

Supporting Information

Direct amidation of carboxylic acids with amines under microwave irradiation using silica gel as a solid support

Andrea Ojeda-Porras,^a Alejandra Hernández-Santana^a and Diego Gamba-Sánchez*^a

^a *Laboratory of Organic Synthesis, Bio and Organocatalysis, Chemistry Department, Universidad de los Andes, Cra 1 No. 18A-12; Q:305, Bogotá 111711, Colombia*

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General information

Proton nuclear magnetic resonance (^1H NMR) spectra were recorded on a 400 MHz spectrometer. The chemical shifts are expressed in parts per million (ppm) referenced to TMS. Data are reported as follows: δ , chemical shift; multiplicity (recorded as br, broad; s, singlet; d, doublet; t, triplet; q, quadruplet; quint, quintuplet and m, multiplet), coupling constants (J in Hertz, Hz) and integration. Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on the same instruments at 100 MHz. The chemical shifts are expressed in parts per million (ppm), referenced to TMS. Infrared spectra (IR) are reported in terms of absorption frequency (ν , cm^{-1}) using KBr. Mass spectrometry (MS) was performed in electron impact (EI; 70 eV) mode. Mass spectrums data are reported as m/z . High resolution mass spectrometry (HRMS) was performed on a Q-TOF LC/MS instrument.

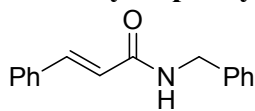
Known compounds were found to be identical to those described in literature, in consequence in some cases only ^1H NMR spectra is shown.

General Procedure

Amine (1.5 mmol) and carboxylic acid (1.5 mmol) were dissolved in ethyl acetate (15 mL), then silica gel 60 230-400 mesh (1.0 g) was added. The solvent was removed under reduced pressure, the reactant mixture was transferred to a microwaves tube, and it was set to react in a CEM microwave reactor in cycles of 20 minutes at a power of 200 W maintaining constant temperature at 130 °C, and a hold time of 2 minutes. The reactant mixture was allowed to cool to room temperature, sonicated for 20 minutes with 30 mL of ethyl acetate, filtered, and the silica was washed with another 30 mL of ethyl acetate. The organic phase was washed with a saturated solution of NaHCO_3 and HCl (10%), dried over MgSO_4 , filtered and the solvent was withdrawn under reduced pressure to obtain the pure product. In some cases flash chromatography was and it is indicated in the characterization data.

Characterizations of products

N-Benzyl-3-phenyl-acrylamide (3)



Following the general procedure, compound was isolated and analysis of the sample indicated 94% yield (335 mg). mp: 107 °C (Lit.¹ 106-107°C)

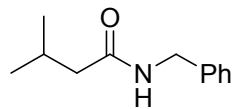
^1H NMR (400 MHz, CDCl_3) δ (ppm) 7.66 (d, $J = 15.6$ Hz, 1H), 7.25-7.48(m, 10H), 6.43 (d, $J = 15.7$ Hz, 1H), 6.15 (s, 1H), 4.55 (d, $J = 5.7$ Hz, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm) 165.8, 141.4, 138.2, 134.8, 129.7, 128.8, 128.7, 127.9, 127.7, 127.6, 120.5, 43.9.

IR: 3267, 3028, 1652, 1616, 1543 cm^{-1} .

MS (EI): 237 [M]⁺ (44), 131 (100), 103 (79), 91 (21), 77 (39).

***N*-Benzyl-3-methyl-butylamide (4)**



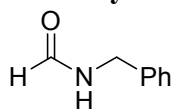
Following the general procedure, compound was isolated and analysis of the sample indicated 78% yield (224 mg). mp: 55 °C (Lit.² 58-59°C)

¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.26 – 7.36 (m, 5H), 4.45 (d, *J* = 5.6 Hz, 2H), 2.13 (m, 1H), 2.08 (d, *J* = 6.6 Hz, 2H), 0.97 (d, *J* = 6.4 Hz, 6H)
¹³C NMR (101 MHz, CDCl₃) δ (ppm) 172.3, 138.4, 128.7, 127.8, 127.5, 46.1, 43.6, 26.2, 22.5.

IR: 3291, 2955, 1636, 1548.

MS (EI): 191 [M]⁺ (29), 149 (47), 106 (69), 91 (100), 57 (34).

***N*-Benzyl-formamide (5)**

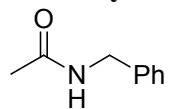


Following the general procedure, compound was isolated and analysis of the sample indicated 85% yield (172 mg). The presence of two rotamers was observed in the NMR spectra in an 0.85 (M):0.15 (m) ratio

¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.23 (M) (s, 1H), 8.15 (m) (d, *J* = 11.9 Hz, 1H), 7.21– 7.40 (m, 5H), 6.07 (bs, 1H), 4.46 (M)(d, *J* = 5.9 Hz, 2H), 4.39 (m) (d, *J* = 6.5 Hz, 2H).

¹³C NMR, IR and MS (EI) spectra and mp were identical to those reported previously³

***N*-Benzyl-acetamide (6)**

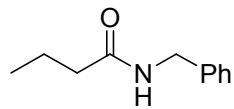


Following the general procedure, compound was isolated and analysis of the sample indicated 83% yield (186 mg) using acetic acid. The same product was obtained in quantitative yield (224 mg) using malonic acid.

¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.25-7.34 (m, 5H), 6.05 (bs, 1H), 4.40 (d, *J* = 5.7 Hz, 2H), 1.99 (s, 3H).

¹³C NMR, IR and MS (EI) spectra and mp were identical to those reported previously³

***N*-Benzyl-butylamide (7)**



Following the general procedure, compound was isolated and analysis of the sample indicated 94% yield (250 mg). mp: 45°C (Lit.⁴ 47-48°C)

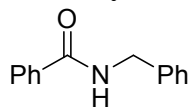
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.24 – 7.31 (m, 5H), 6.16 (bs, 1H), 4.10 – 4.39 (m, 2H), 2.17 (t, *J* = 7.5 Hz, 2H), 1.63-1.69 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 172.9, 138.4, 128.5, 127.6, 127.3, 43.4, 38.5, 19.1, 13.7.

IR: 3292, 2962, 2931, 1633, 1552 cm⁻¹.

MS (EI): 177 [M]⁺ (54), 149 (25), 106 (96), 91 (100), 43 (52).

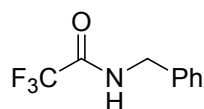
***N*-Benzyl-benzamide (8)**



Following the general procedure, compound was isolated and analysis of the sample indicated 96% yield (304 mg).

¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.79 (d, *J* = 7.2 Hz, 2H), 7.19 – 7.55 (m, 8H), 6.49 (bs, 1H), 4.64 (dd, *J* = 5.6, 1.7 Hz, 2H).

¹³C NMR, IR and MS (EI) spectra and mp were identical to those reported previously³



***N*-Benzyl-2,2,2-trifluoro-acetamide (9)**

Following the general procedure, compound was isolated and analysis of the sample indicated 94% yield (286 mg). mp: 69 °C (Lit.⁵ 70-71°C).

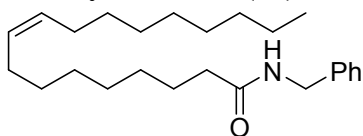
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.21 – 7.48 (m, 5H), 6.65 (s, 1H), 4.47 – 4.58 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 157.2 (q, *J*_{C-F}=37.3 Hz), 135.9, 129.1, 128.3, 128.0, 115.9 (q, *J*_{C-F}=287.9 Hz), 44.0.

