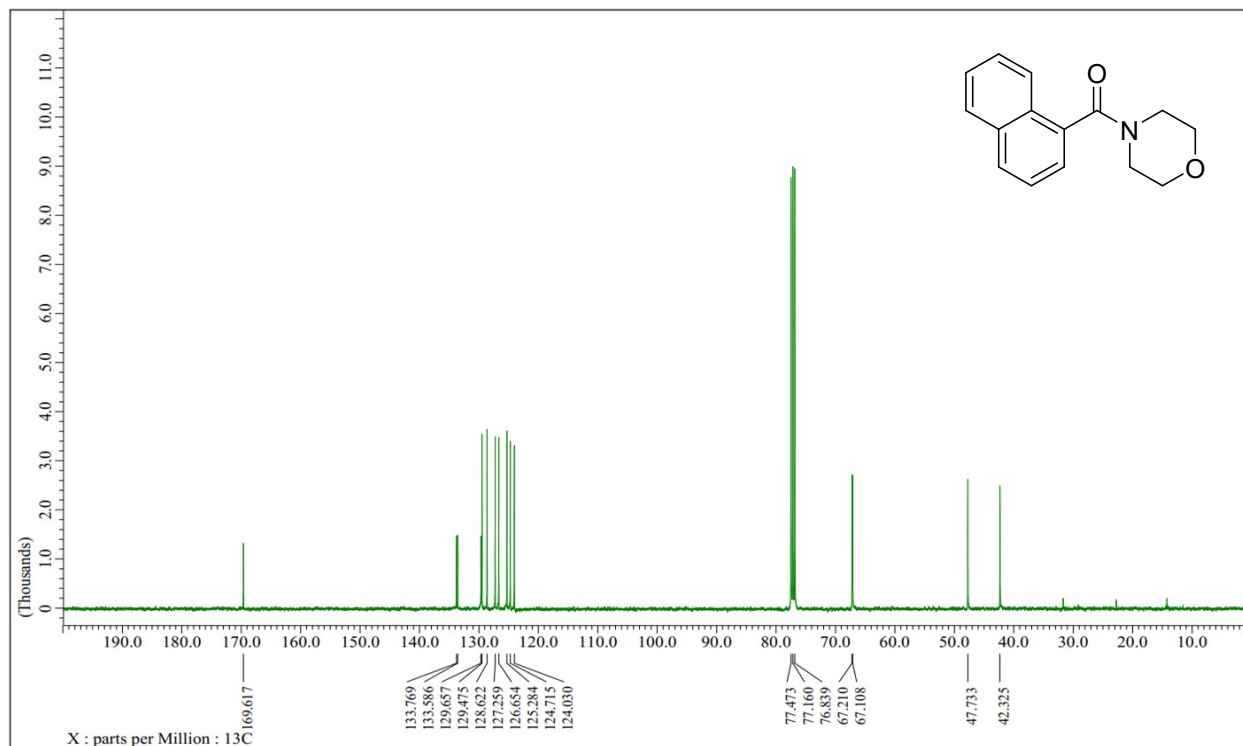


¹³C NMR spectrum of 4-(morpholine-4-carbonyl)phenyl (t-butoxycarbonyl)phenylalaninate (3m)

Morpholino(naphthalen-1-yl)methanone (3n)

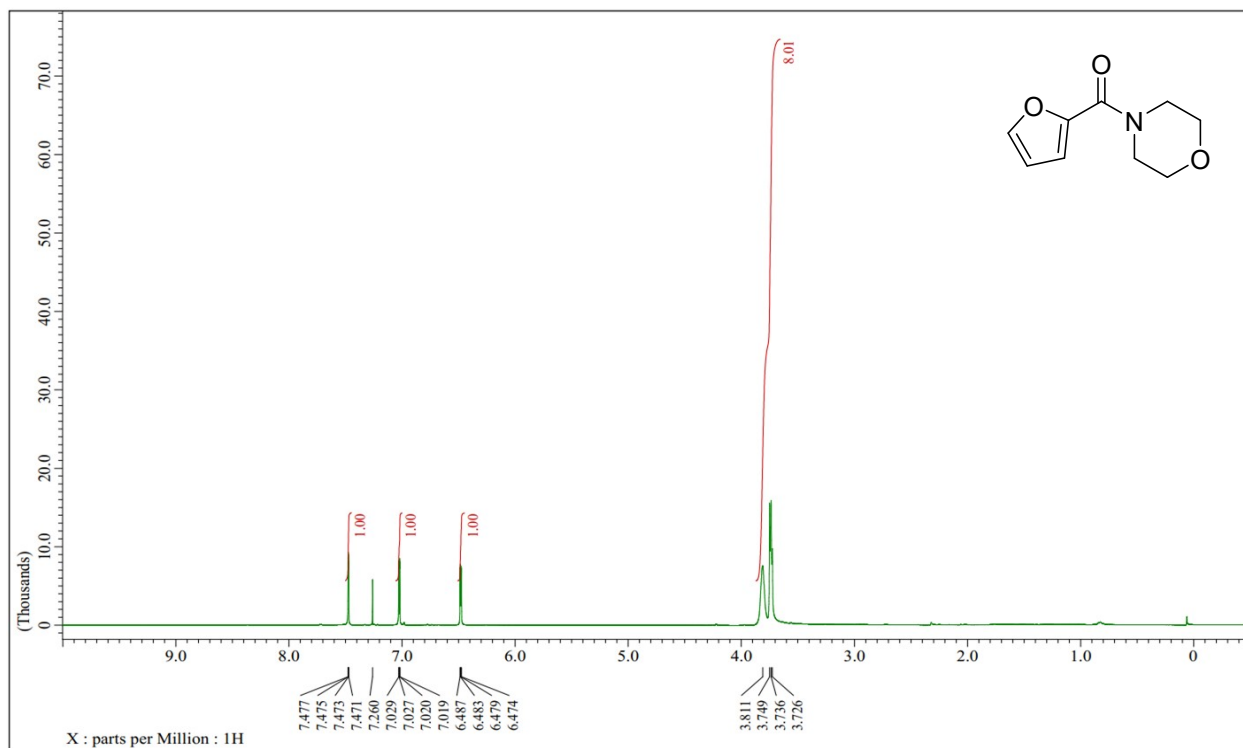


¹H NMR spectrum of morpholino(naphthalen-1-yl)methanone (3n)

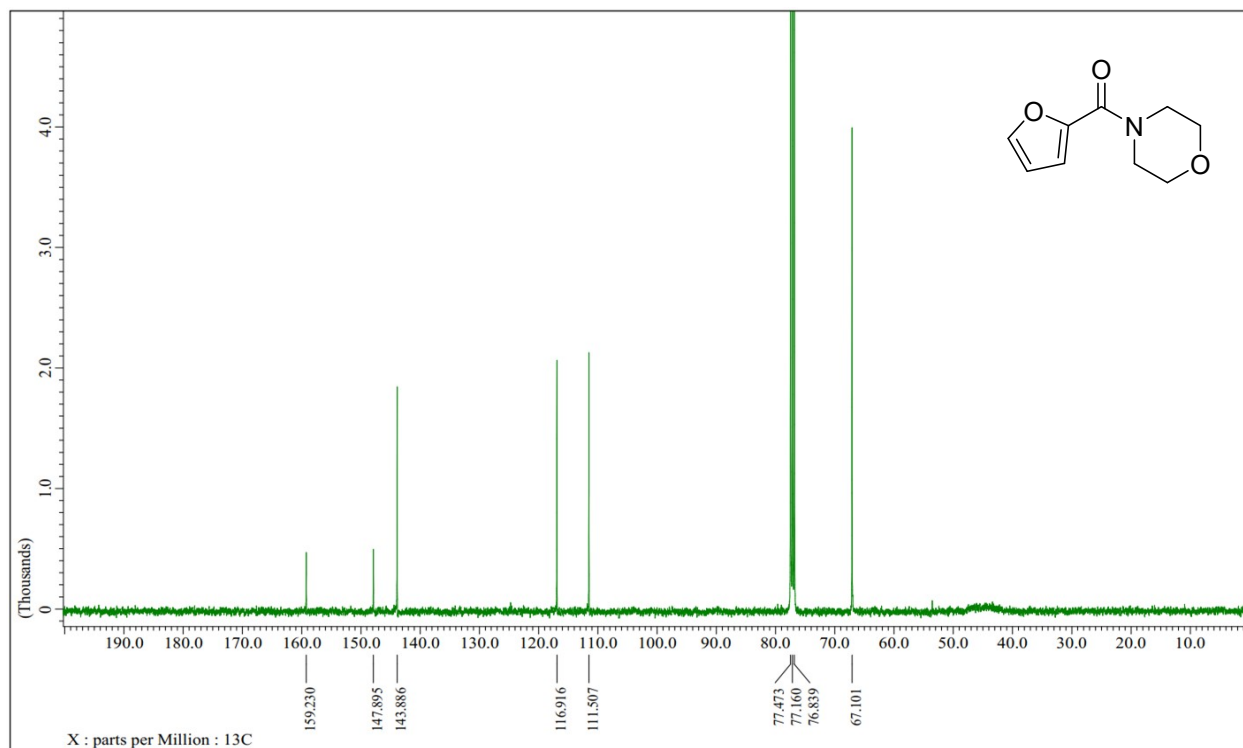


¹³C NMR spectrum of morpholino(naphthalen-1-yl)methanone (3n)

Furan-2-yl(morpholino)methanone (3o)

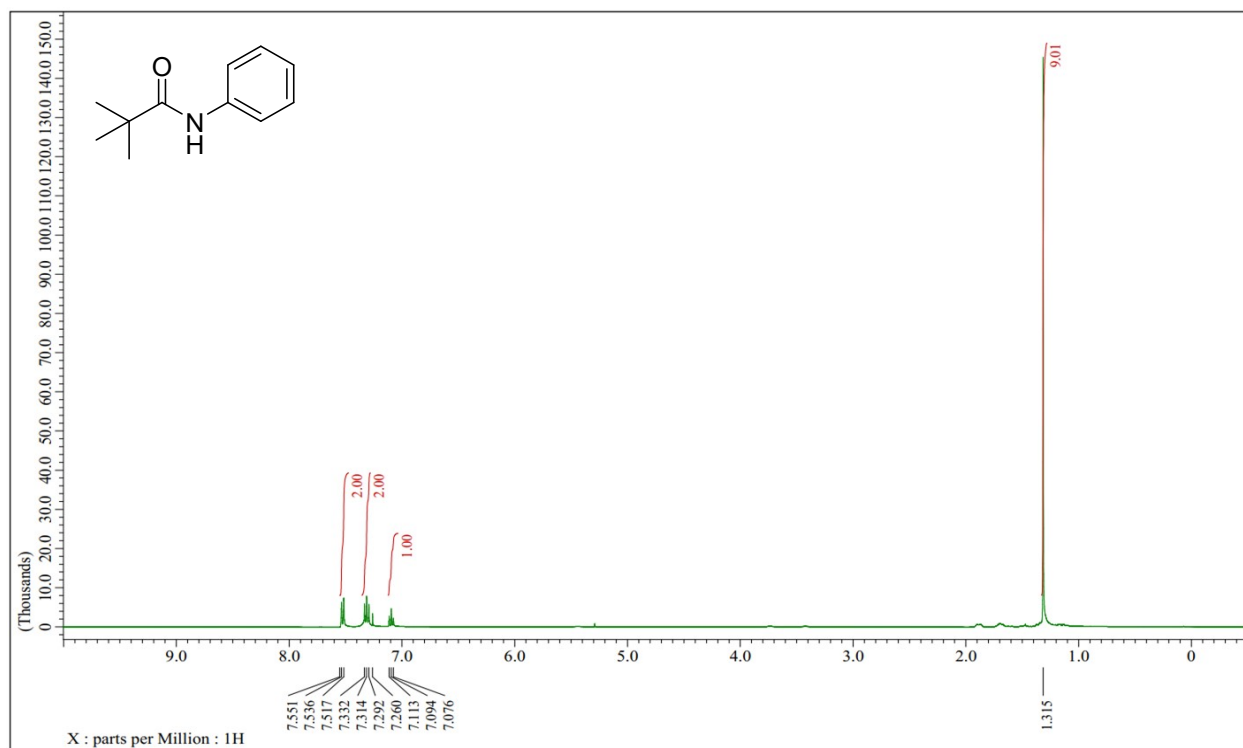


¹H NMR spectrum of furan-2-yl(morpholino)methanone (3o)

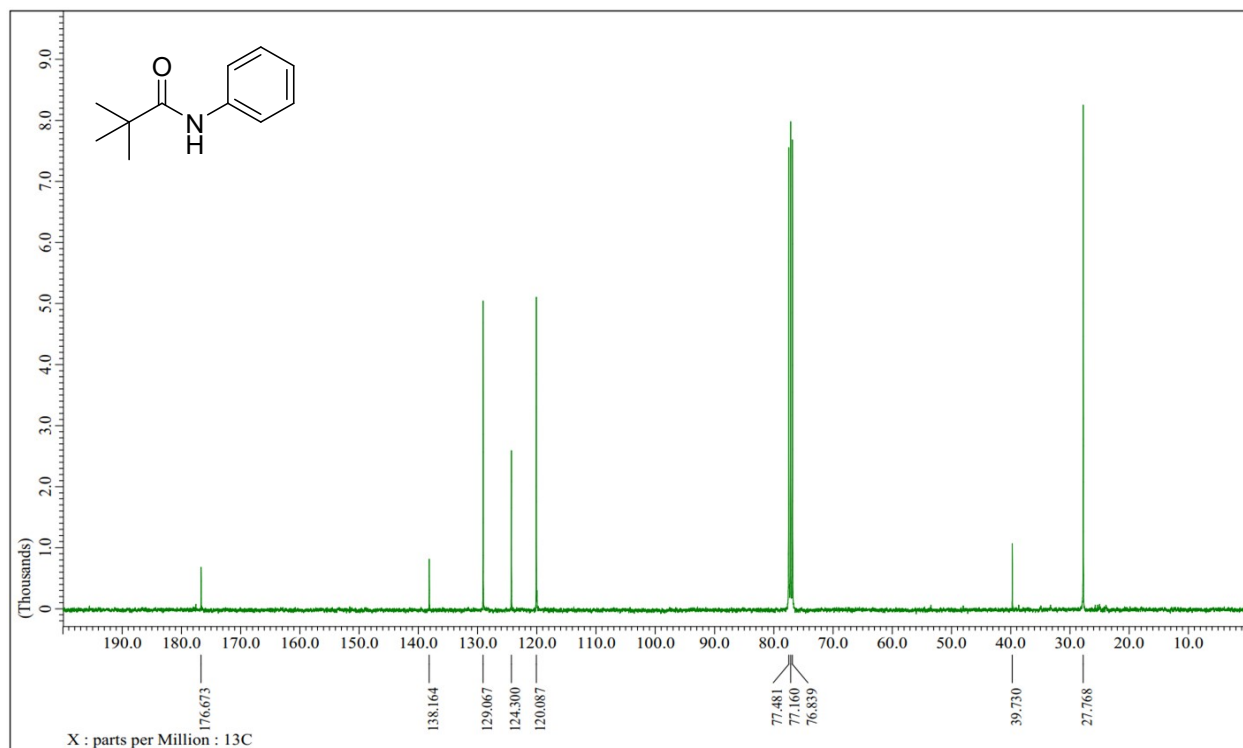


¹³C NMR spectrum of furan-2-yl(morpholino)methanone (3o)

***N*-Phenylpivalamide (4a)**

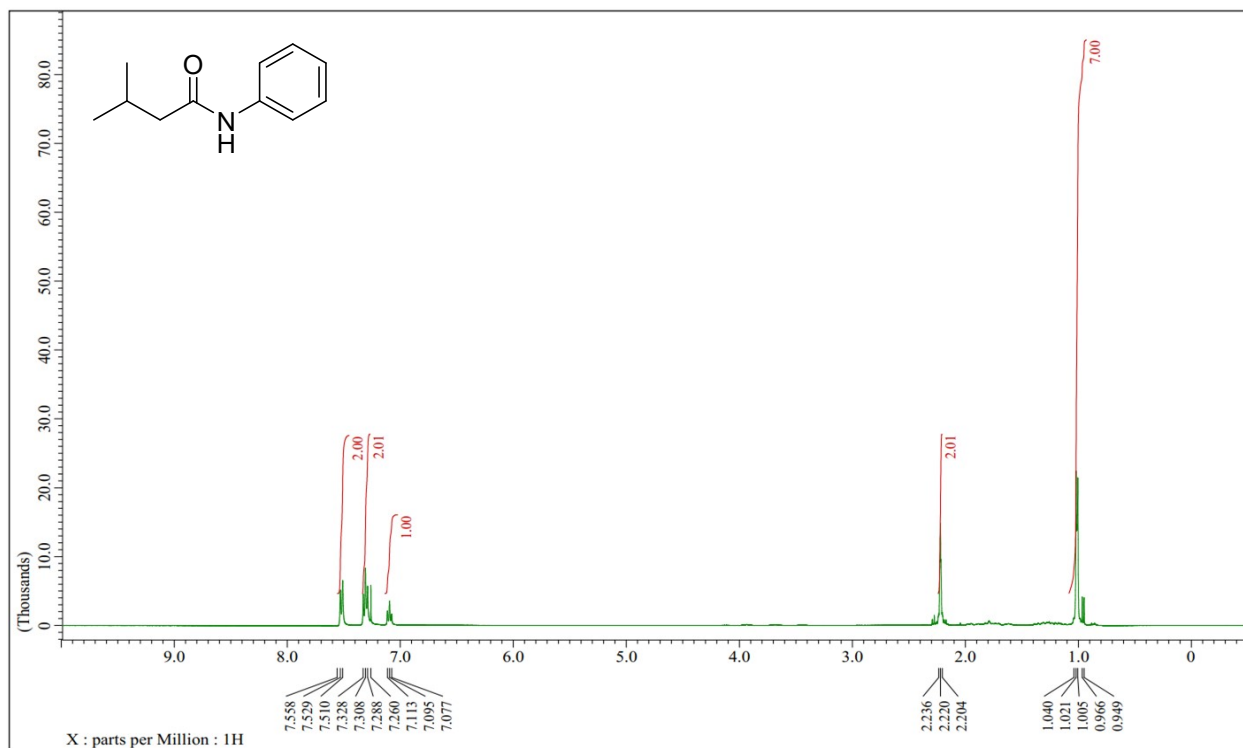


¹H NMR spectrum of *N*-phenylpivalamide (4a)

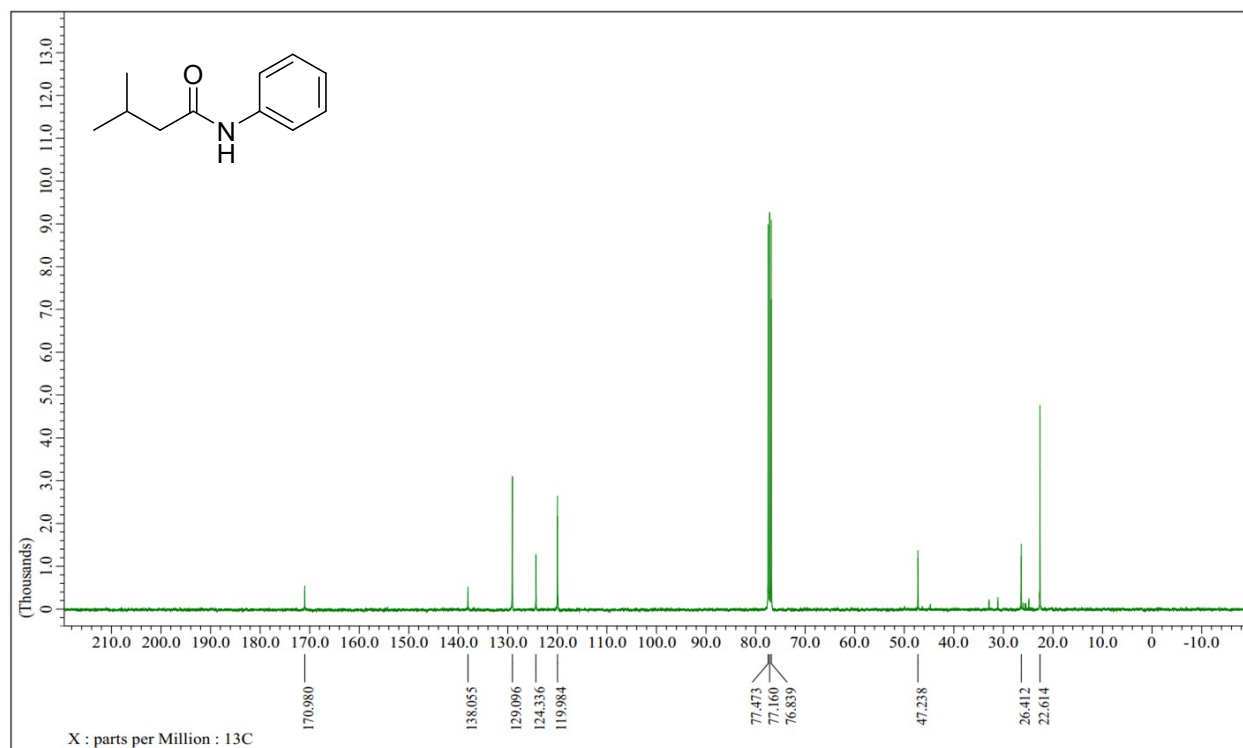


¹³C NMR spectrum of *N*-phenylpivalamide (4a)

3-Methyl-*N*-phenylbutanamide (4b)

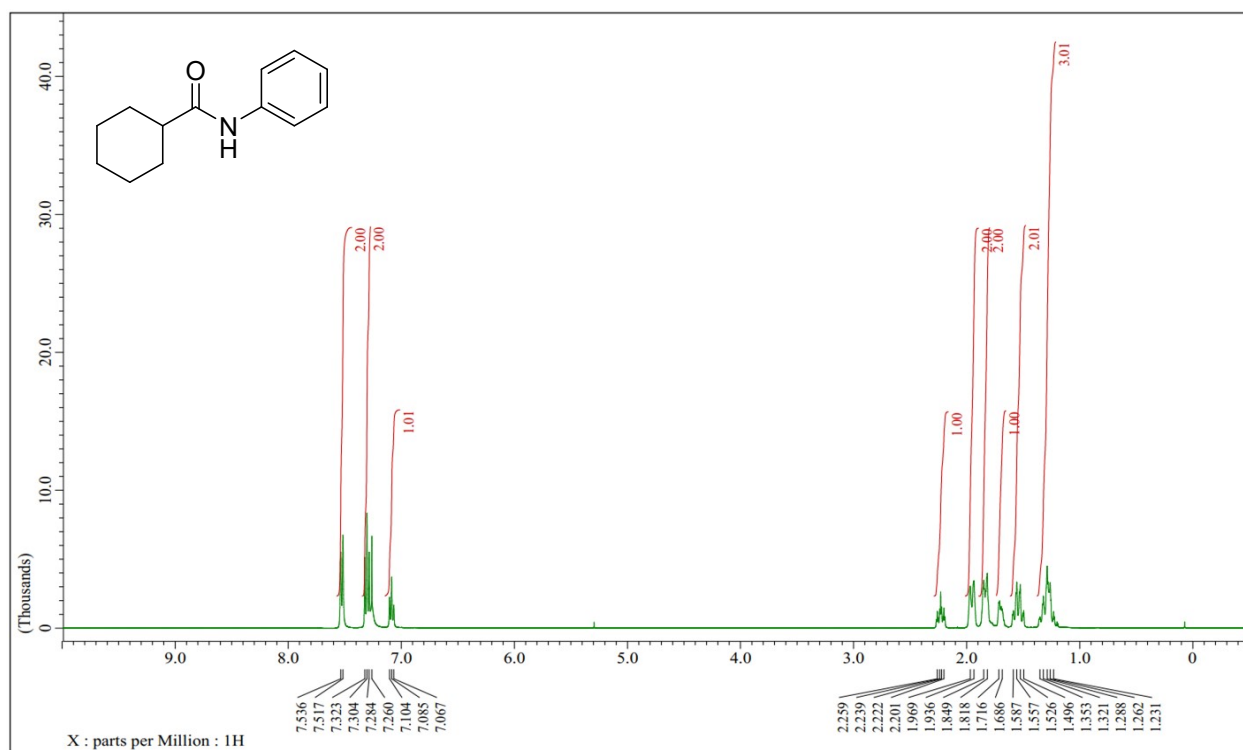


¹H NMR spectrum of 3-methyl-*N*-phenylbutanamide (4b)

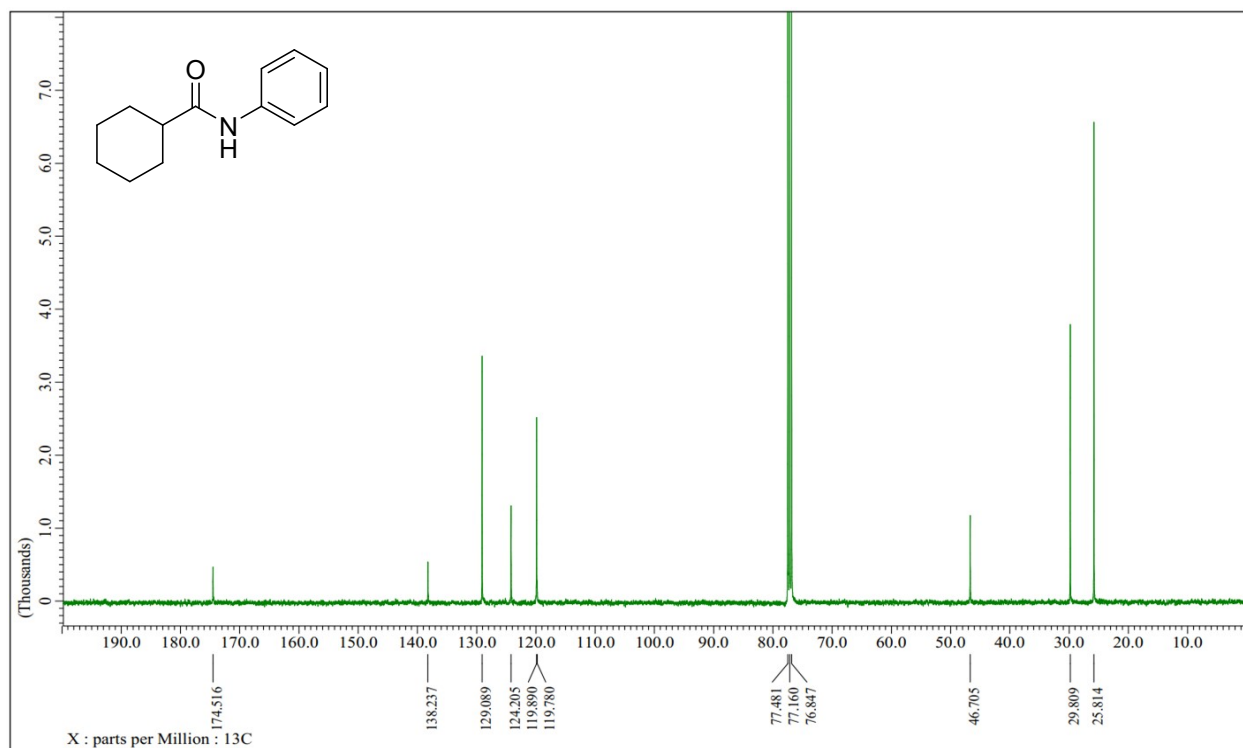


¹³C NMR spectrum of 3-methyl-*N*-phenylbutanamide (4b)

***N*-Phenylcyclohexanecarboxamide (4c)**

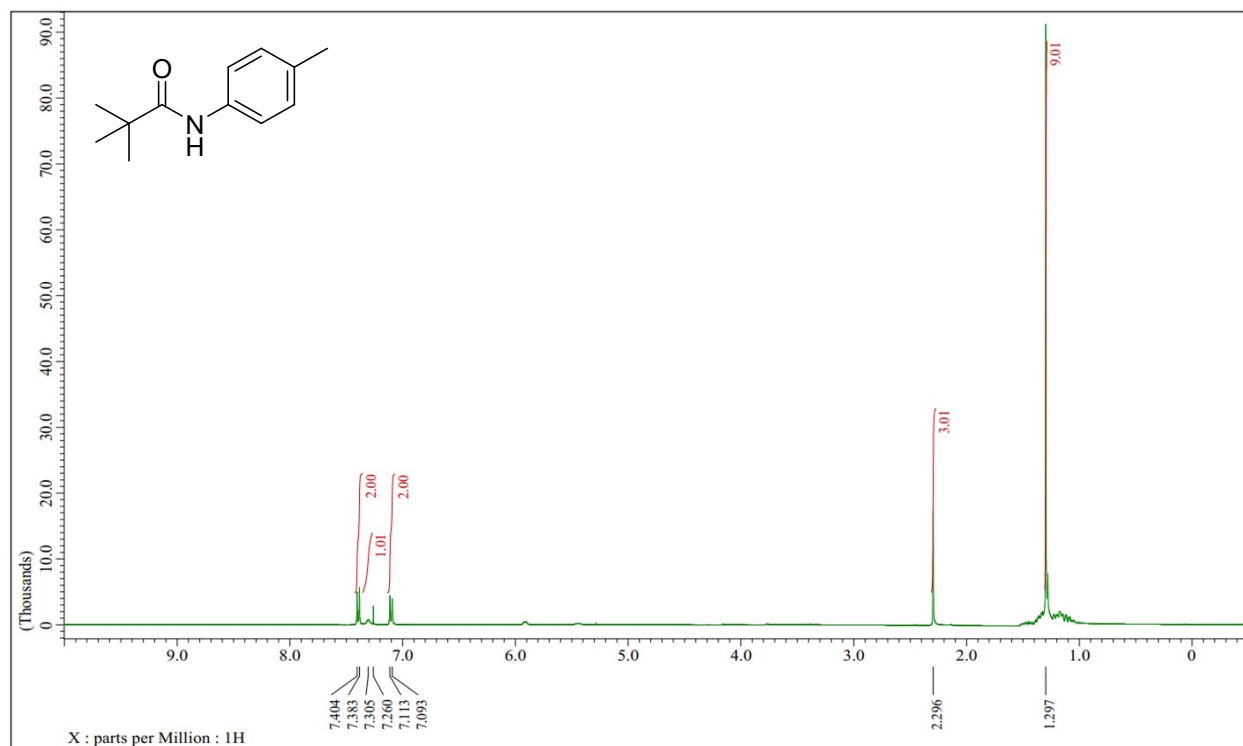


¹H NMR spectrum of *N*-phenylcyclohexanecarboxamide (4c)

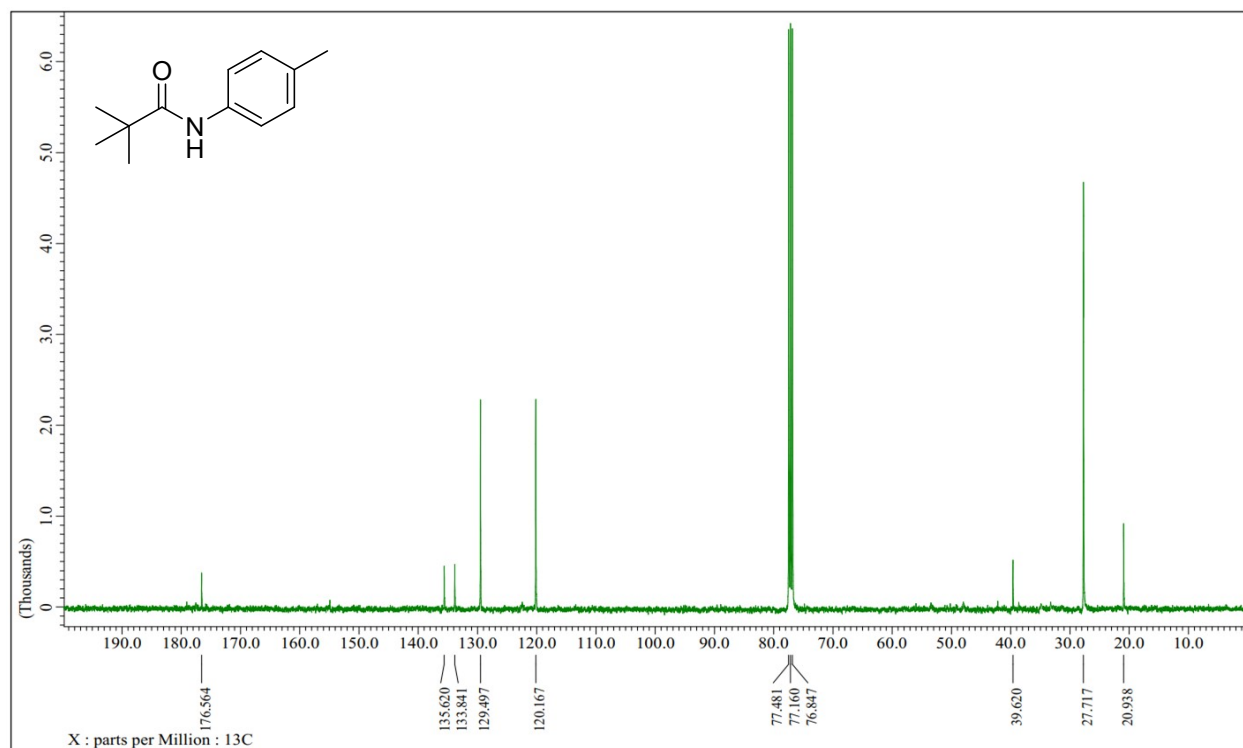


¹³C NMR spectrum of *N*-phenylcyclohexanecarboxamide (4c)

N-(p-tolyl)pivalamide (4d)