IR: 3302, 3109, 1702, 1560 cm⁻¹.

MS (EI): 203 [M]⁺ (52), 134 (28), 91 (100).

***N*-benzyleamide (10)**



Following the general procedure, compound was isolated and analysis of the sample indicated 86 % yield (479 mg). mp: 51-53 °C (Lit.⁶ 48-50°C). The product was purified by flash chromatography DCM/cyclohexane (9:1).

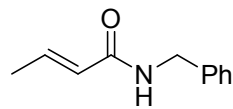
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.27- 7.38 (m, 5H), 5.70 (brs, 1H), 5.26-5.41 (m, 2H), 4.44 (d, *J* = 5.6 Hz, 2H), 2.21 (t, *J* = 7.6 Hz, 2H), 1.91-2.09 (m, 4H), 1.56-1.72(m, 2H), 1.13-1.39 (m, 20H), 0.88 (t, *J* = 5.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 172.96, 138.42, 130.01, 129.76, 128.73, 127.85, 127.52, 43.63, 36.83, 31.91, 29.78, 29.70, 29.53, 29.33, 29.30, 29.25, 29.14, 27.23, 27.18, 25.77, 22.69, 14.12.

IR: 3300.12, 2920, 2850, 1639, 1558, 1455 cm⁻¹.

HRMS: calcd for C₂₅H₄₂NO, 372.3266; found, 372.3283.

2-butenic acid benzylamide (11)



Following the general procedure, compound was isolated and analysis of the sample indicated quantitative yield (262 mg). mp: 114-115°C (Lit.⁷ 118-119°C)

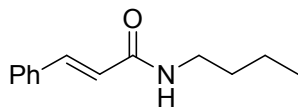
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.26 – 7.35 (m, 5H), 6.83 – 6.92 (m, 1H), 5.82 (d, *J* = 15.2 Hz, 1H), 4.49 (d, *J* = 5.7 Hz, 2H), 1.83 (d, *J* = 6.9 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 165.8, 140.2, 138.4, 128.7, 127.8, 124.9, 43.6, 17.7.

IR: 3265, 3082, 1671, 1627, 1559 cm⁻¹.

MS (EI): 175 [M]⁺ (14), 160 (100), 106 (38), 91 (51), 69 (61), 41 (61).

***N*-Butyl-3-phenyl-acrylamide (12)**



Following the general procedure, compound was isolated and analysis of the sample indicated 98% yield (299 mg) mp:74-75°C (Lit.⁸ 77°C)

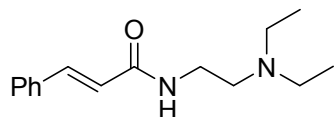
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.62 (d, *J* = 15.6 Hz, 1H), 7.48 (m, 2H), 7.29 – 7.40 (m, 3H), 6.37 – 6.47 (m, 1H), 5.91 (s, 1H), 3.38 (dd, *J* = 8.8, 3.8 Hz, 2H), 1.50 – 1.60 (m, 2H), 1.32 – 1.44 (m, 2H), 0.90 – 0.99 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 165.9, 140.0, 134.9, 129.6, 128.8, 127.7, 120.8, 39.5, 31.7, 20.1, 13.7.

IR: 3289, 2957, 2931, 1655, 1617, 1559, 1342 cm⁻¹.

MS (EI): 203 [M]⁺ (10), 146 (25), 131 (100), 103 (55), 77 (24).

***N*-(2-Diethylamino-ethyl)-3-phenyl-acrylamide (13)**



Following the general procedure, compound was isolated and analysis of the sample indicated 86% yield (318 mg).

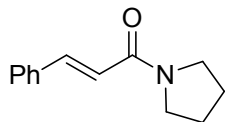
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.62 (d, *J* = 15.6 Hz, 1H), 7.51 (dd, *J* = 7.4, 1.6 Hz, 2H), 7.36 (d, *J* = 6.8 Hz, 3H), 6.42 (d, *J* = 15.6 Hz, 1H), 3.45 (dd, *J* = 11.3, 5.5 Hz, 2H), 2.56 - 2.64 (m, 6H), 1.05 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 165.8, 140.6, 135.0, 129.5, 128.7, 127.8, 121.0, 51.4, 46.7, 37.0, 11.6.

IR: 3423, 3282, 2970, 1657, 1621, 1545 cm⁻¹.

MS (EI): calcd for C₁₅H₂₃N₂O, 247.1805; found, 247.1829.

***N*-pyrrolidyl-1-cinnamoylamide (14)**



Following the general procedure, compound was isolated and analysis of the sample indicated 88% yield (266 mg). mp: 93-94°C (Lit.⁹ 97-98°C)

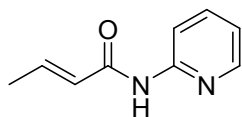
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.70 (d, *J* = 15.5 Hz, 1H), 7.47 – 7.58 (m, 2H), 7.36 (d, *J* = 7.4 Hz, 3H), 6.74 (d, *J* = 15.5 Hz, 1H), 3.58 – 3.65 (m, 4H), 1.96 – 2.05 (m, 2H), 1.88 – 2.03 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 164.7, 141.6, 135.3, 129.4, 128.7, 127.8, 118.9, 46.5, 46.0, 26.1, 24.3.

IR: 2966, 2873, 1651, 1600, 1432 cm⁻¹

MS (EI): 201 [M]⁺ (25), 131 (100), 103 (66), 70 (49).

(*E*)-*N*-(pyridin-2-yl)but-2-enamide (15)



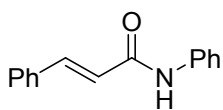
Following the general procedure, compound was isolated and analysis of the sample indicated 85% yield (207mg). mp: 76.8-78.7 °C

¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.78 (s, 1H), 8.26 – 8.34 (m, 2H), 7.72 (ddd, *J* = 8.5, 7.5, 1.9 Hz, 1H), 7.09 – 6.98 (m, 2H), 5.99 (d, *J* = 15.2 Hz, 1H), 1.91 (dd, *J* = 6.9, 1.7 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 164.2, 151.8, 147.5, 142.4, 138.4, 125.2, 119.6, 114.5, 17.9.

HRMS: calcd for C₉H₁₁N₂O, 163.0871; found, 163.0874.

***N*-phenylcinnamamide (16)**



A slight modification of general procedure was used for this compound. 1.0 equivalents of cinnamic acid were fixed on the silica gel and 0.3 equivalents of freshly distilled aniline. After irradiation for 20 min, a second addition of 0.3 equiv. of aniline was made and after each period of irradiation 0.3 equiv. were added (four addition), then standard procedure was applied, compound was isolated and analysis of the sample indicated 65% yield (217 mg). mp: 152 °C (Lit.¹⁰ 151-153°C)

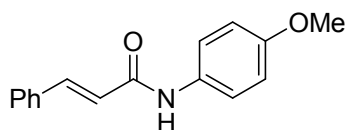
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.76 (d, *J* = 15.5 Hz, 1H), 7.62 (d, *J* = 6.5 Hz, 2H), 7.45 – 7.55 (m, 3H), 7.31 – 7.41 (m, 5H), 7.13 (t, *J* = 7.3 Hz, 1H), 6.57 (d, *J* = 15.5 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 164.3, 142.3, 138.1, 134.6, 129.97, 129.0, 128.8, 127.9, 124.4, 120.9, 120.1.

IR: 3271, 3034, 1661, 1625, 1596, 1545, 1442, 1350 cm⁻¹.

MS (EI): 223 [M]⁺ (14), 131 (100), 103 (77), 93 (37), 77 (36).

***N*-(4-Methoxy-phenyl)-3-phenyl-acrylamide (17)**



Following the general procedure, compound was isolated and analysis of the sample indicated 76% yield (289 mg). mp: 155 °C (Lit.¹¹ 152-155°C)

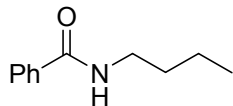
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.73 (d, *J* = 15.5 Hz, 1H), 7.36-7.52(m, 7H), 6.88 (d, *J* = 8.9 Hz, 2H), 6.54 (d, *J* = 15.5 Hz, 1H), 3.80 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 163.8, 156.5, 141.9, 134.7, 131.2, 129.8, 128.8, 127.9, 121.7, 120.9, 114.2, 55.5.

IR: 3304, 3056, 1657, 1623, 1540, 1509 cm⁻¹

HRMS: calcd for C₁₆H₁₆NO₂, 254.1181; found, 254.1193.

***N*-Butyl-benzamide (18)**



Following the general procedure, compound was isolated and analysis of the sample indicated 96% yield (255 mg). mp: 39 °C (Lit.⁴ 37-38°C).

When the reaction was carried out using a equimolar mixture of benzoic acid, aniline and *n*-butylamine, the product was purified by flash chromatography DCM/cyclohexane (9:1).

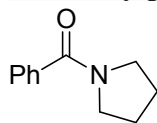
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.70 – 7.80 (m, 2H), 7.39 – 7.53 (m, 3H), 3.41 – 3.51 (m, 2H), 1.56 – 1.65 (m, 2H), 1.39-1.45 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 167.5, 134.9, 131.3, 128.5, 126.8, 39.8, 31.8, 20.2, 13.8.

IR: 2959, 2931, 2872, 1639, 1578, 1543, 1379 cm⁻¹.

MS (EI): 177 [M]⁺ (7), 105 (100), 77 (28).

***N*-benzoylpyrrolidine (19)**



Following the general procedure, compound was isolated and analysis of the sample indicated 64% (96% brsm) (168 mg) yield. mp: 49 °C (Lit.¹² 46-47°C)

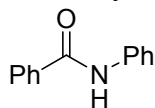
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.31 – 7.60 (m, 5H), 3.27 - 3.64 (m, 4H), 1.72 - 2.07 (m, 4H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 169.7, 137.4, 129.7, 128.2, 127.1, 49.6, 46.2, 26.4, 24.5.

IR: 2973, 2873, 1616, 1575, 1444, 1413, 1341, 1250. cm⁻¹.

MS (EI): 175 [M]⁺ (27), 105 (100), 77 (44)

***N*-Phenyl-benzamide (20)**



Following the general procedure, compound was isolated and analysis of the sample indicated 84% yield (248 mg). mp: 162 -164 °C (Lit.¹³ 162-164 °C)

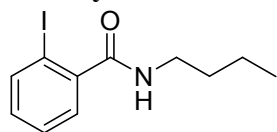
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.88 (d, *J* = 7.2 Hz, 2H), 7.81 (bs, 1H), 7.65 (d, *J* = 7.8 Hz, 2H), 7.48-7.58 (m, 3H), 7.38 (t, *J* = 7.9 Hz, 2H), 7.16 (t, *J* = 7.4 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 165.7, 137.9, 135.1, 131.8, 129.1, 128.8, 127.0, 124.6, 120.2.

IR: 3343, 1656, 1599, 1534, 1438 cm⁻¹.

MS (EI): 197 [M]⁺ (19), 105 (100), 77 (36).

***N*-Butyl-2-iodo-benzamide (21)**



Following the general procedure, compound was isolated and analysis of the sample indicated 77% yield (350 mg). mp: 92°C (Lit.¹⁴ 94-96°C)

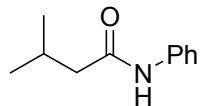
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.85 (d, *J* = 7.8 Hz, 1H), 7.36 (dd, *J* = 5.4, 1.8 Hz, 2H), 7.06-7.09 (m, 1H), 3.42 - 3.47 (m, 2H), 1.56 – 1.67 (m, 2H), 1.44 (dd, *J* = 15.2, 7.4 Hz, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 169.3, 142.6, 139.8, 130.9, 128.2, 128.1, 92.4, 39.8, 31.4, 20.2, 13.7.

IR: 3275, 2947, 2865, 1637, 1547 cm⁻¹.

MS (EI): 303 [M]⁺ (11), 261 (31), 231 (100), 203 (23), 105 (22), 76 (42).

3-Methyl-*N*-phenyl-butylamide (22)



Following the general procedure, compound was isolated and analysis of the sample indicated 98% yield (260 mg). mp: 107 °C (Lit.¹⁵ 109-110 °C)

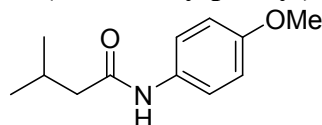
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.24 – 7.62 (m, 5H), 7.10 (d, *J* = 7.2 Hz, 1H), 2.21 (s, 3H), 0.94 – 1.18 (m, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 171.0, 137.9, 128.9, 124.2, 119.9, 47.0, 26.2, 22.4.

IR: 3243, 2969, 1651, 1597, 1539, 1503 cm⁻¹.

MS (EI): 177 [M]⁺ (9), 93 (100), 57 (17).

***N*-(4-Methoxy-phenyl)-3-methyl-butylamide (23)**



Following the general procedure, compound was isolated and analysis of the sample indicated 93% yield (289 mg). The

reaction was performed using 1.95 mmol of acid and 1.5 mmol of amine. mp: 119.3-122.9 °C (Lit.¹⁶ 126°C)

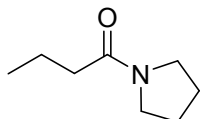
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.41 (d, *J* = 8.9 Hz, 2H), 7.18 (s, 1H), 6.84 (d, *J* = 9.0 Hz, 2H), 3.78 (s, 3H), 2.11 – 2.27 (m, 3H), 1.01 (d, *J* = 6.4 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 170.7, 156.3, 131.2, 121.7, 114.1, 55.5, 46.9, 26.3, 22.5.

IR: 3294, 2955, 1656, 1520, 1513, 1245 cm⁻¹.

MS (EI): 207 [M]⁺ (17), 123 (100), 108 (73).

***N*-butanoylpyrrolidine (24)**



Following the general procedure, compound was isolated and analysis of the sample indicated 62% yield (131 mg).

¹H NMR (400 MHz, CDCl₃) δ (ppm) 3.45 (bs, 4H), 2.27 (dd, *J* = 19.7, 12.3 Hz, 2H), 1.90 (bs, 4H), 1.69 (dd, *J* = 14.9, 7.4 Hz, 2H), 0.97 (t, *J* =

7.4 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 171.9, 46.6, 45.7, 36.7, 26.1, 24.3, 18.4, 14.00.

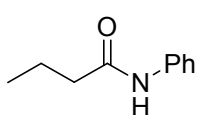
¹H NMR (400 MHz, DMSO) δ (ppm) 3.18 – 3.41 (m, 4H), 2.19 (t, *J* = 7.4 Hz, 2H), 1.73 - 1.87 (m, 4H), 1.45 – 1.56 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (101 MHz, DMSO) δ (ppm) 170.7, 46.3, 45.6, 36.2, 26.1, 24.4, 18.2, 14.3.