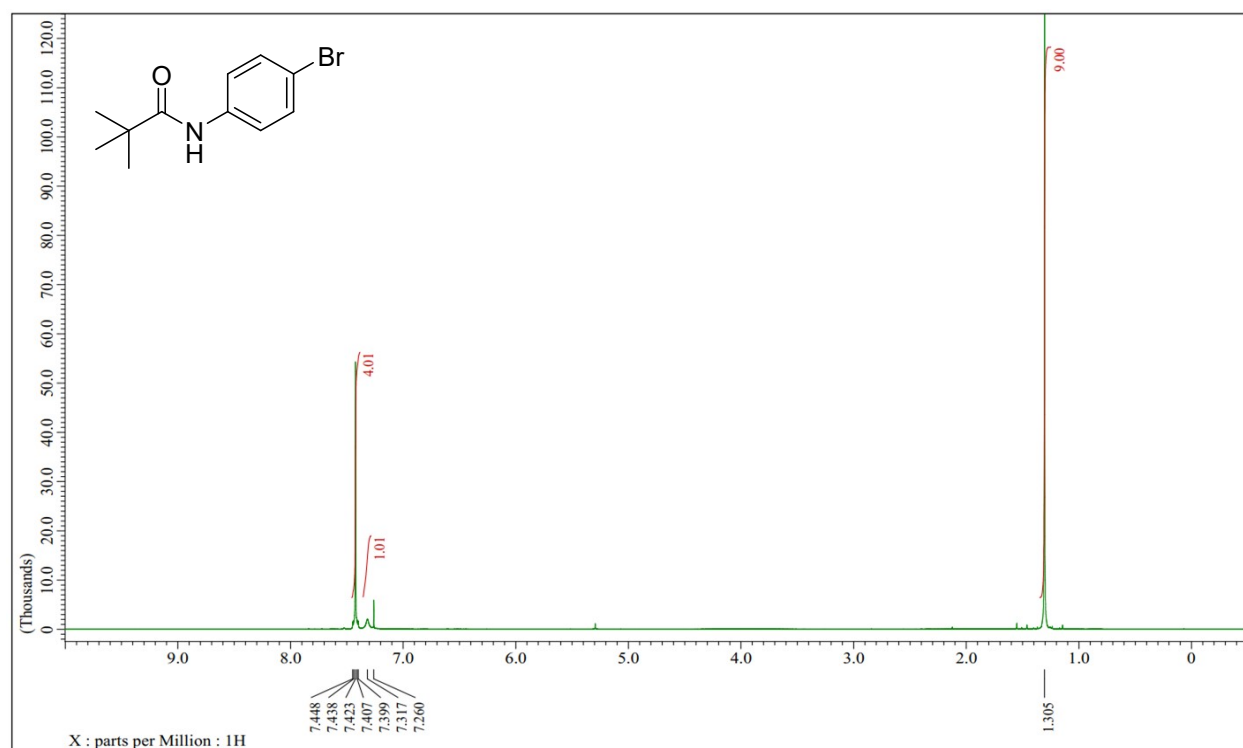


¹H NMR spectrum of N-(p-tolyl)pivalamide (4d)

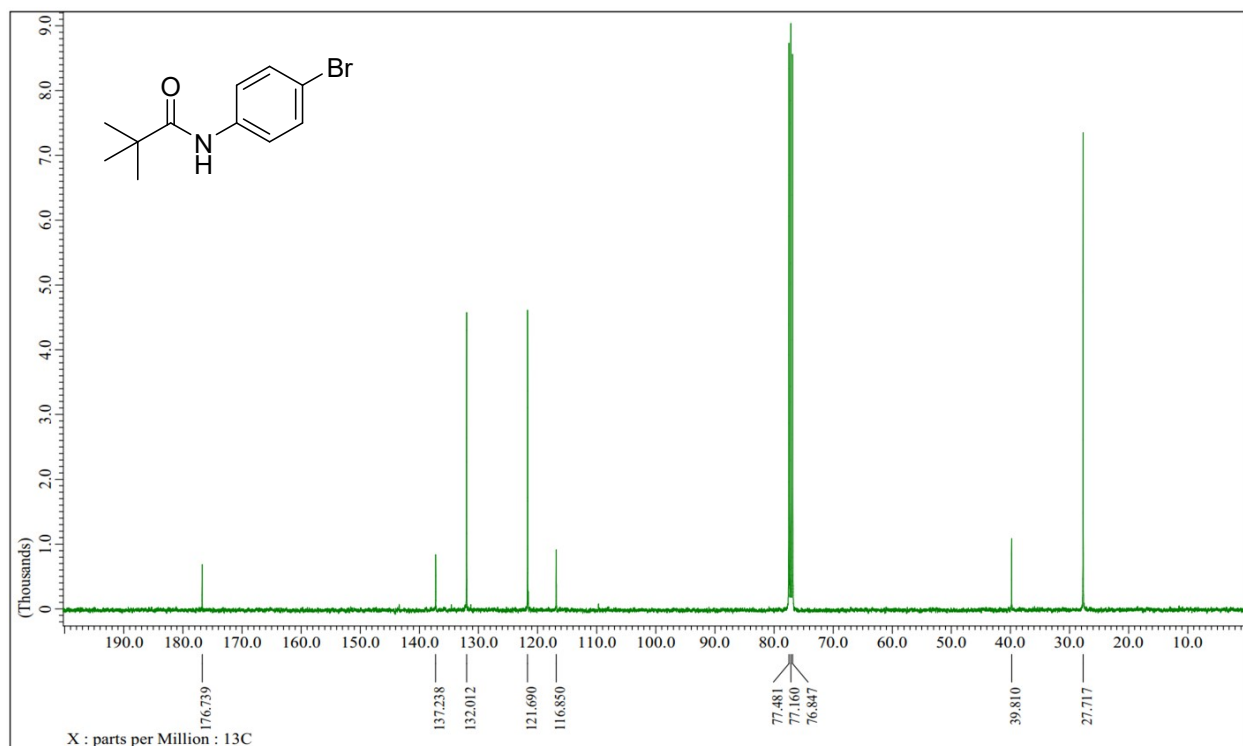


¹³C NMR spectrum of N-(p-tolyl)pivalamide (4d)

N-(4-bromophenyl)pivalamide (4e)

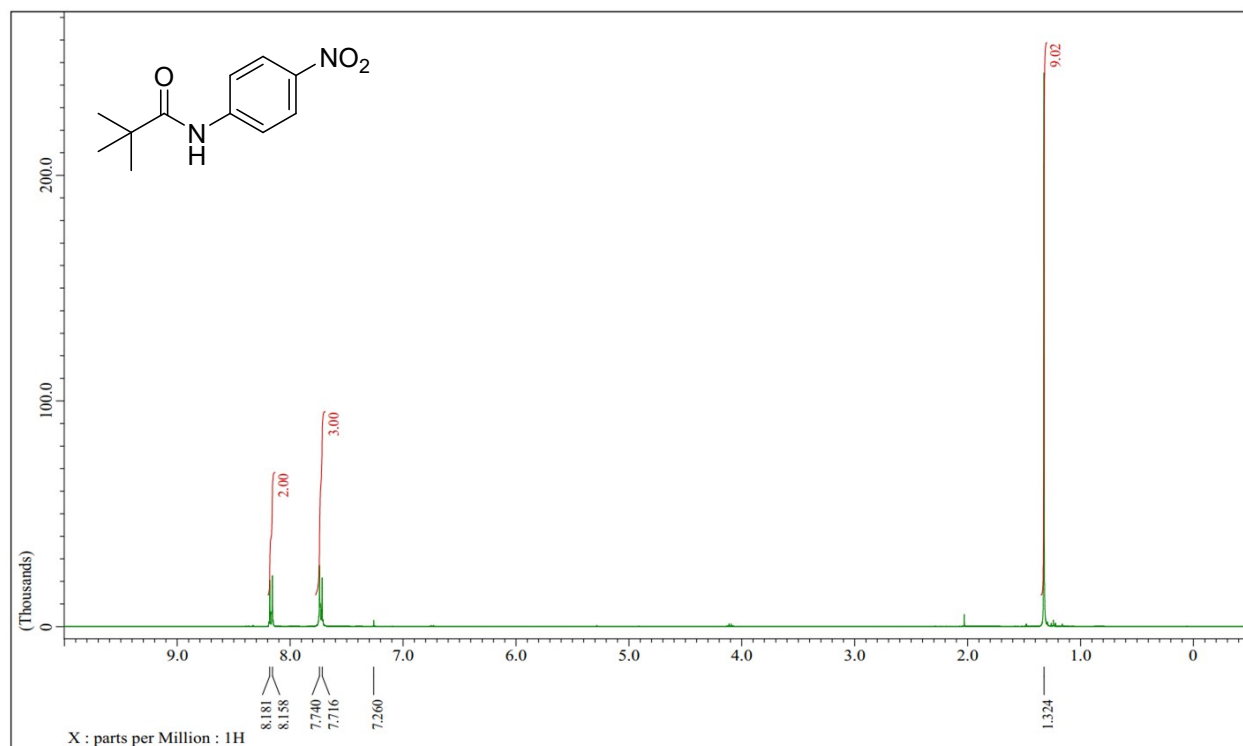


¹H NMR spectrum of N-(4-bromophenyl)pivalamide (4e)

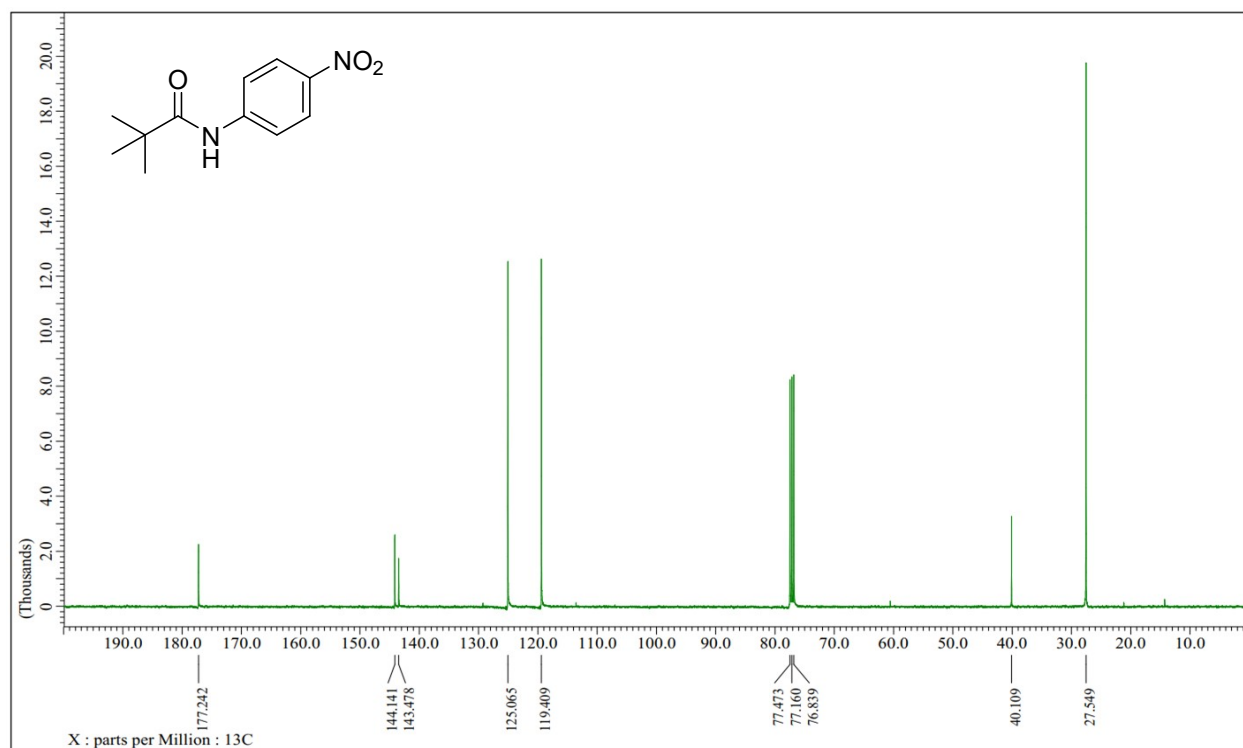


¹³C NMR spectrum of N-(4-bromophenyl)pivalamide (4e)

N-(4-nitrophenyl)pivalamide (4f)

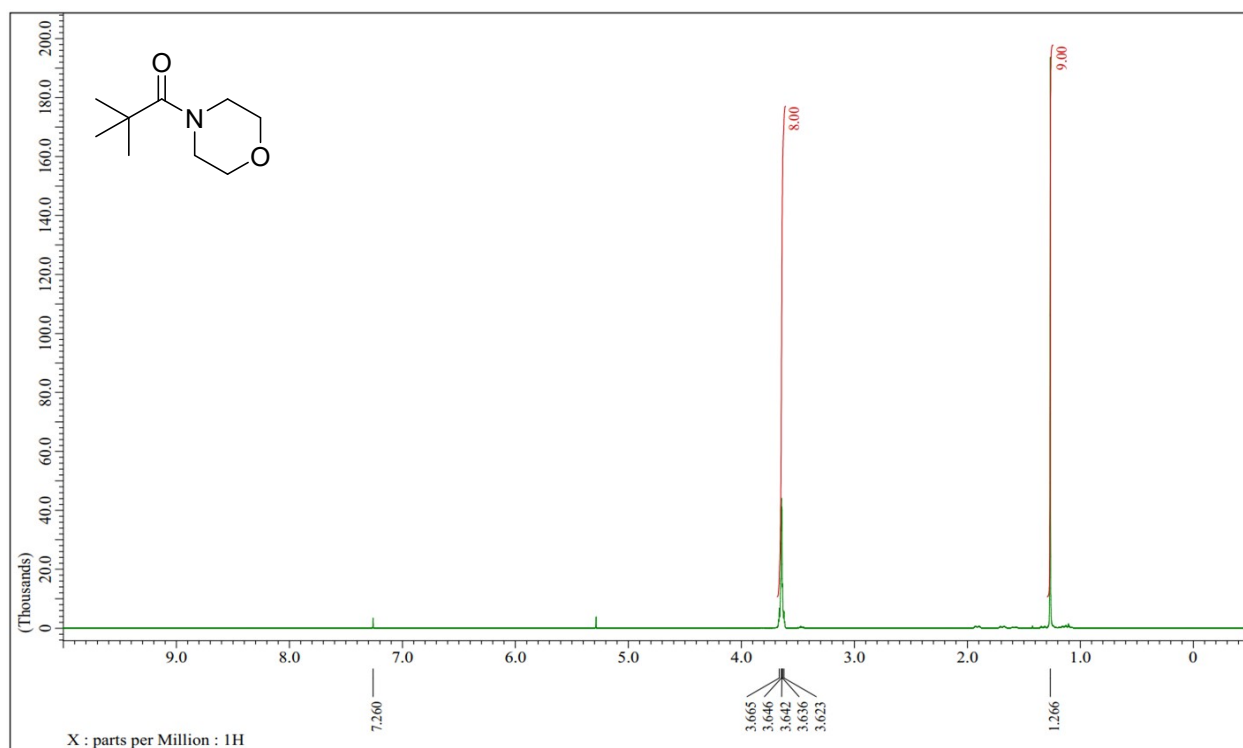


¹H NMR spectrum of N-(4-nitrophenyl)pivalamide (4f)

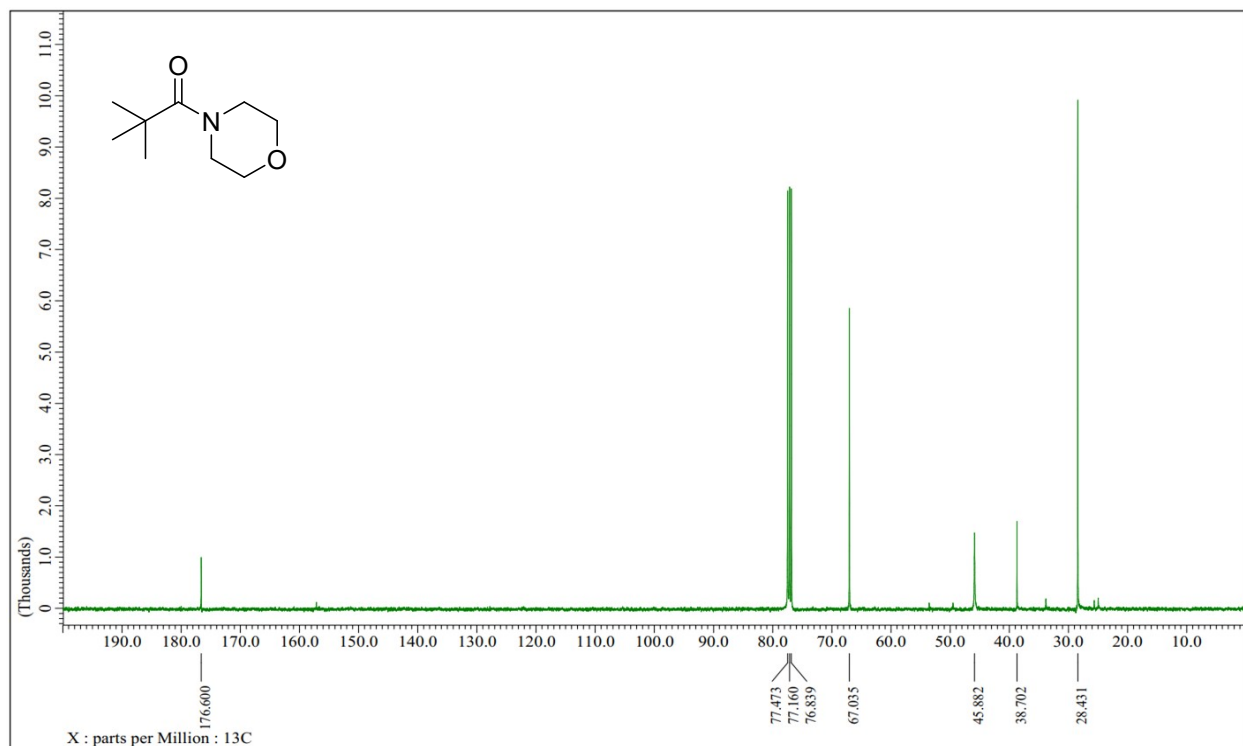


¹³C NMR spectrum of N-(4-nitrophenyl)pivalamide (4f)