IR: 2965, 2875, 1625, 1451 cm⁻¹.

MS (EI): 141 [M]⁺ (21), 113 (49), 70 (65), 43 (100).

***N*-Phenyl-butyramide (25)**



Following the general procedure, compound was isolated and analysis of the sample indicated 63% yield (154 mg). mp: 94°C (Lit.¹⁷ 91-93°C)

¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.24 – 7.31 (m, 5H), 6.16 (bs, 1H), 4.10 – 4.39 (m, 2H), 2.17 (t, *J* = 7.5 Hz, 2H), 1.66 (m, 2H), 0.93 (t, *J* = 7.4

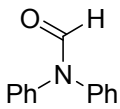
Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 172.9, 138.4, 128.5, 127.6, 127.3, 43.4, 38.5, 19.1, 13.7.

IR: 3284, 2961, 1657, 1600, 1546 cm⁻¹

MS (EI): 163 [M]⁺ (9), 93 (100), 43 (39).

***N,N*-Diphenyl-formamide (26)**



Following the general procedure, compound was isolated and analysis of the sample indicated 77% yield (228 mg). mp: 71-73°C (Lit.¹⁸ 70-72°C)

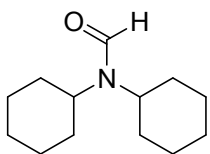
¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.66 (s, 1H), 7.36 – 7.41 (m, 4H), 7.33 – 7.24 – 7.32 (m, 4H), 7.16 (d, *J* = 7.5 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 161.6, 141.7, 139.6, 129.6, 129.1, 127.0, 126.7, 126.0, 125.0.

IR: 3041, 2919, 1688, 1593, 1494 cm⁻¹.

MS (EI): 197 [M]⁺ (70), 168 (100), 104 (28), 77 (31), 66 (41), 51 (49).

***N,N*-Dicyclohexyl-formamide (27)**

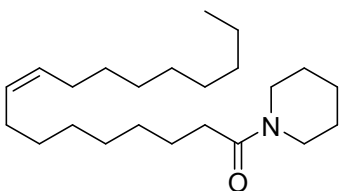


Following the general procedure, compound was isolated and analysis of the sample indicated 16% yield (50 mg).

¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.22 (s, 1H), 3.87 – 4.03 (m, 1H), 2.97 – 3.13 (m, 1H), 1.03 – 1.91 (m, 20H).

¹³C NMR, IR and MS (EI) spectra and mp were identical to those reported previously³

(Z)-1-(piperidin-1-yl)octadec-9-en-1-one (28)



Following the general procedure, compound was isolated and analysis of the sample indicated 92% yield (482 mg). The product was purified by flash chromatography DCM/cyclohexane (9:1).

¹H NMR (400 MHz, CDCl₃) δ (ppm) 5.29 – 5.43 (m, 2H), 3.47 (d, *J* = 58.1 Hz, 4H), 2.32 (d, *J* = 7.6 Hz, 2H), 1.93 –

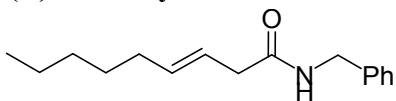
2.09 (m, 5H), 1.47 – 1.69 (m, 8H), 1.13 – 1.43 (m, 19H), 0.88 (td, *J* = 6.8, 3.9 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 171.65, 129.96, 129.78, 46.82, 42.71, 33.46, 31.89, 31.51, 29.76, 29.71, 29.62, 29.50, 29.33, 29.31, 29.16, 27.20, 25.62, 25.50, 24.57, 22.67, 22.56, 14.09.

IR: 2926, 2853, 1648, 1434 cm⁻¹.

HRMS: calcd for C₂₃H₄₄NO, 364.3423; found, 350.3438.

(E)-N-benzylnon-3-enamide (29)



Following the general procedure, compound was isolated and analysis of the sample indicated 70% yield (258 mg). mp: 43 °C. The product was purified by flash

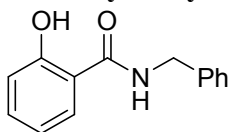
chromatography DCM/cyclohexane (7:3).

¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.22-7.39 (m, 5H), 5.90 (brs, 1H), 5.46-5.69 (m, 2H), 4.45 (d, *J* = 5.6 Hz, 2H), 3.00 (d, *J* = 6.8 Hz, 2H), 1.98-2.11 (m, 2H), 1.17-1.38 (m, 6H), 0.80-0.91 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 171.3, 137.0, 128.7, 127.7, 127.5, 122.3, 43.6, 40.5, 32.5, 31.3, 28.8, 22.4, 14.0.

HRMS: calcd for C₁₆H₂₄NO, 246.1858; found, 246.1860.

N-Benzyl-2-hydroxy-benzamide (32)



Following the general procedure, compound was isolated and analysis of the sample indicated 69% yield (236 mg). mp: 137 °C (Lit.¹⁹ 134-136°C). The product was purified by flash chromatography DCM/cyclohexane (1:1).

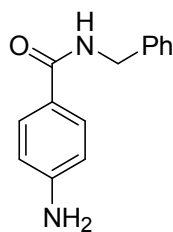
¹H NMR (400 MHz, CDCl₃) δ (ppm) 12.30 (s, 1H), 7.19 – 7.42 (m, 7H), 6.98 (dd, *J* = 8.3, 0.9 Hz, 1H), 6.78 – 6.84 (m, 1H), 6.64 (bs, 1H), 4.62 (d, *J* = 5.7 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 169.8, 161.6, 137.4, 134.3, 128.9, 127.9, 125.3, 118.7, 118.6, 114.1, 43.7.

IR: 3357, 3939, 1642, 1591, 1544, 1496, 1523 cm⁻¹.

HRMS: calcd for C₁₄H₁₄NO₂, 228.1025; found, 228.1035.

4-Amino-*N*-benzyl-benzamide (33)



Following the general procedure, compound was isolated and analysis of the sample indicated 66% yield (224 mg). mp: 89-91 °C (Lit.²⁰ 90°C). The product was purified by flash chromatography AcOEt /pentane (7:3).

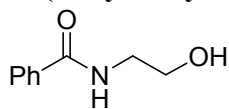
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.56 – 7.65 (m, 2H), 7.21 – 7.38 (m, 5H), 6.60 (d, *J* = 8.7 Hz, 2H), 6.44 (bs, 1H), 4.58 (d, *J* = 5.7 Hz, 2H), 3.99 (bs, 2H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 167.1, 149.7, 138.6, 128.7, 128.6, 127.8, 127.3, 123.7, 114.0, 43.8.

IR: 3442, 3344, 3237, 1635, 1597, 1537, 1502 cm⁻¹

MS (EI): 226 [M]⁺ (29), 120 (100), 92 (34), 65 (24), 39(18).

N-(2-Hydroxy-ethyl)-benzamide (39)



Following the general procedure, compound was isolated and analysis of the sample indicated 53% yield (131 mg). mp: 60 °C (Lit.²¹ 61-63°C)

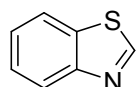
¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.77 (d, *J* = 7.6 Hz, 2H), 7.33 – 7.56 (m, 3H), 6.87 (bs, 1H), 3.80 (t, *J* = 4.9 Hz, 2H), 3.60 (m, 2H), 3.19 (bs, 1H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm) 168.7, 134.1, 131.6, 128.5, 127.0, 62.0, 42.8.

IR: 3341, 3064, 2946, 1636, 1545, 1314 cm⁻¹

HRMS: calcd for C₉H₁₂NO₂, 166.0868; found, 166.0854.

Benzothiazole (42)

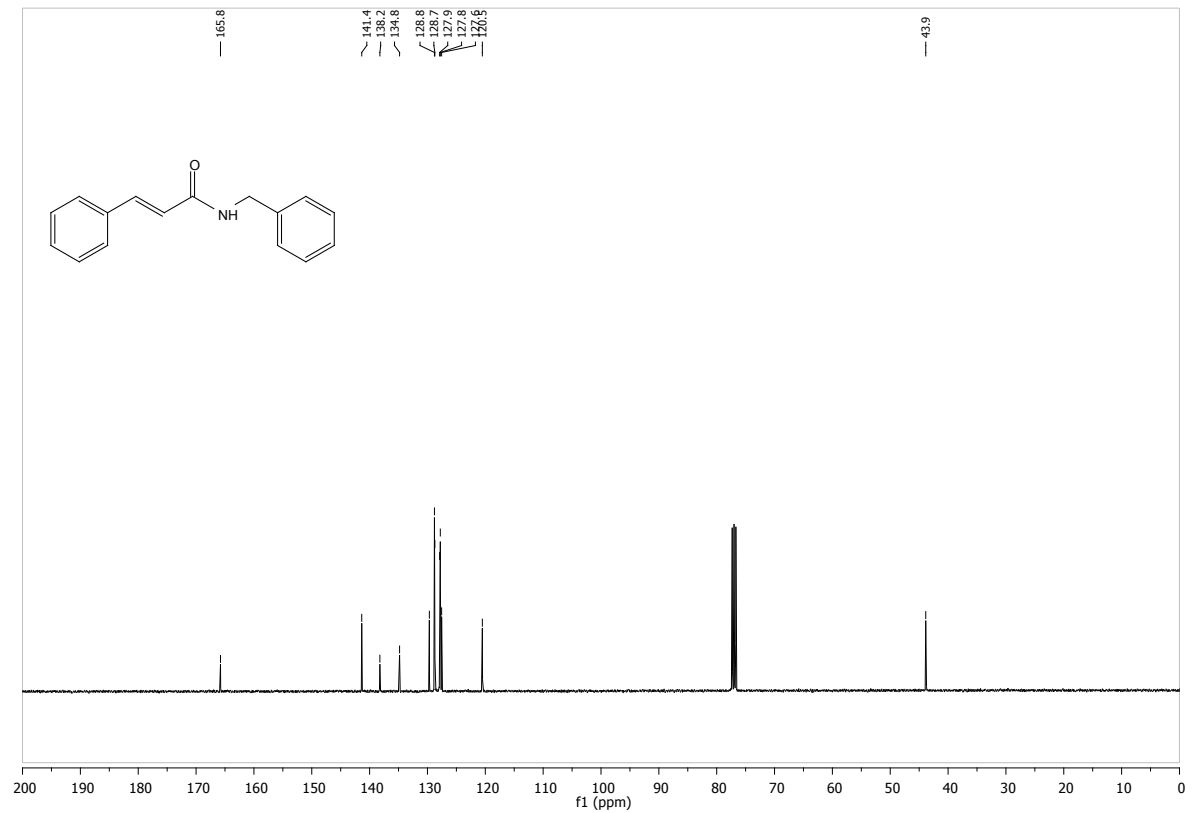
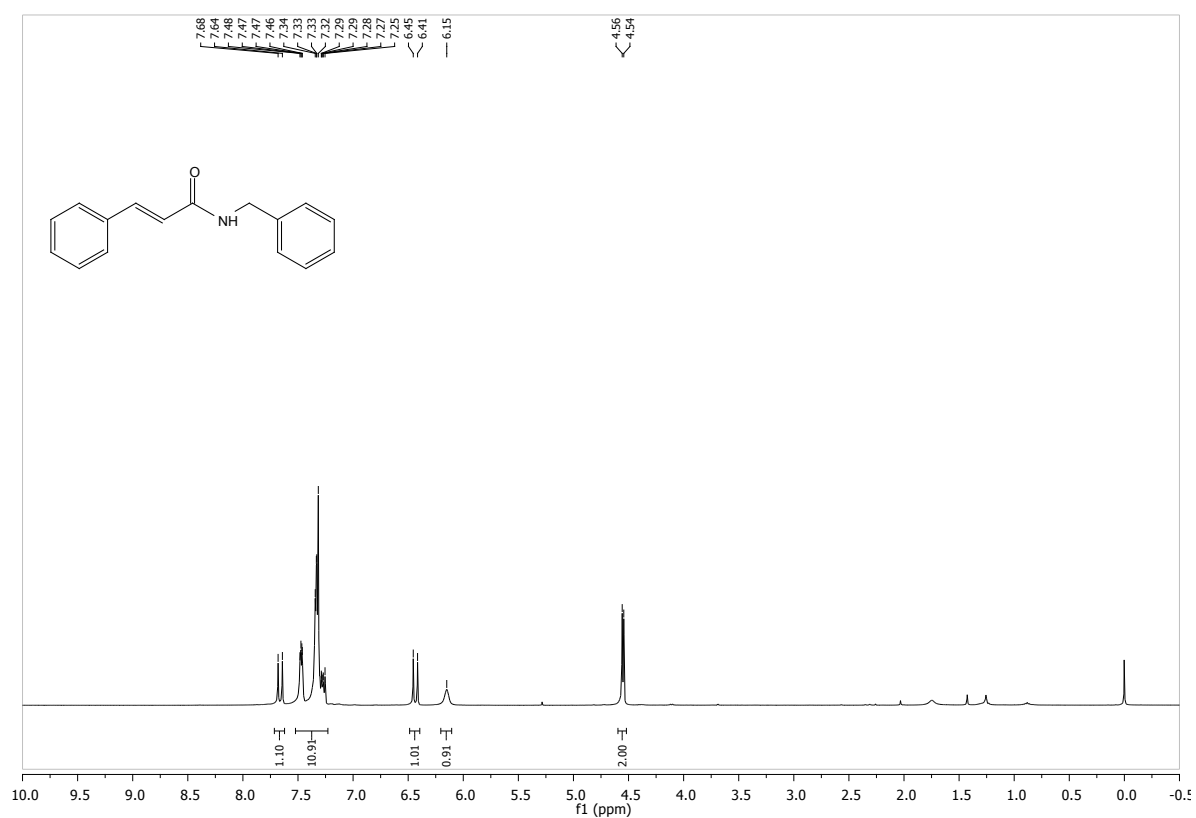


Following the general procedure, compound was isolated and analysis of the sample indicated 34 % yield (69 mg). The product was purified by flash chromatography DCM/cyclohexane (1:1).