2,2-dimethyl-1-morpholinopropan-1-one (4g)

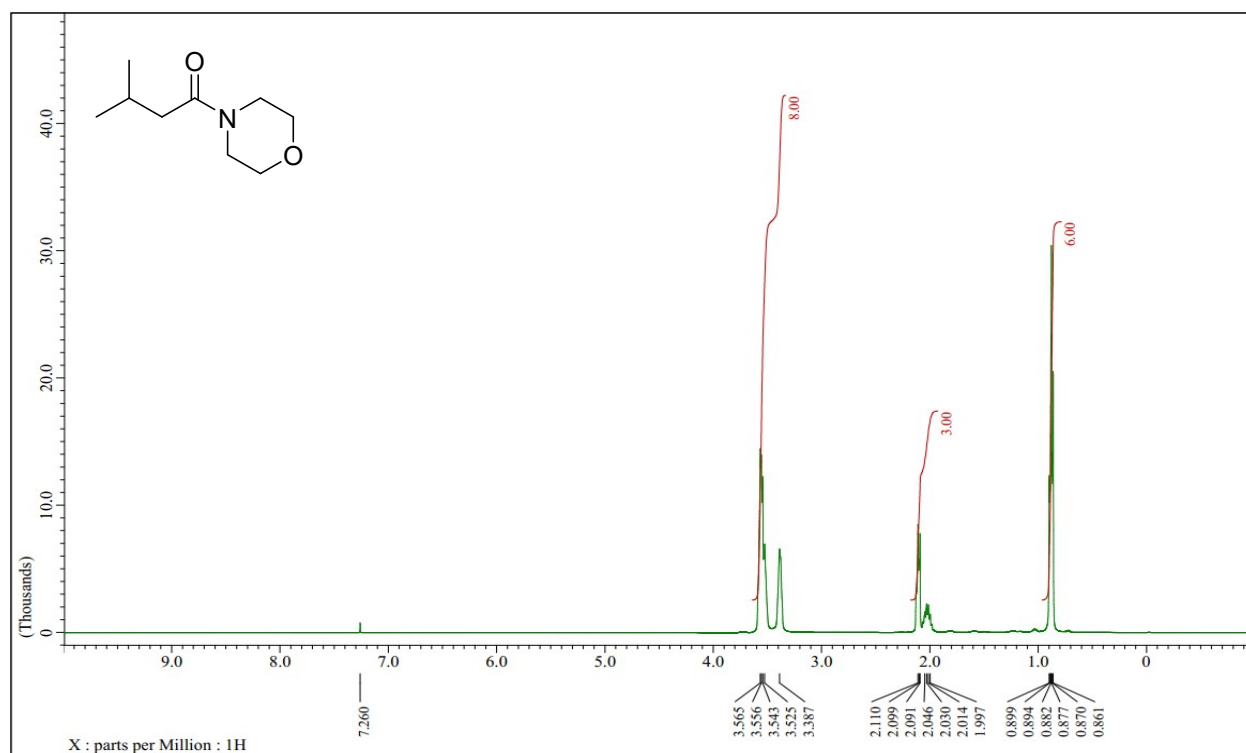


¹H NMR spectrum of 2,2-dimethyl-1-morpholinopropan-1-one (4g)

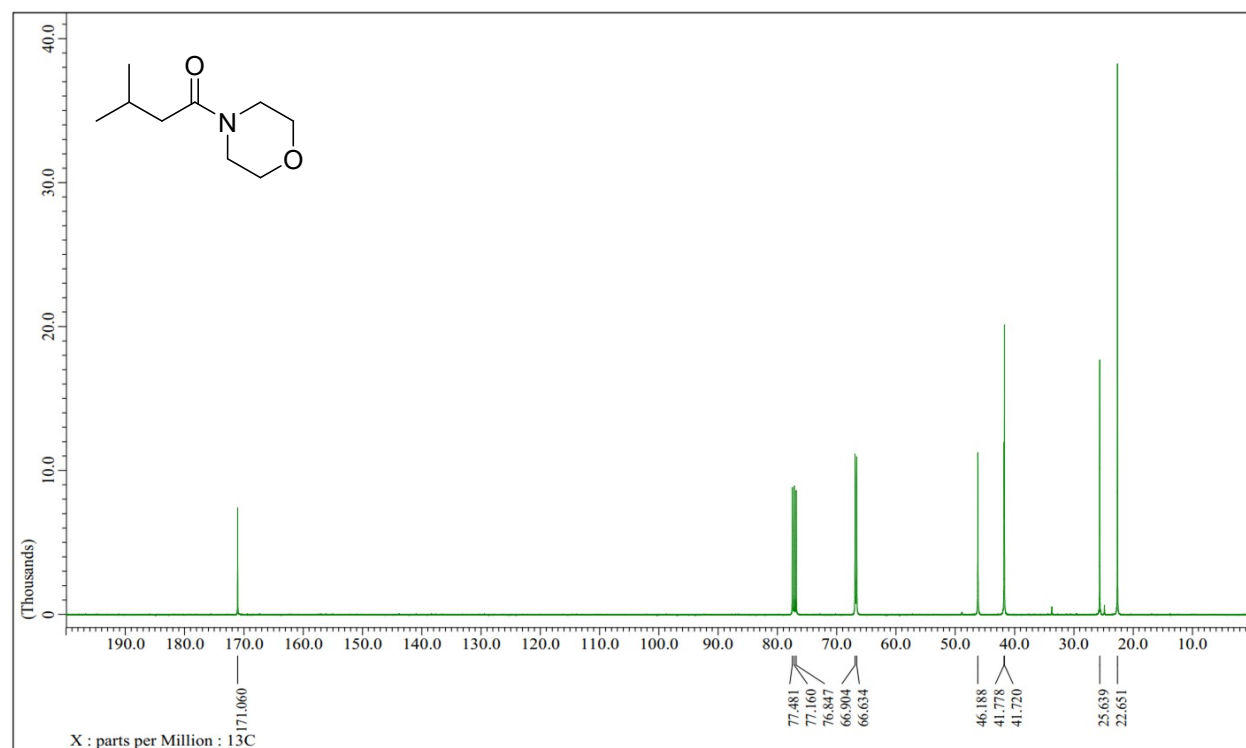


¹³C NMR spectrum of 2,2-dimethyl-1-morpholinopropan-1-one (4g)

3-Methyl-1-morpholinobutan-1-one (4h)

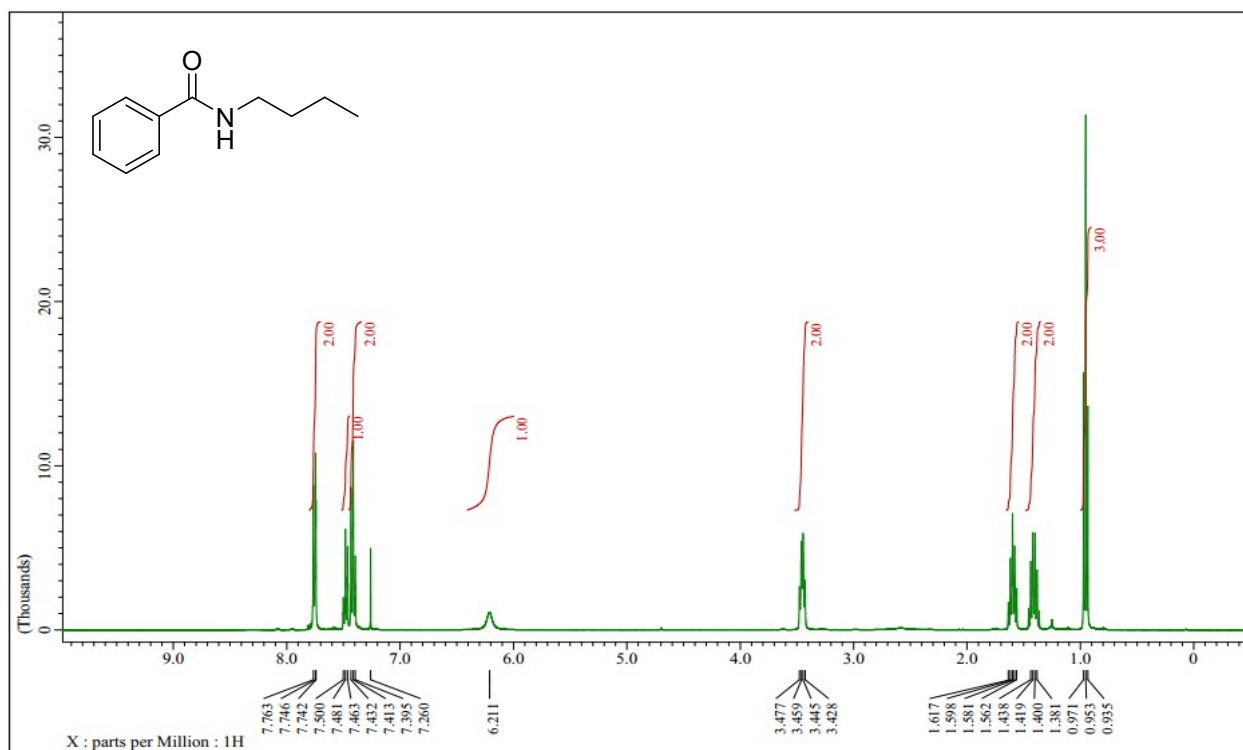


¹H NMR spectrum of 3-methyl-1-morpholinobutan-1-one (4h)

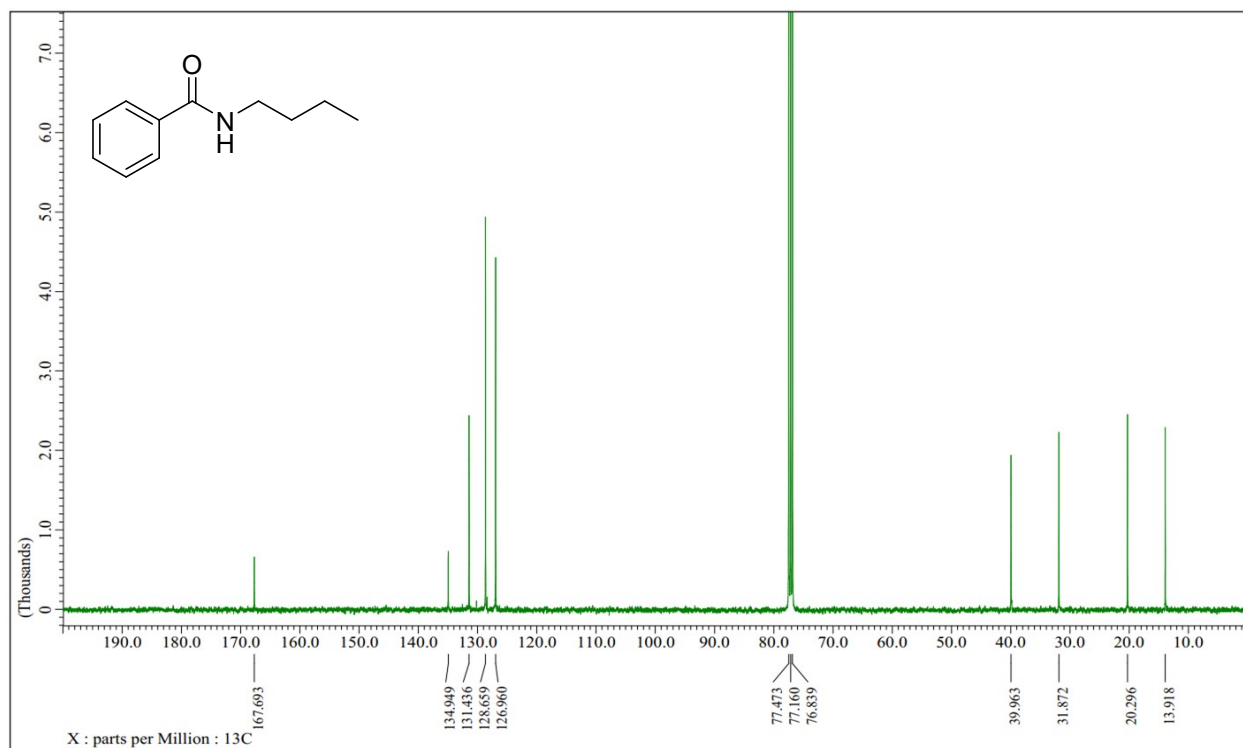


¹³C NMR spectrum of 3-methyl-1-morpholinobutan-1-one (4h)

N-Butylbenzamide (5a)

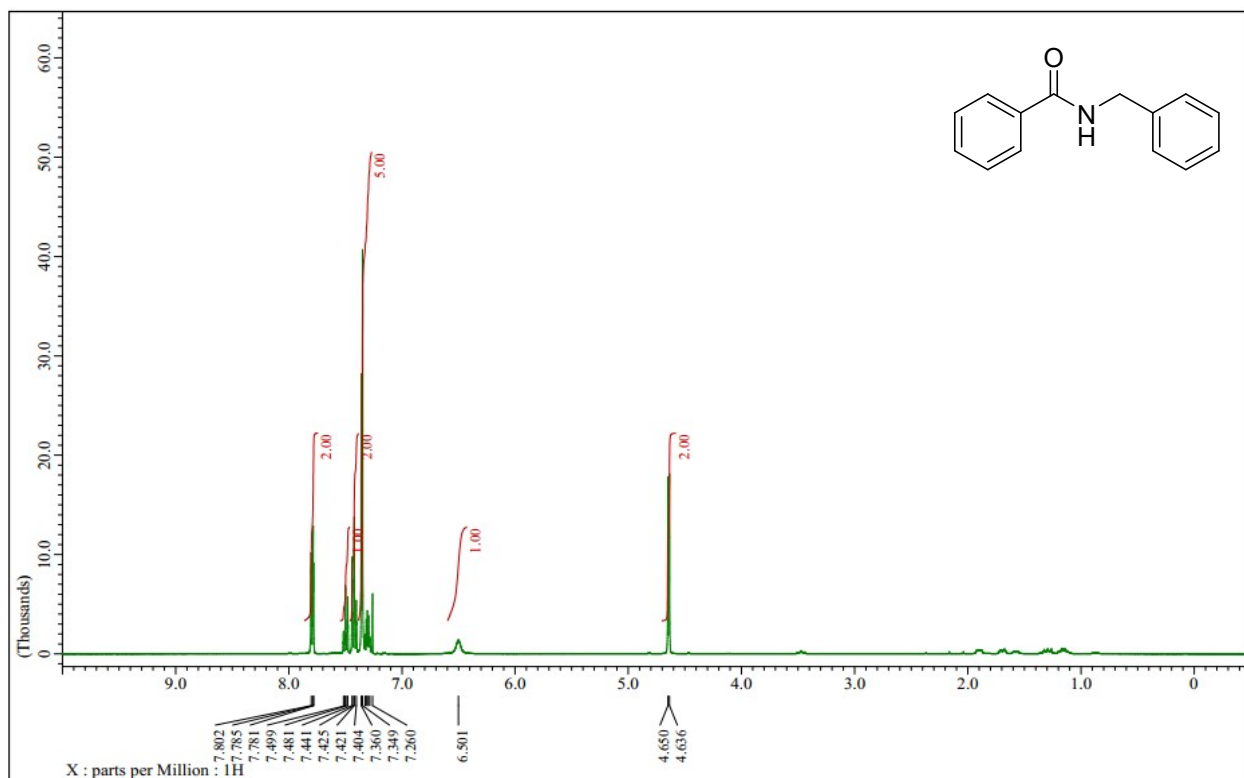


¹H NMR spectrum of *N*-butylbenzamide (5a)

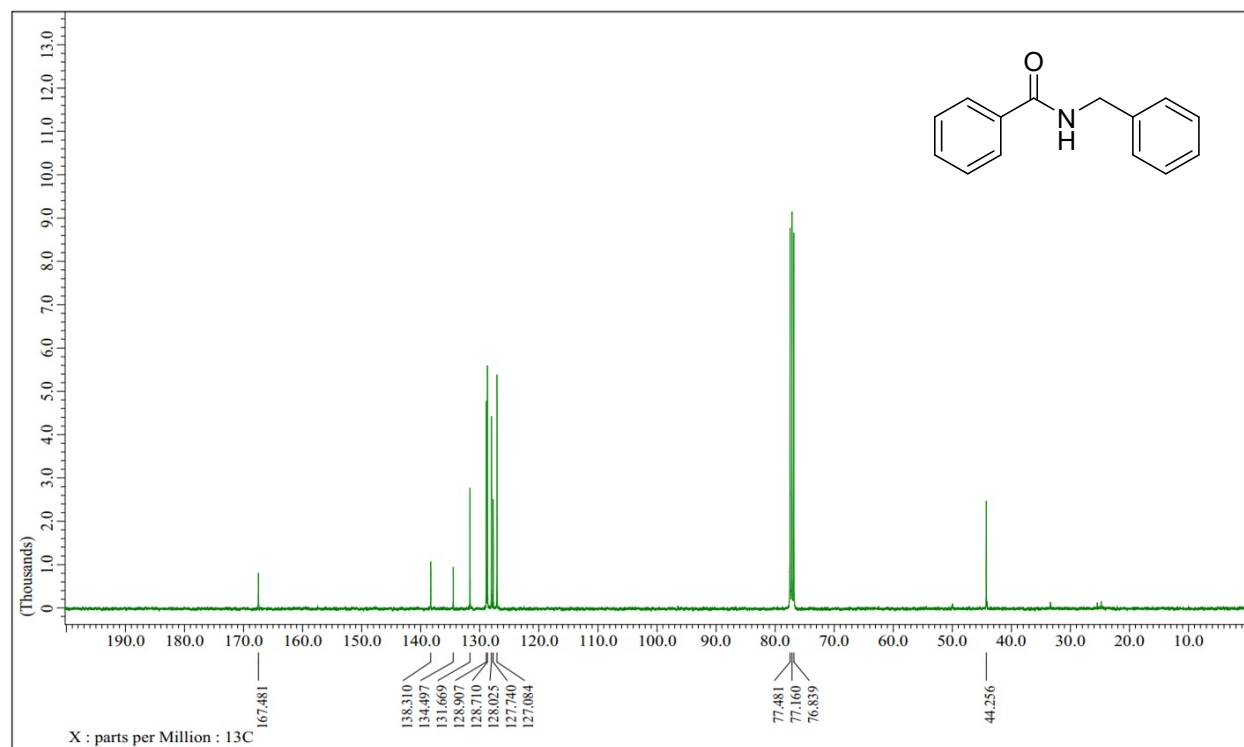


¹³C NMR spectrum of *N*-butylbenzamide (5a)

***N*-Benzylbenzamide (5b)**

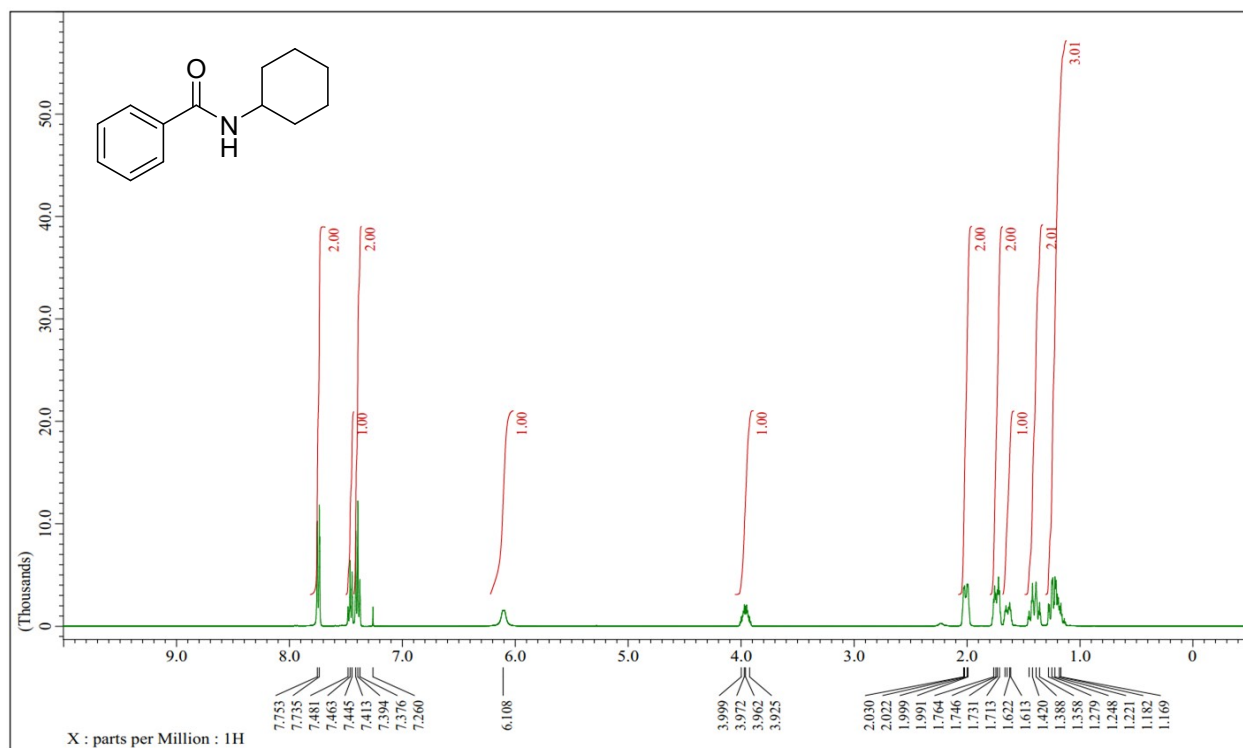


¹H NMR spectrum of *N*-benzylbenzamide (5b)

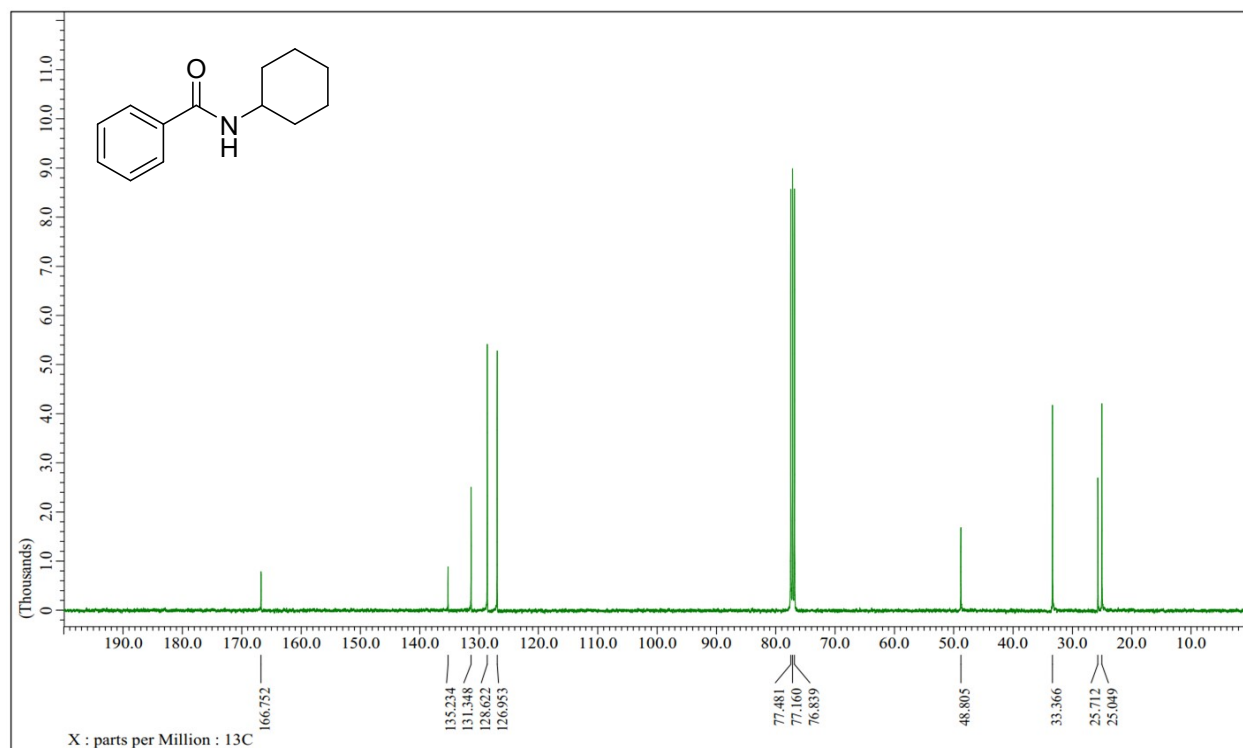


¹³C NMR spectrum of *N*-benzylbenzamide (5b)

N-Cyclohexylbenzamide (**5c**)

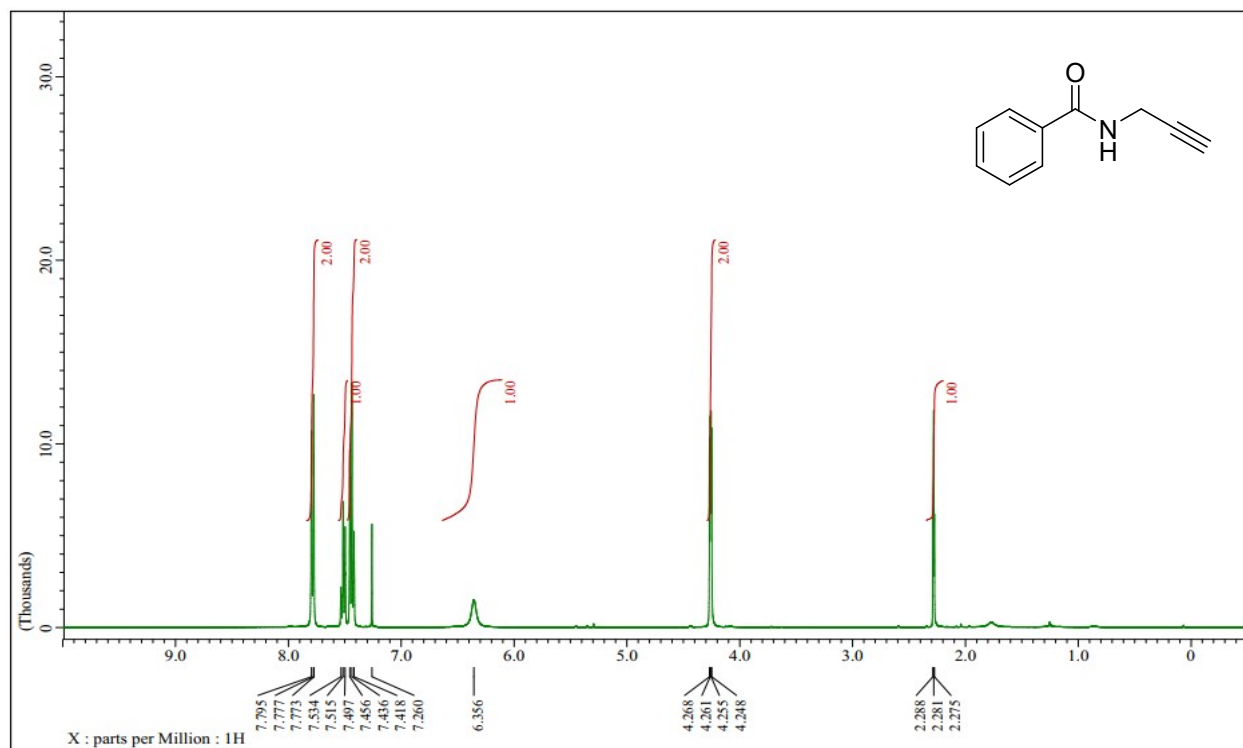


¹H NMR spectrum of *N*-cyclohexylbenzamide (**5c**)

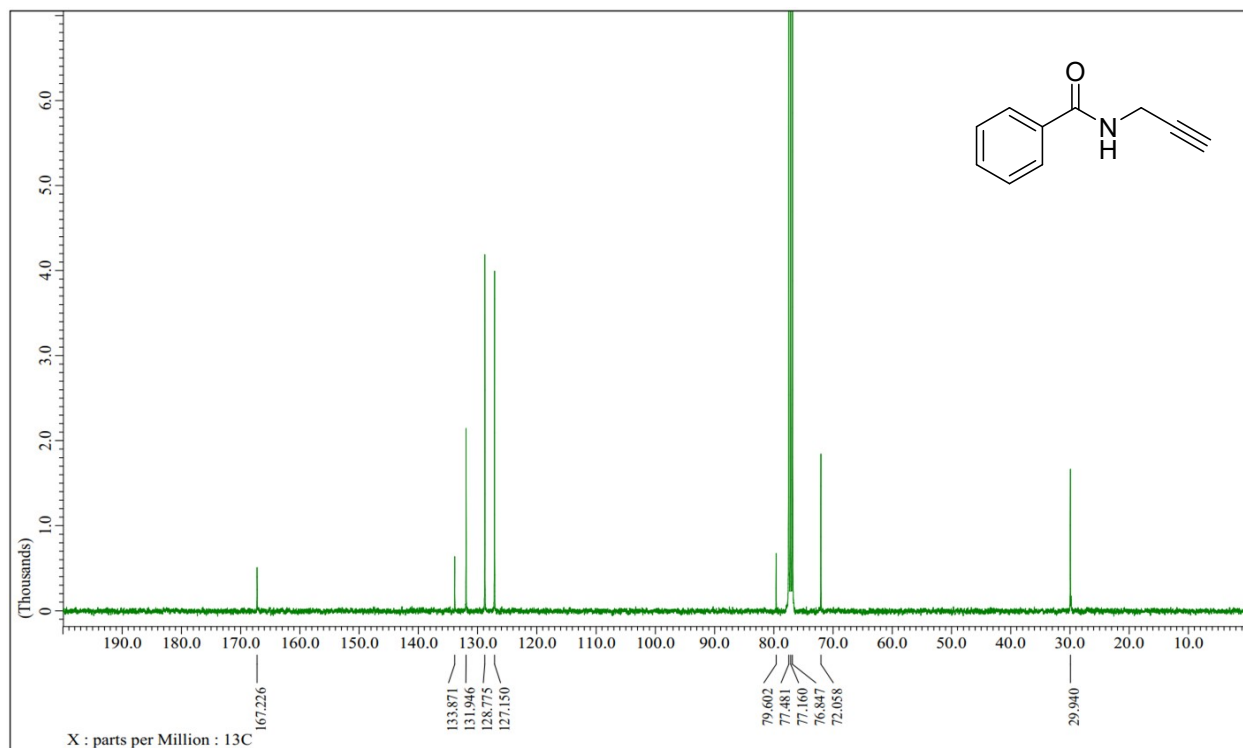


¹³C NMR spectrum of *N*-cyclohexylbenzamide (**5c**)

***N*-(Prop-2-yn-1-yl)benzamide (5d)**

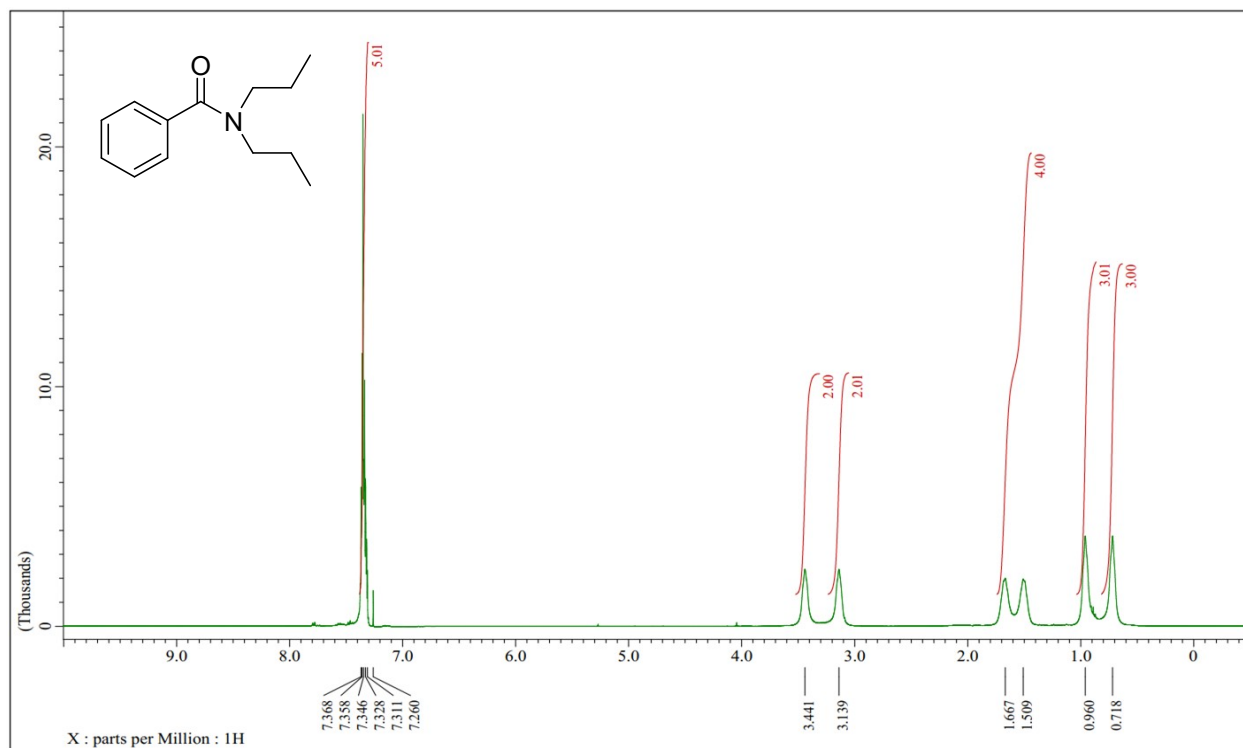


¹H NMR spectrum of *N*-(prop-2-yn-1-yl)benzamide (5d)