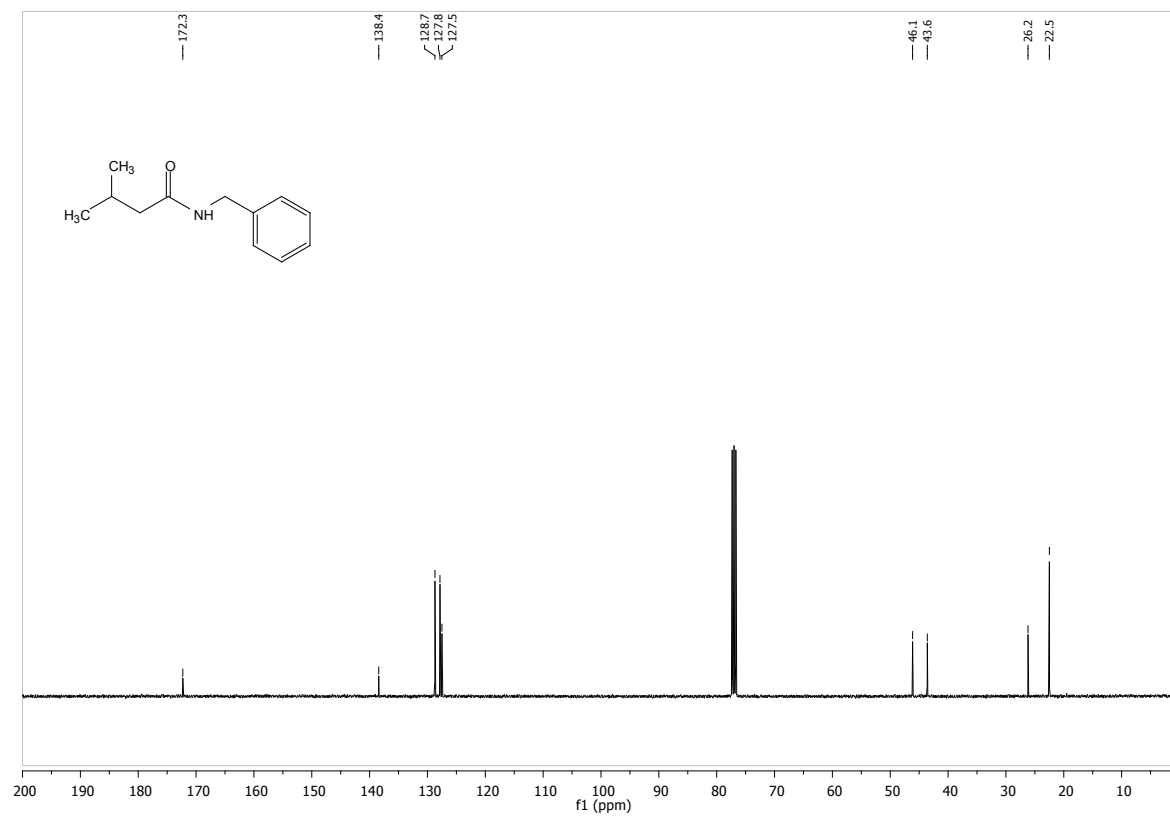
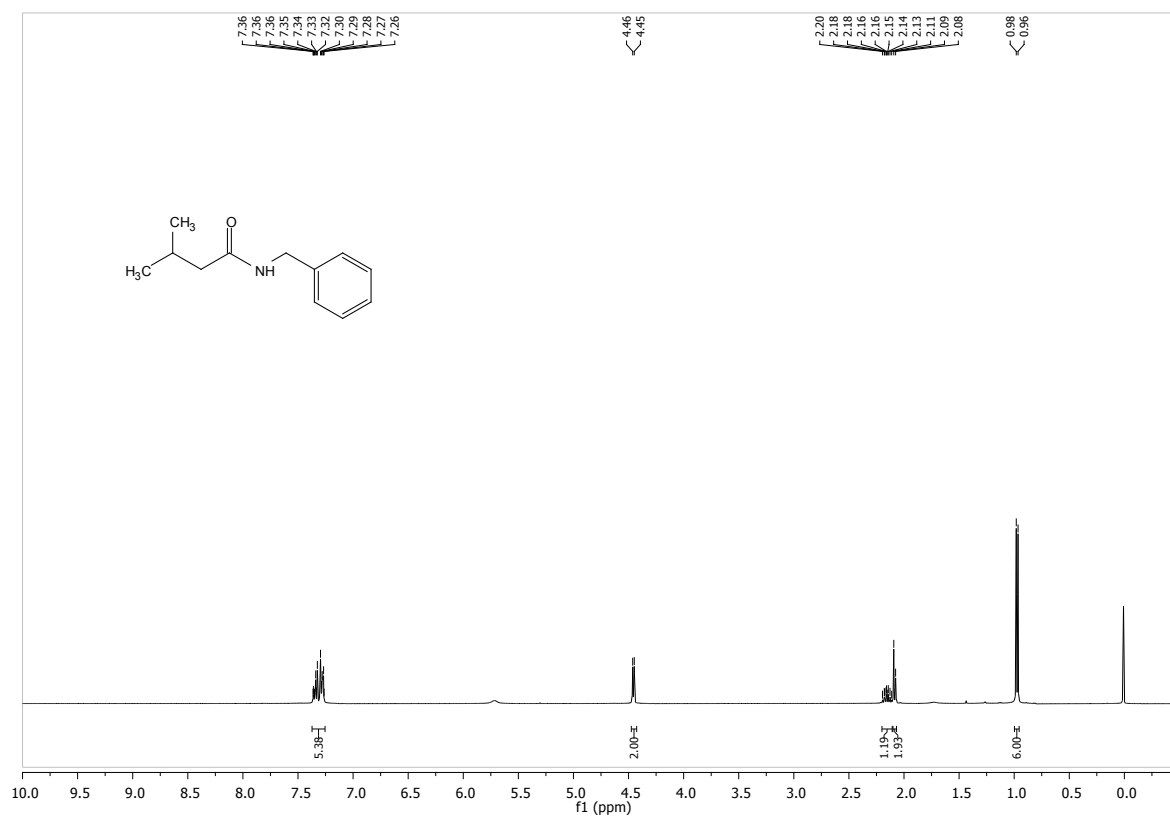
¹H NMR (400 MHz, CDCl₃) δ (ppm) 9.00 (s, 1H), 8.15 (d, *J* = 8.2 Hz, 1H), 7.92-8.01 (m, 1H), 7.30-7.60 (m, 2H).

¹³C NMR, IR and MS (EI) spectra and mp were identical to those reported previously³

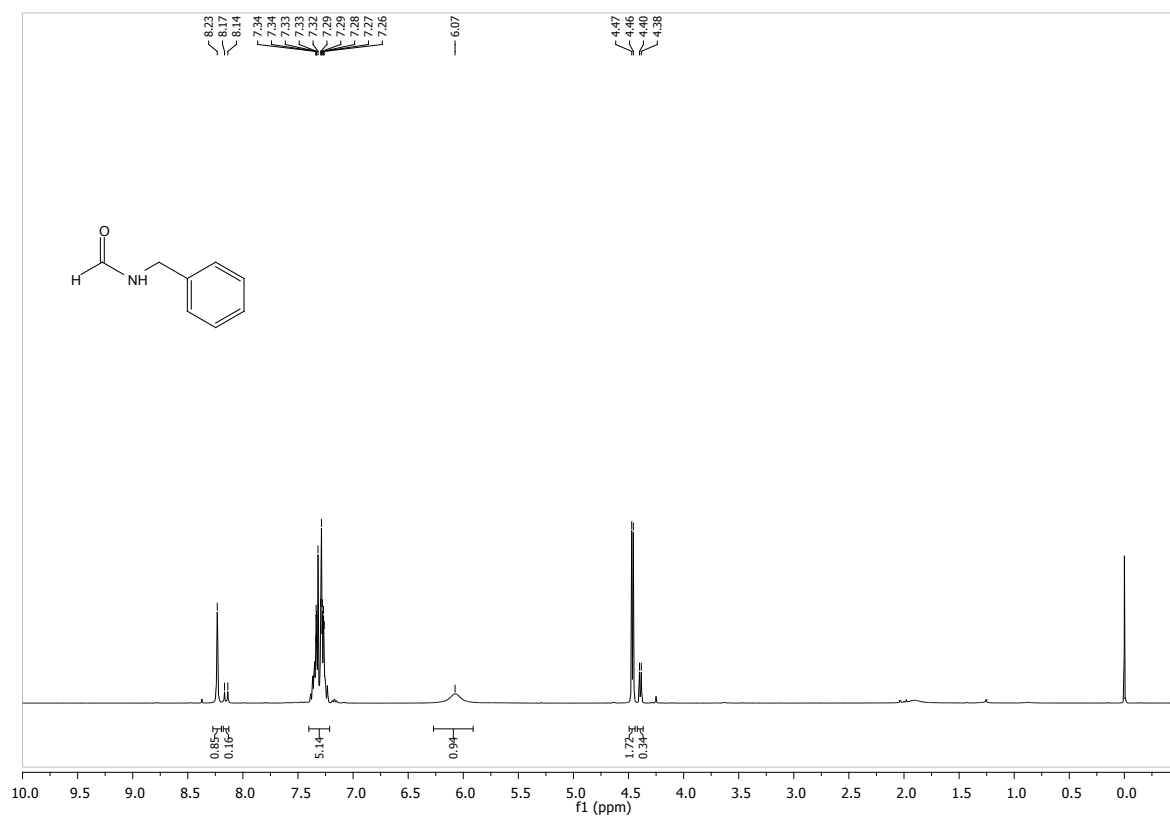
***N*-Benzyl-3-phenyl-acrylamide (3)**



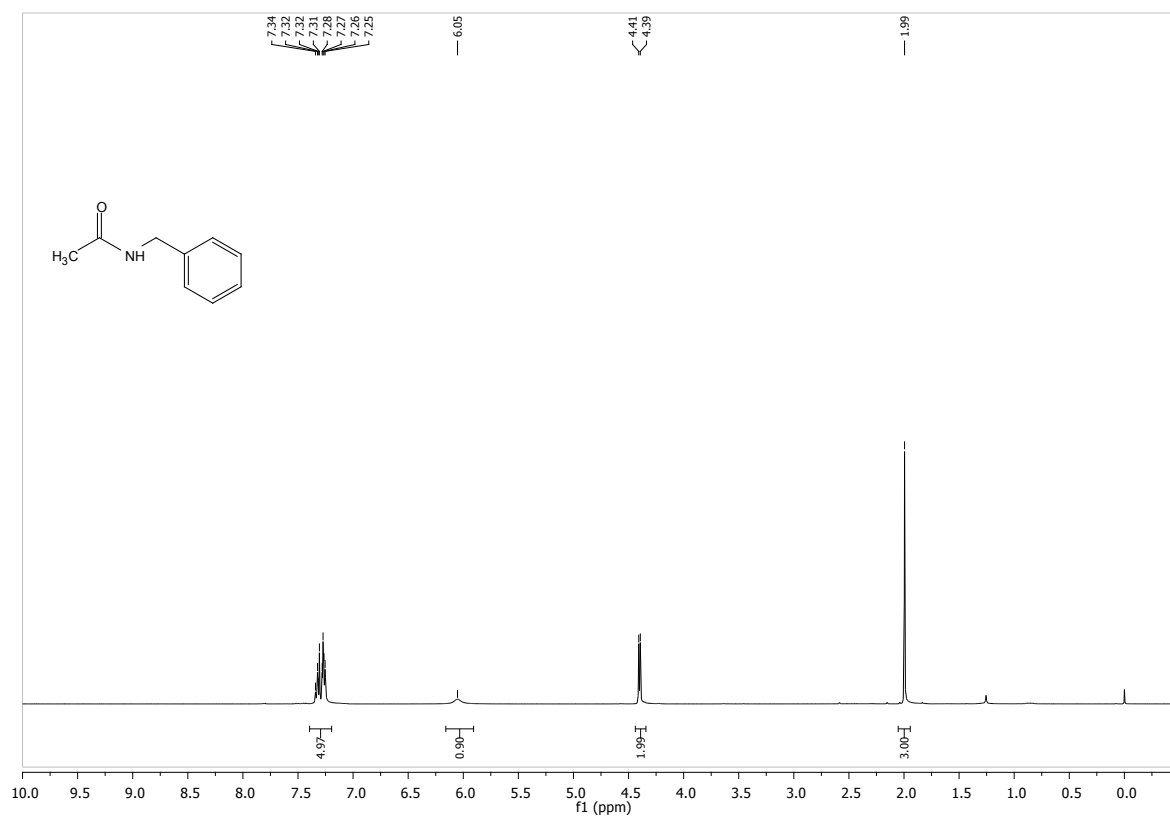
N-Benzyl-3-methyl-butamide (4)