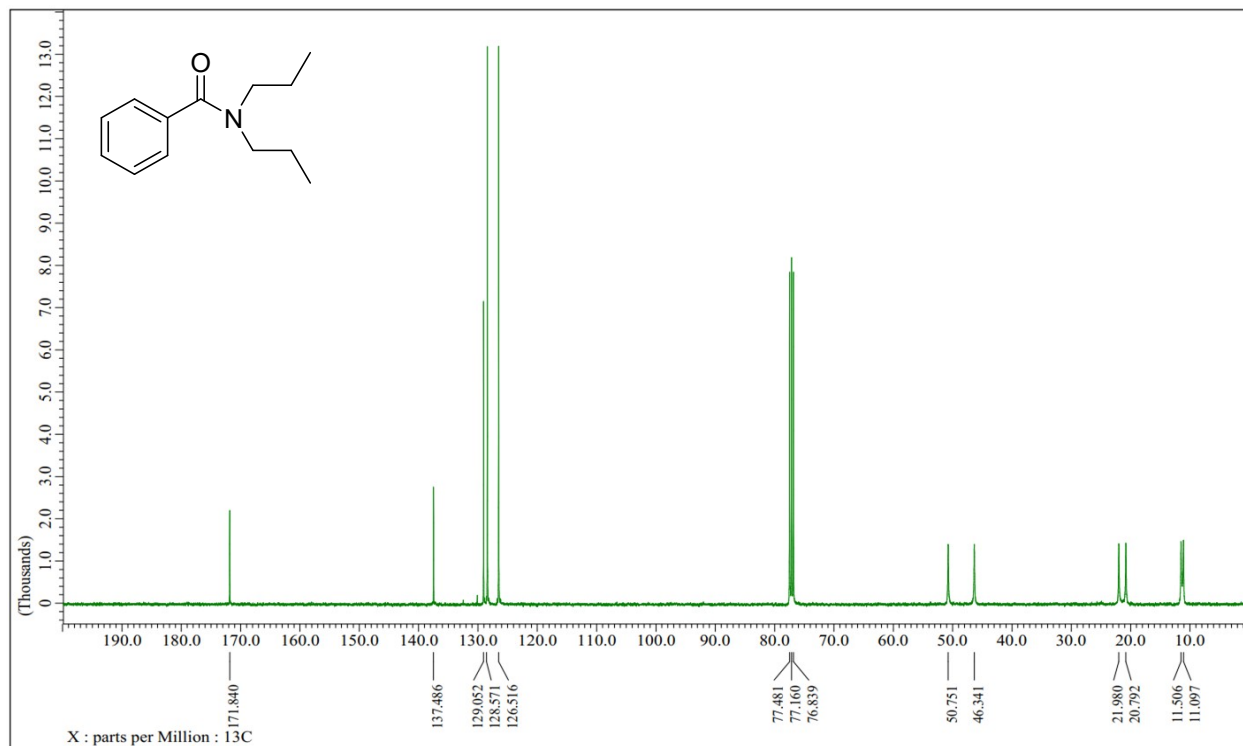


¹³C NMR spectrum of *N*-(prop-2-yn-1-yl)benzamide (5d)

N,N-Dipropylbenzamide (5e)

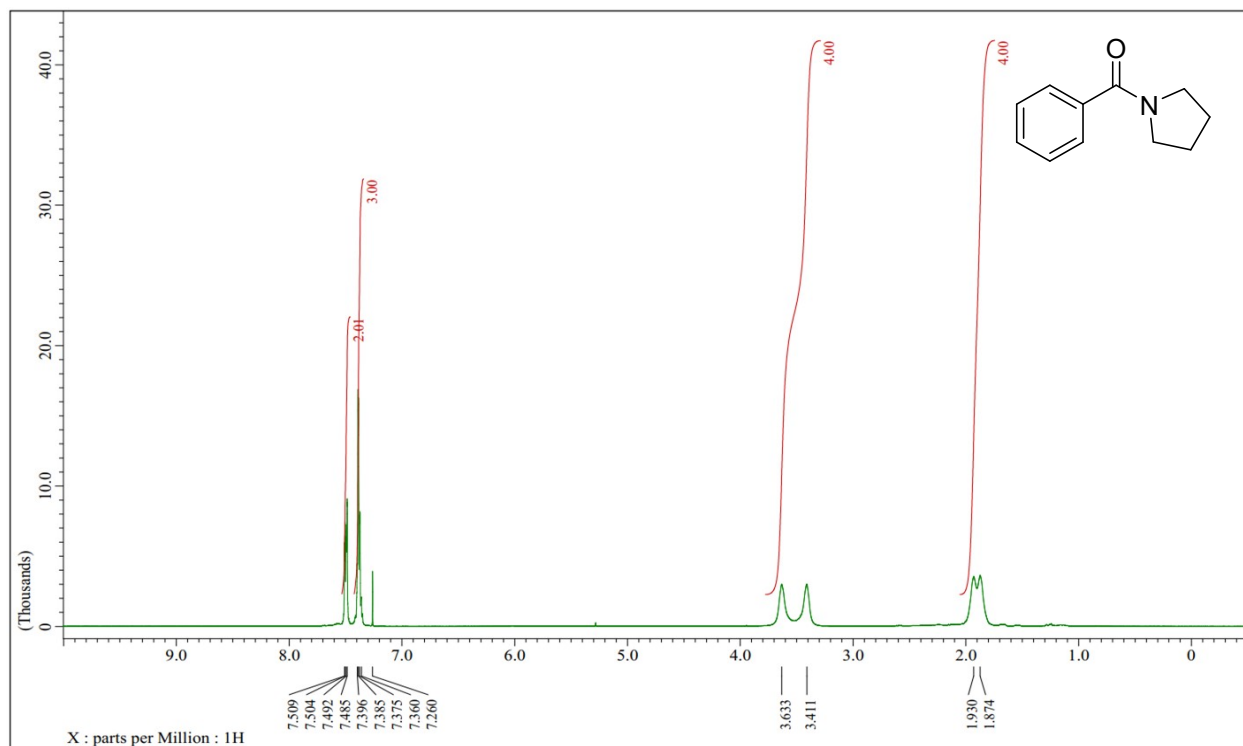


¹H NMR spectrum of *N,N*-dipropylbenzamide (5e)

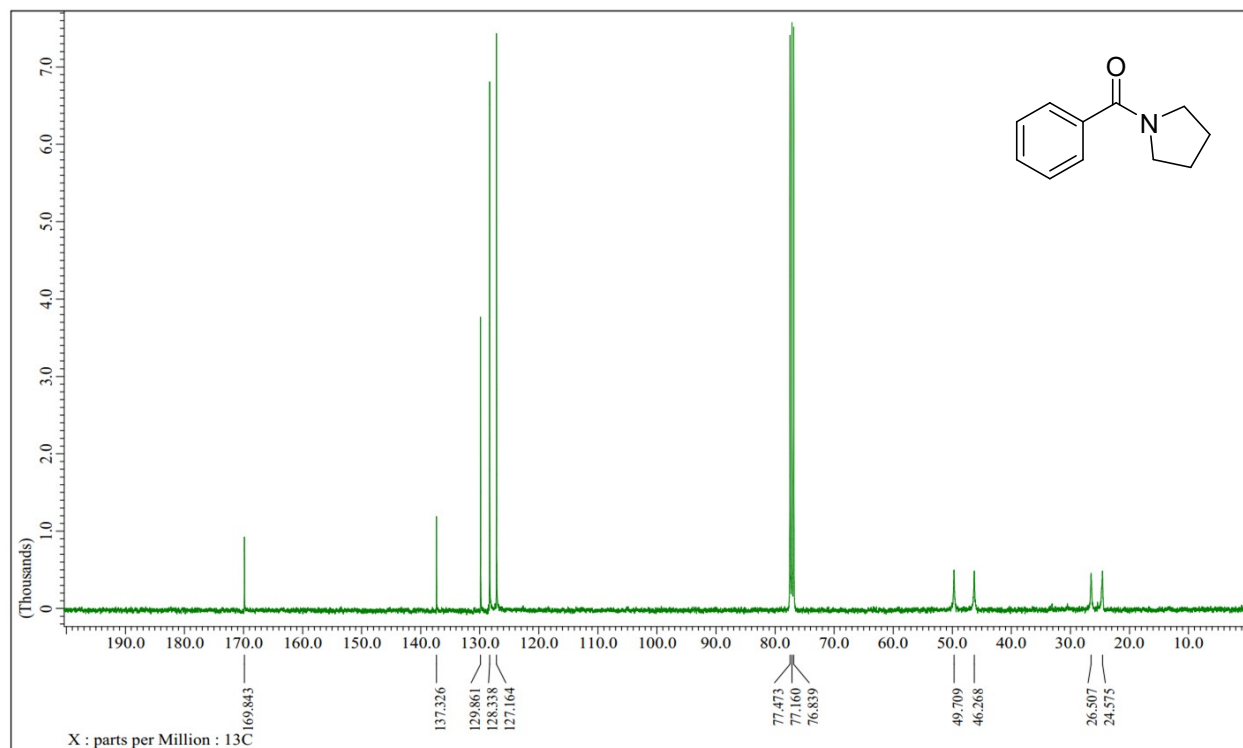


¹³C NMR spectrum of *N,N*-dipropylbenzamide (5e)

Phenyl(pyrrolidin-1-yl)methanone (5f)

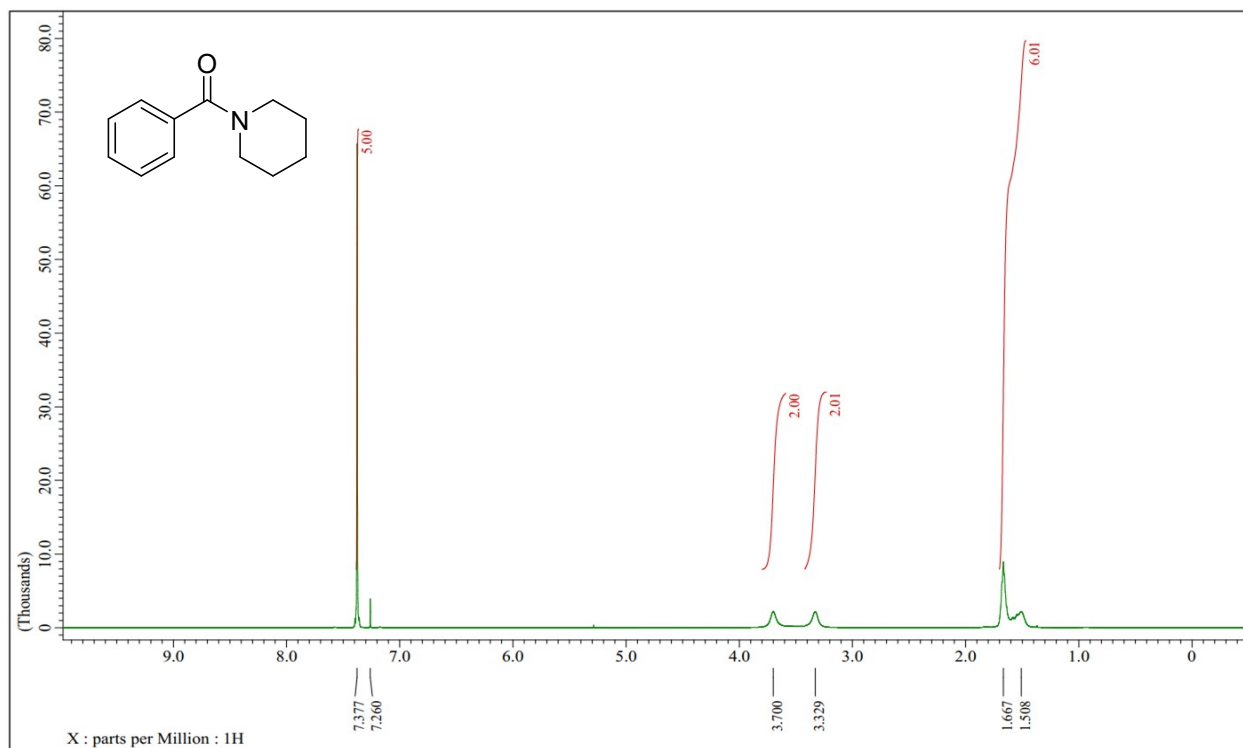


¹H NMR spectrum of phenyl(pyrrolidin-1-yl)methanone (5f)

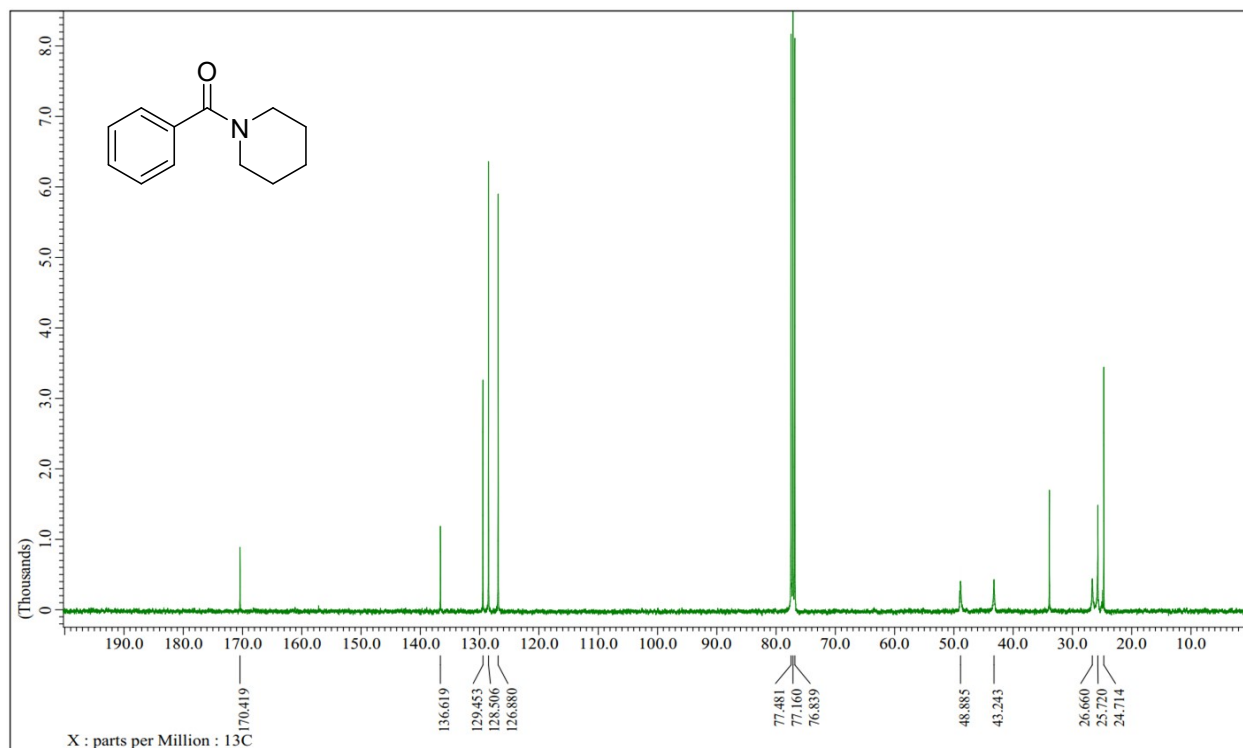


¹³C NMR spectrum of phenyl(pyrrolidin-1-yl)methanone (5f)

Phenyl(piperidin-1-yl)methanone (5g)

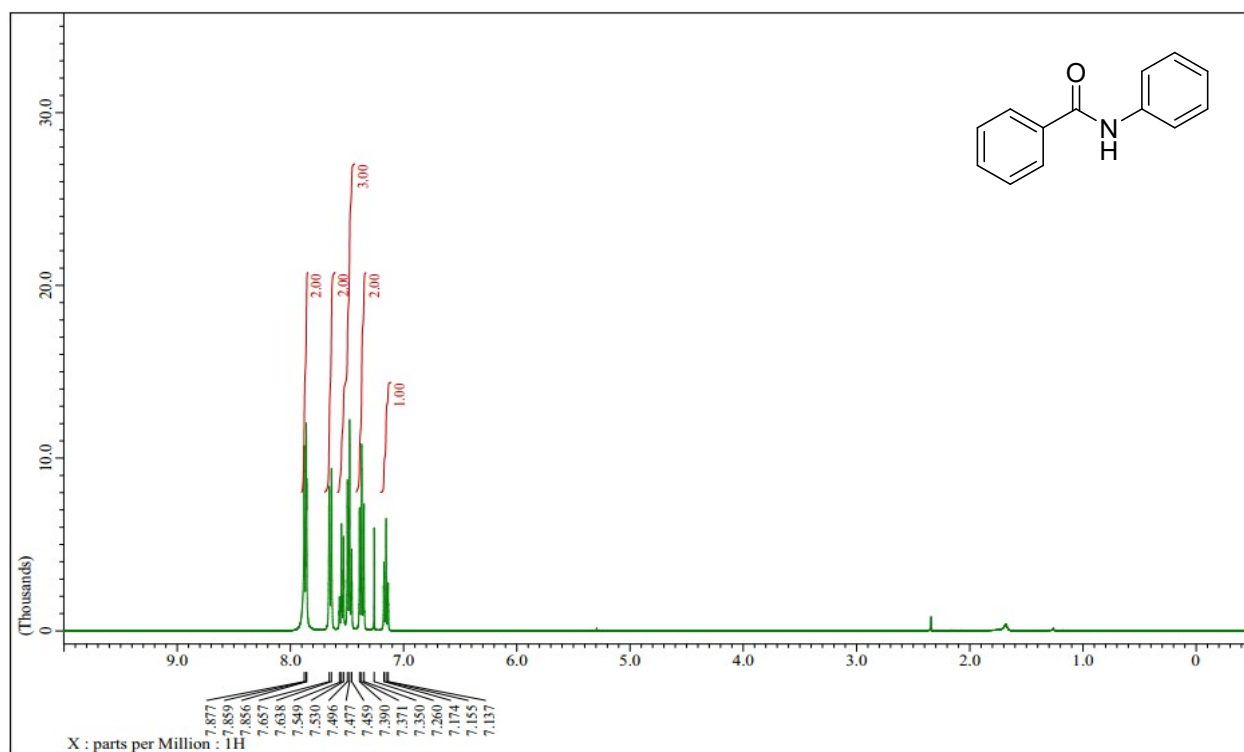


¹H NMR spectrum of phenyl(piperidin-1-yl)methanone (5g)

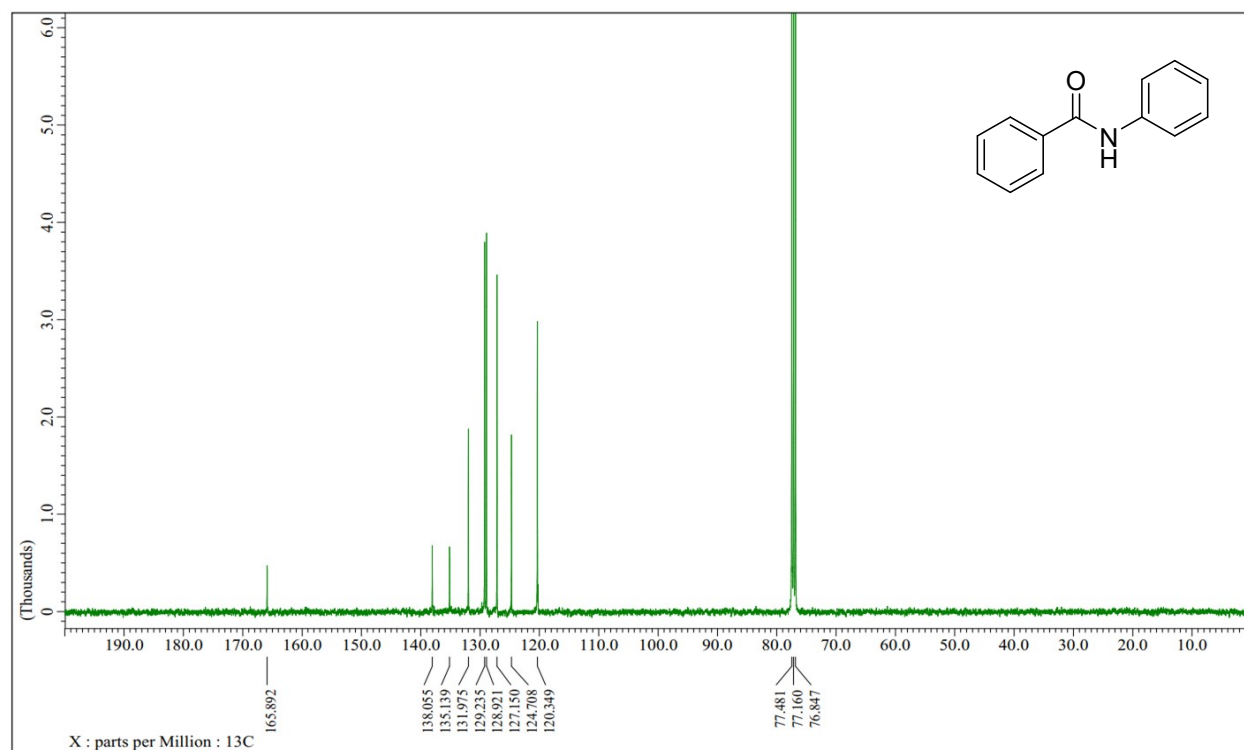


¹³C NMR spectrum of phenyl(piperidin-1-yl)methanone (5g)

***N*-Phenylbenzamide (5h)**

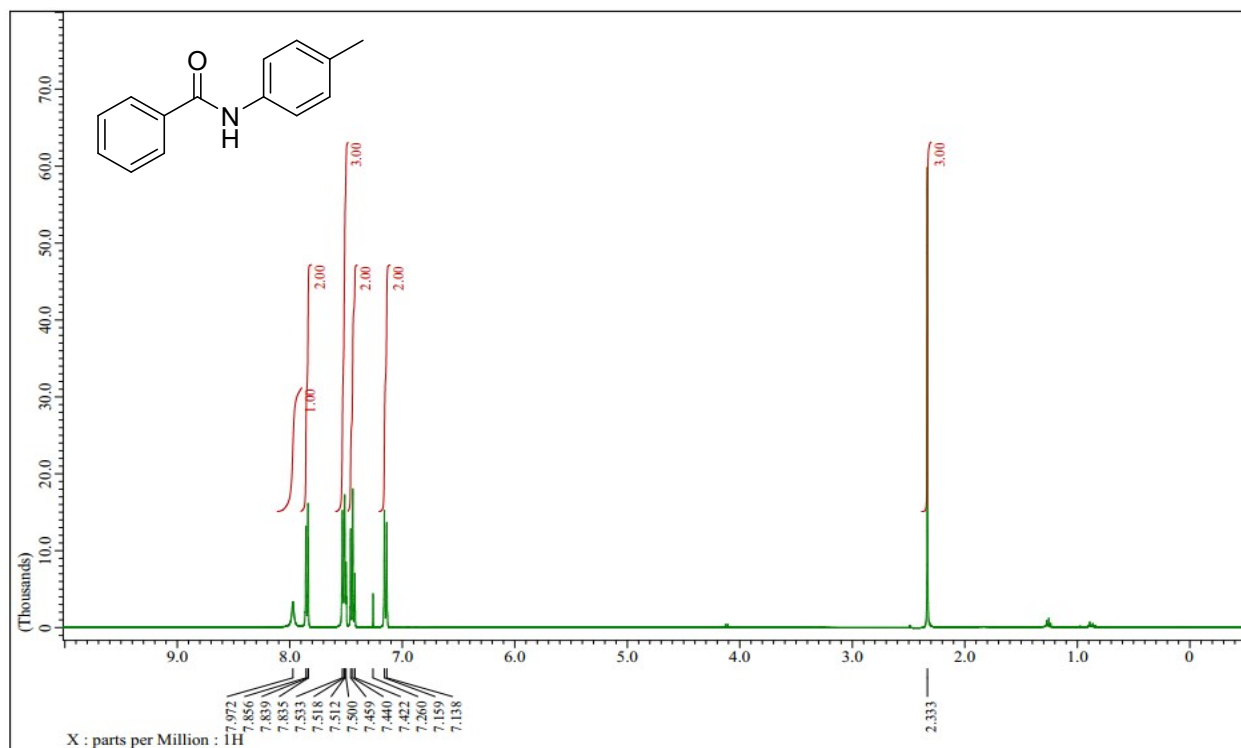


¹H NMR spectrum of *N*-phenylbenzamide (5h)

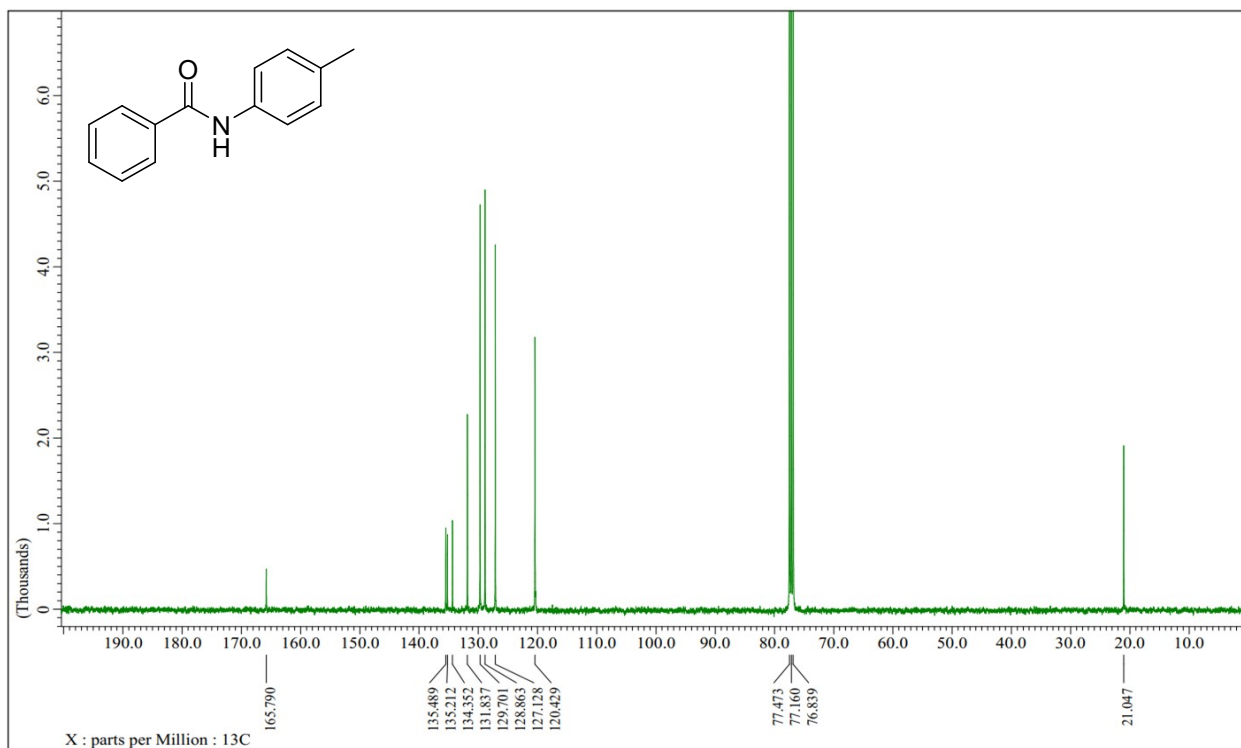


¹³C NMR spectrum of *N*-phenylbenzamide (5h)

***N*-(*p*-Tolyl)benzamide (5i)**

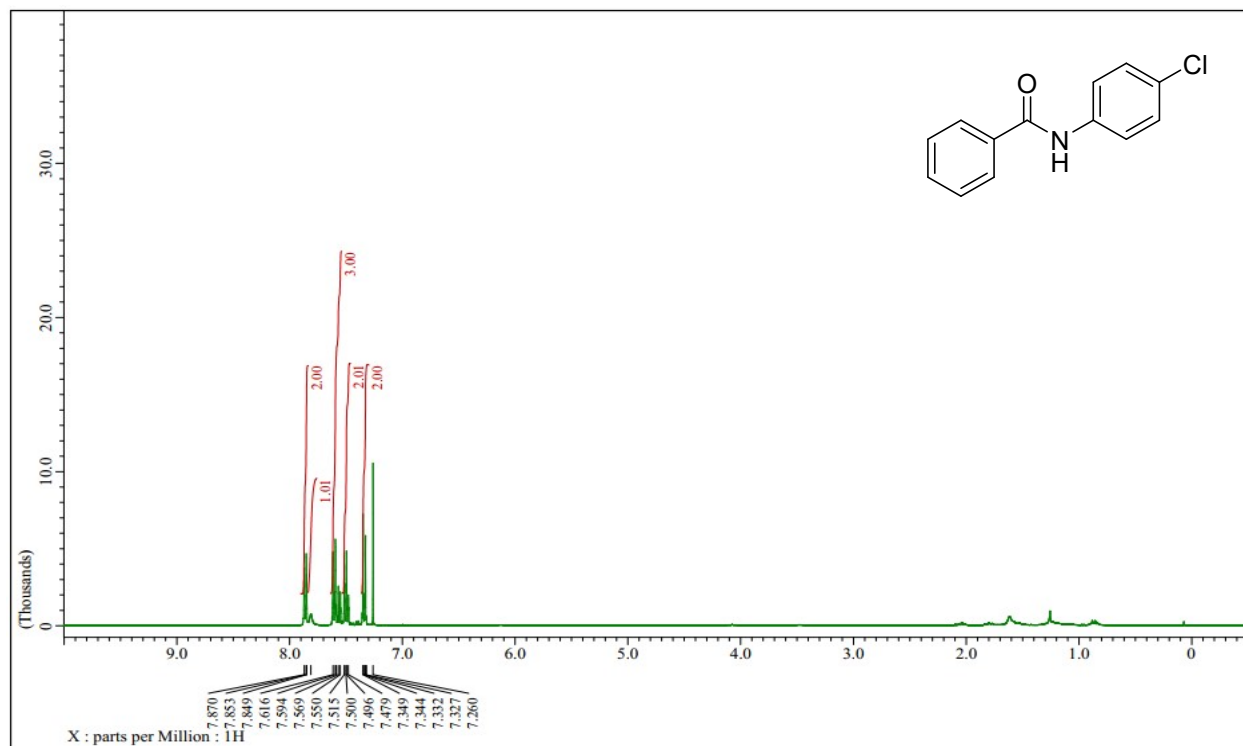


¹H NMR spectrum of *N*-(*p*-tolyl)benzamide (5i)

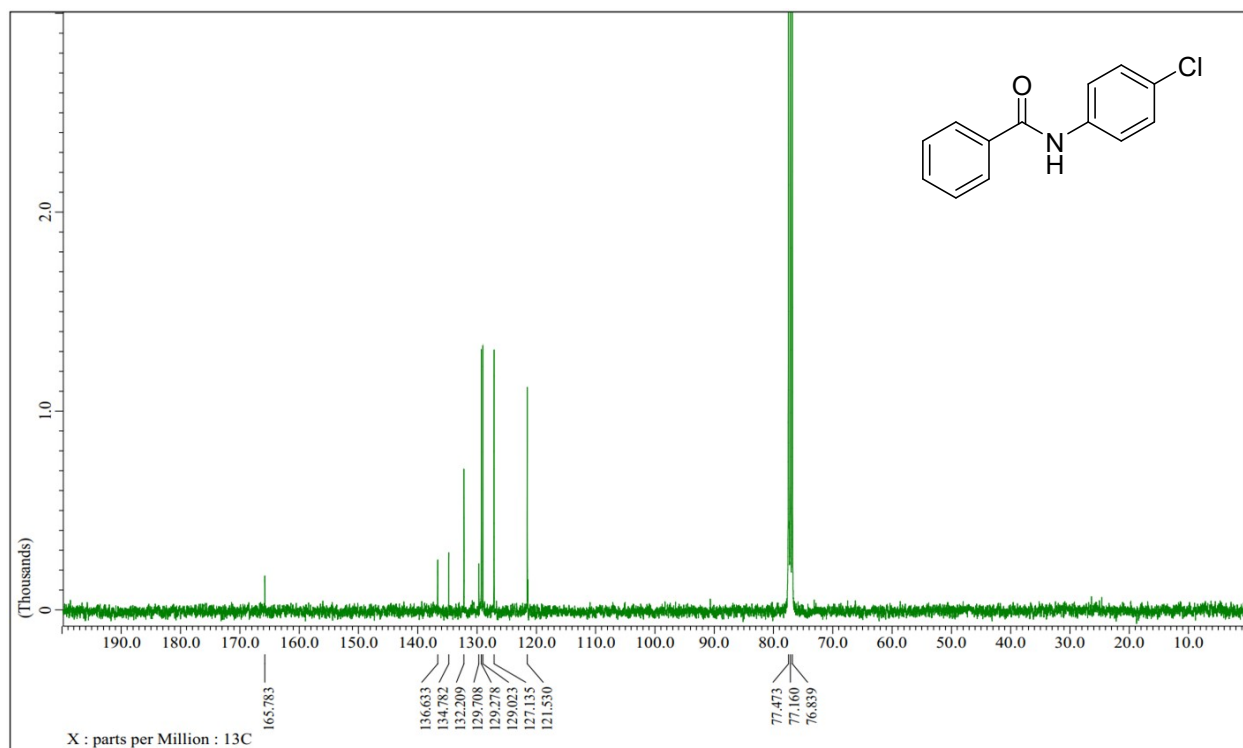


¹³C NMR spectrum of *N*-(*p*-tolyl)benzamide (5i)

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

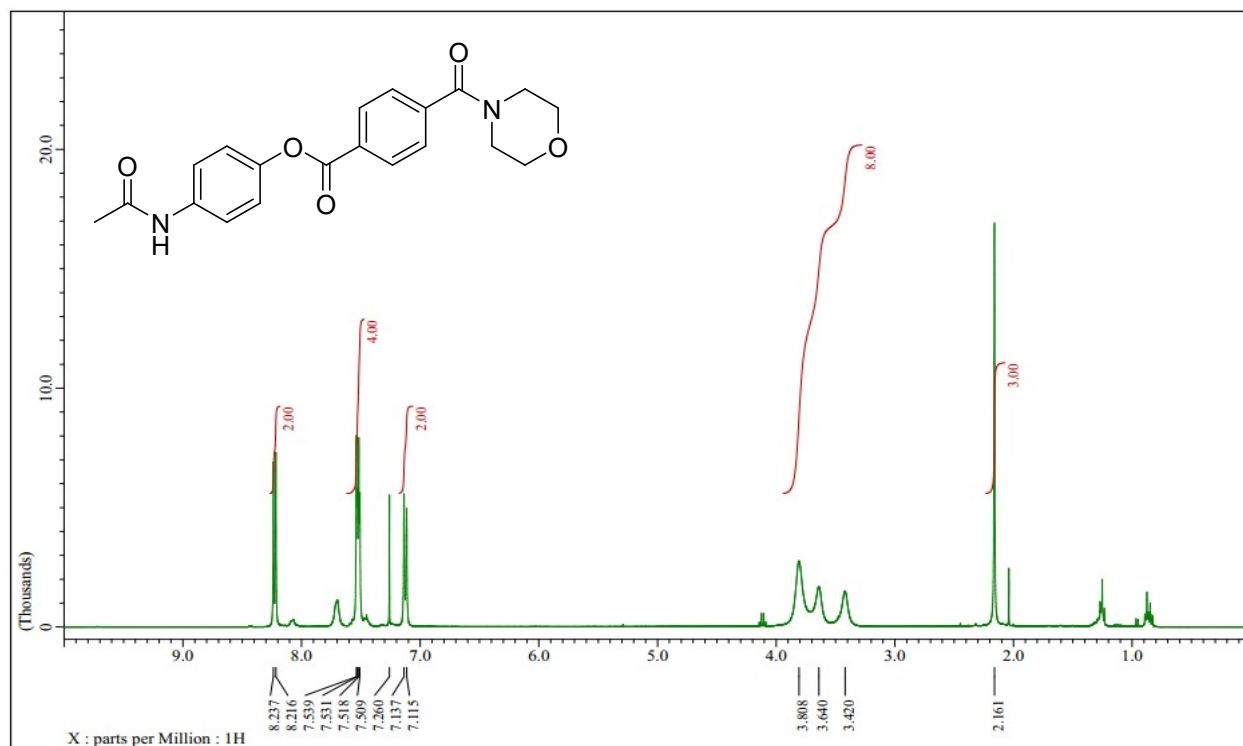


¹H NMR spectrum of *N*-(4-chlorophenyl)benzamide (5j)

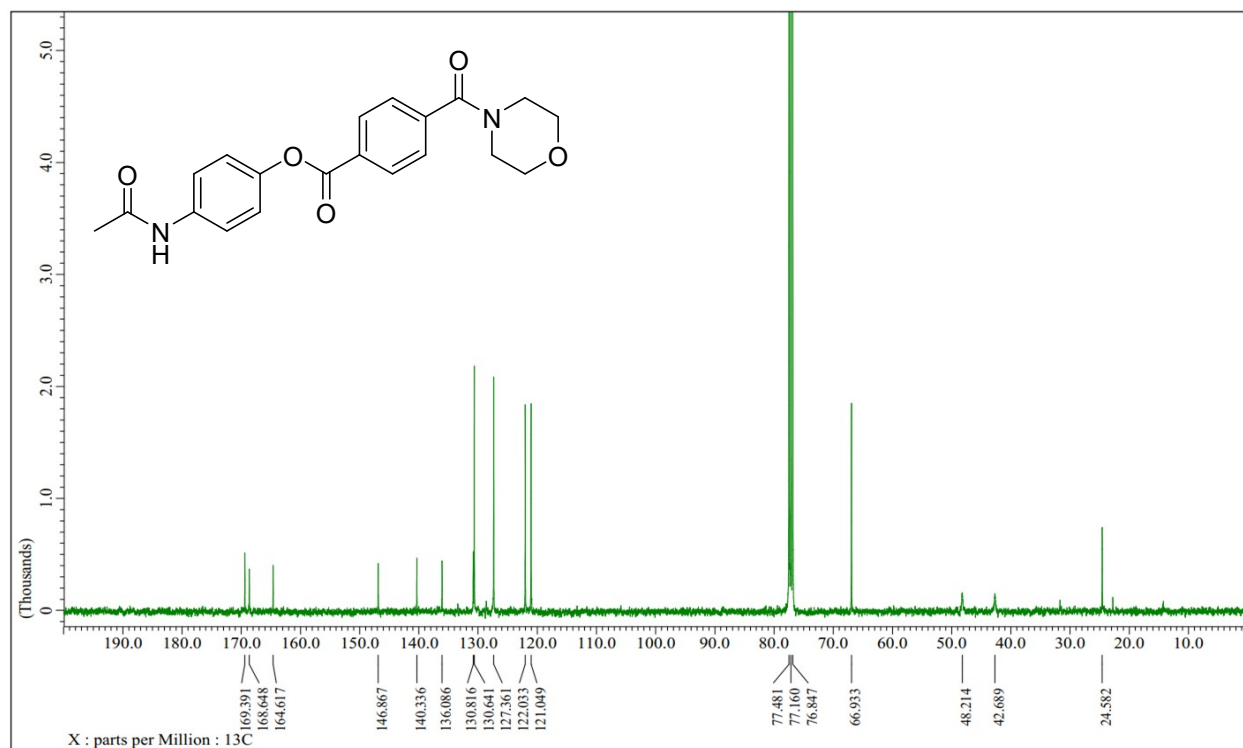


¹³C NMR spectrum of *N*-(4-chlorophenyl)benzamide (5j)

4-Acetamidophenyl 4-(morpholine-4-carbonyl)benzoate (6a)

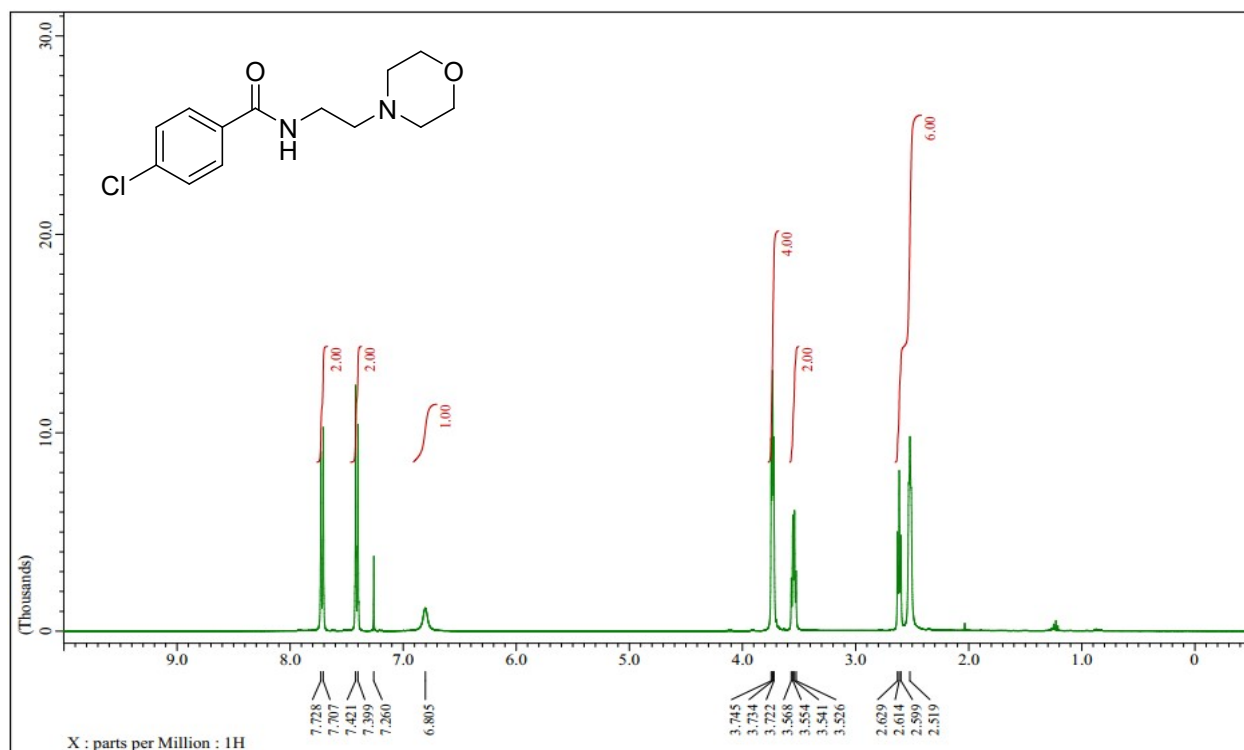


¹H NMR spectrum of 4-acetamidophenyl 4-(morpholine-4-carbonyl)benzoate (6a)

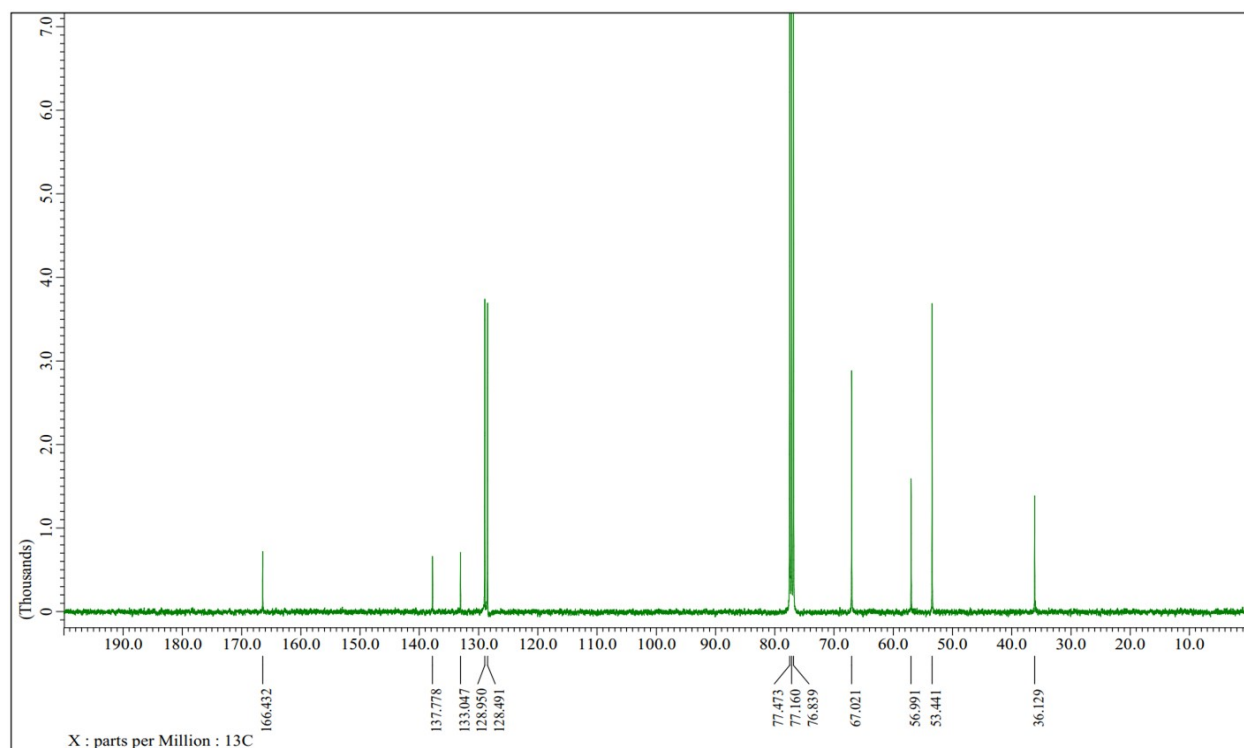


¹³C NMR spectrum of 4-acetamidophenyl 4-(morpholine-4-carbonyl)benzoate (6a)

Moclobemide (6b)

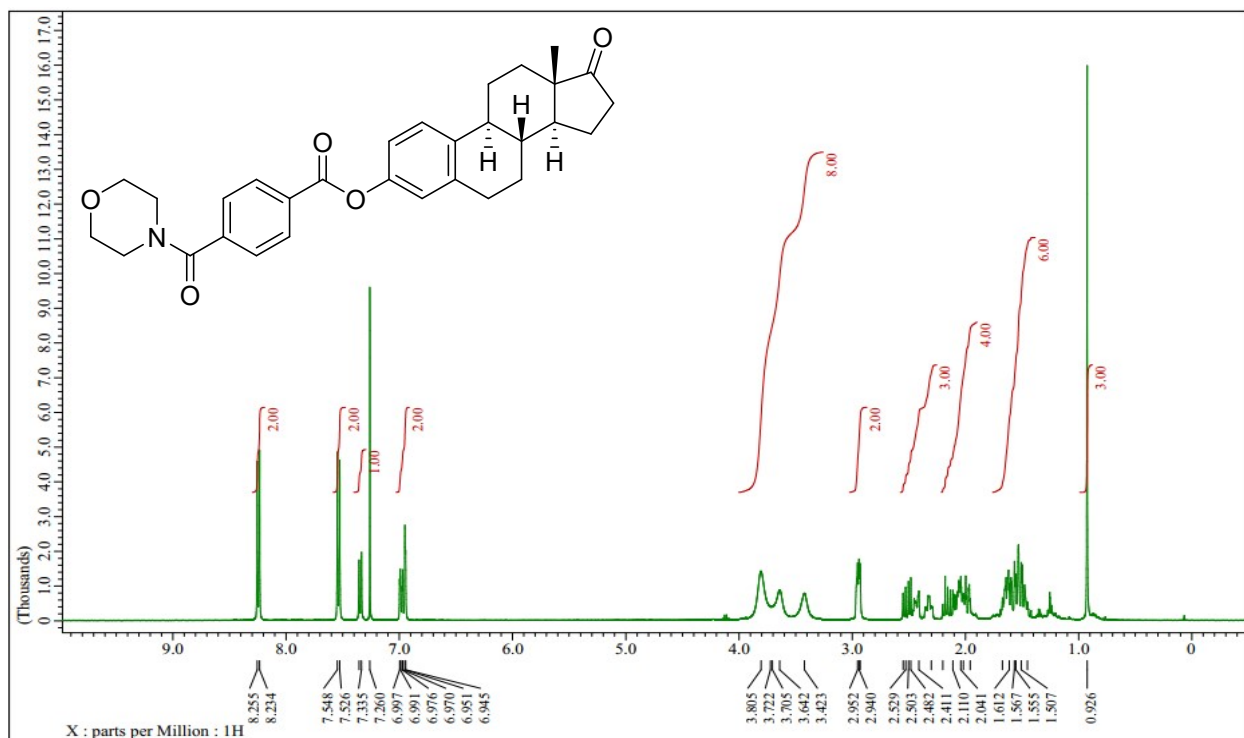


¹H NMR spectrum of moclobemide (6b)

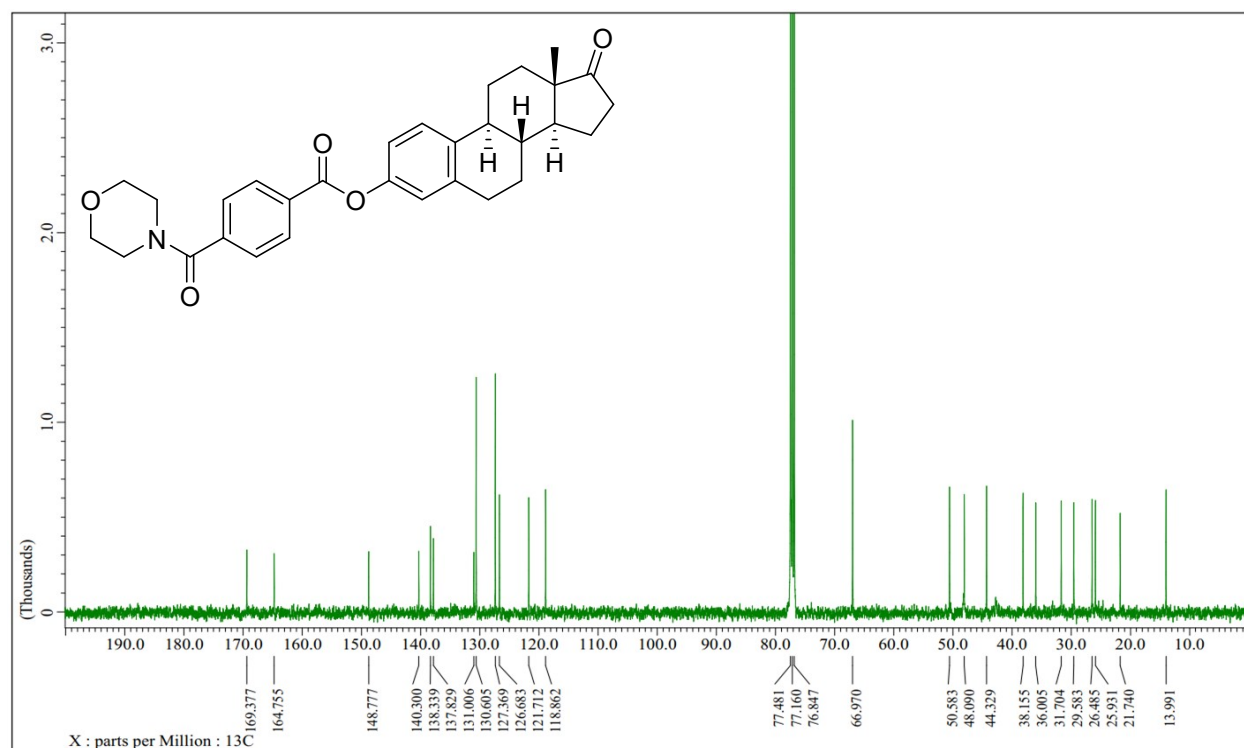


¹³C NMR spectrum of moclobemide (6b)

(8R,9S,13S,14S)-13-Methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl 4-(morpholine-4-carbonyl)benzoate (6c)



¹H NMR spectrum of (**6c**)



¹³C NMR spectrum of (**6c**)