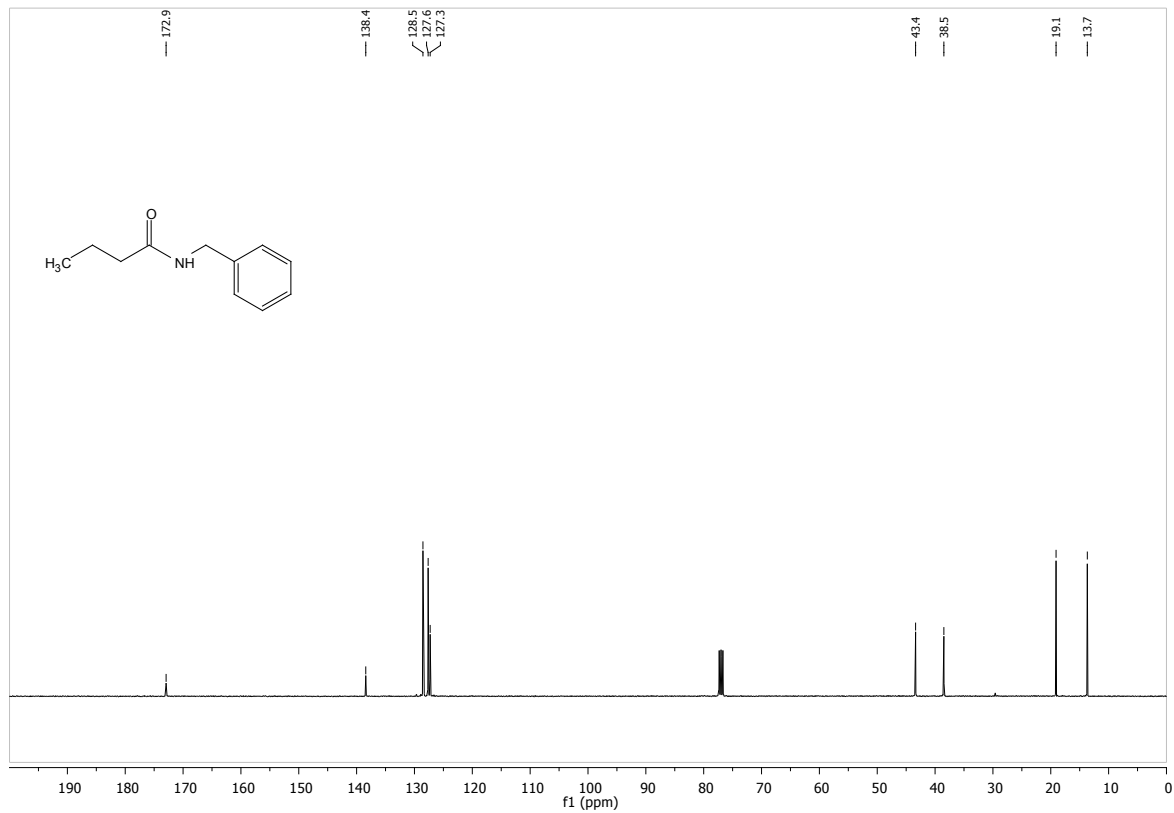
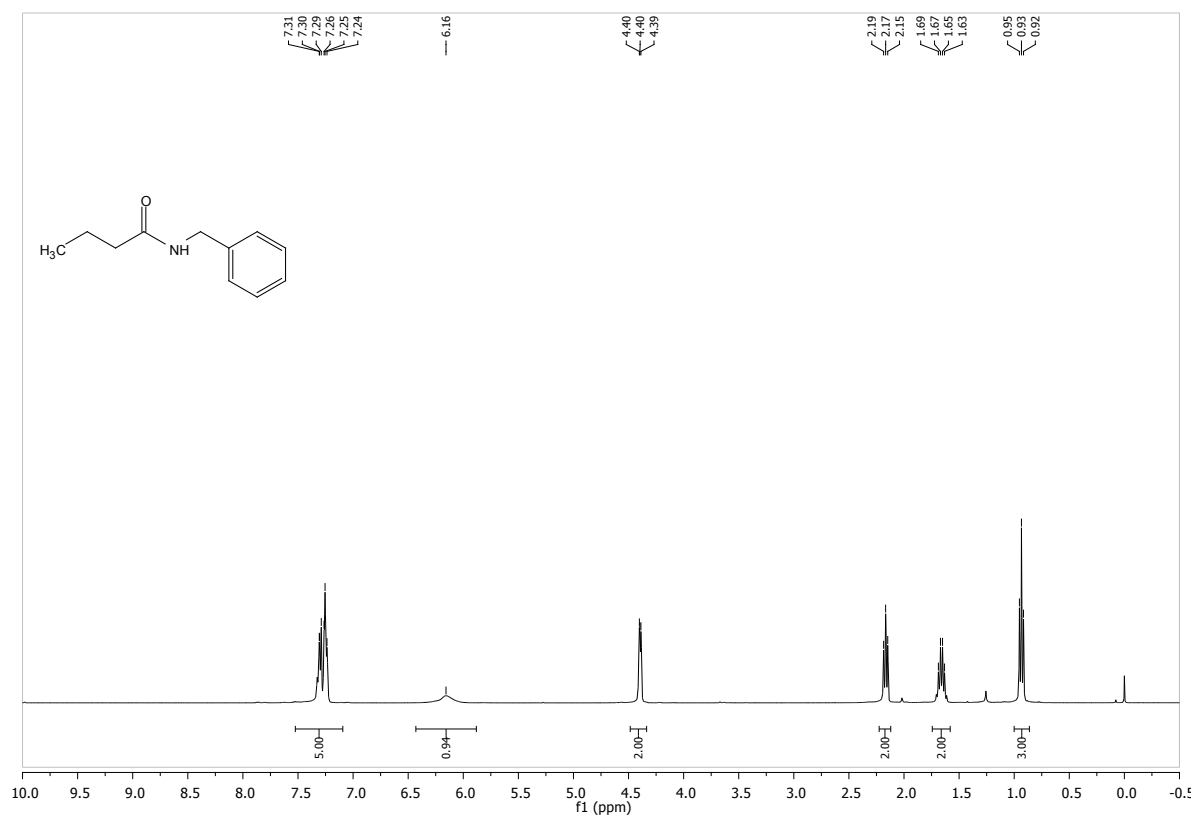
N-Benzyl-formamide (5)



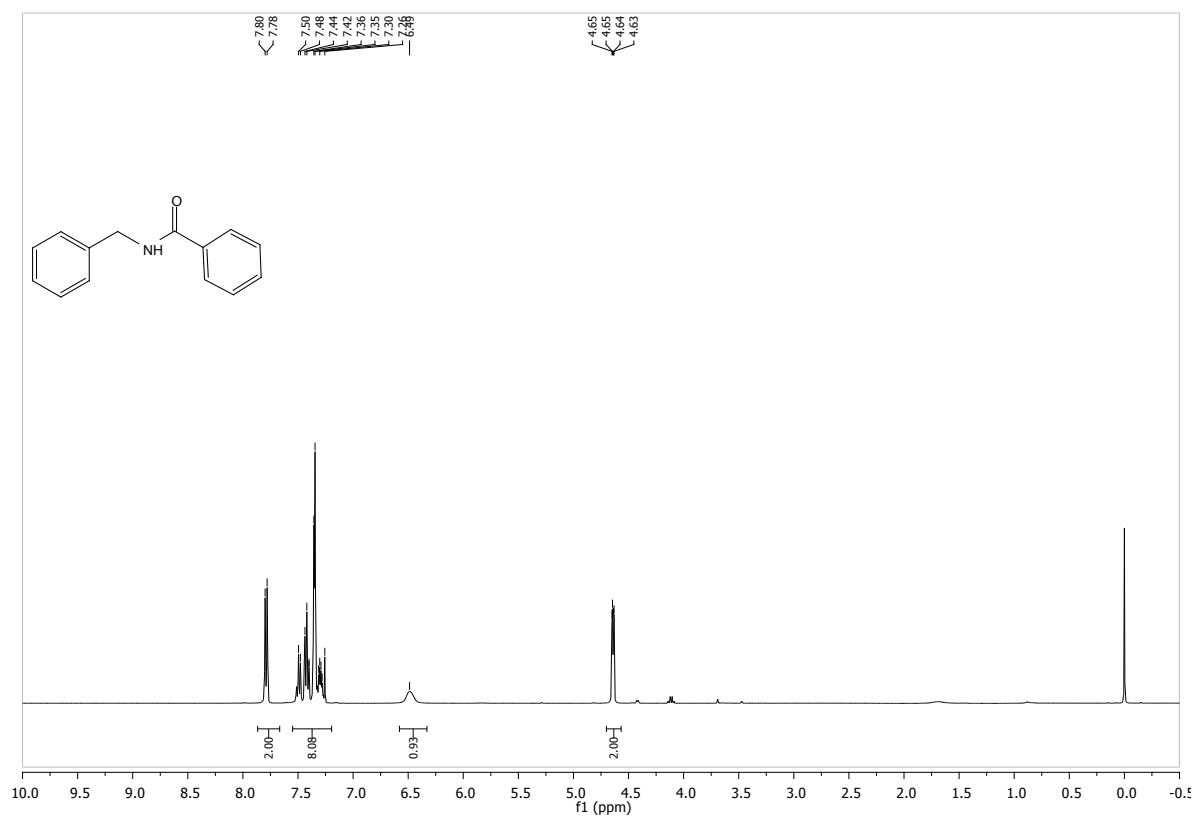
N-Benzyl-acetamide (6)



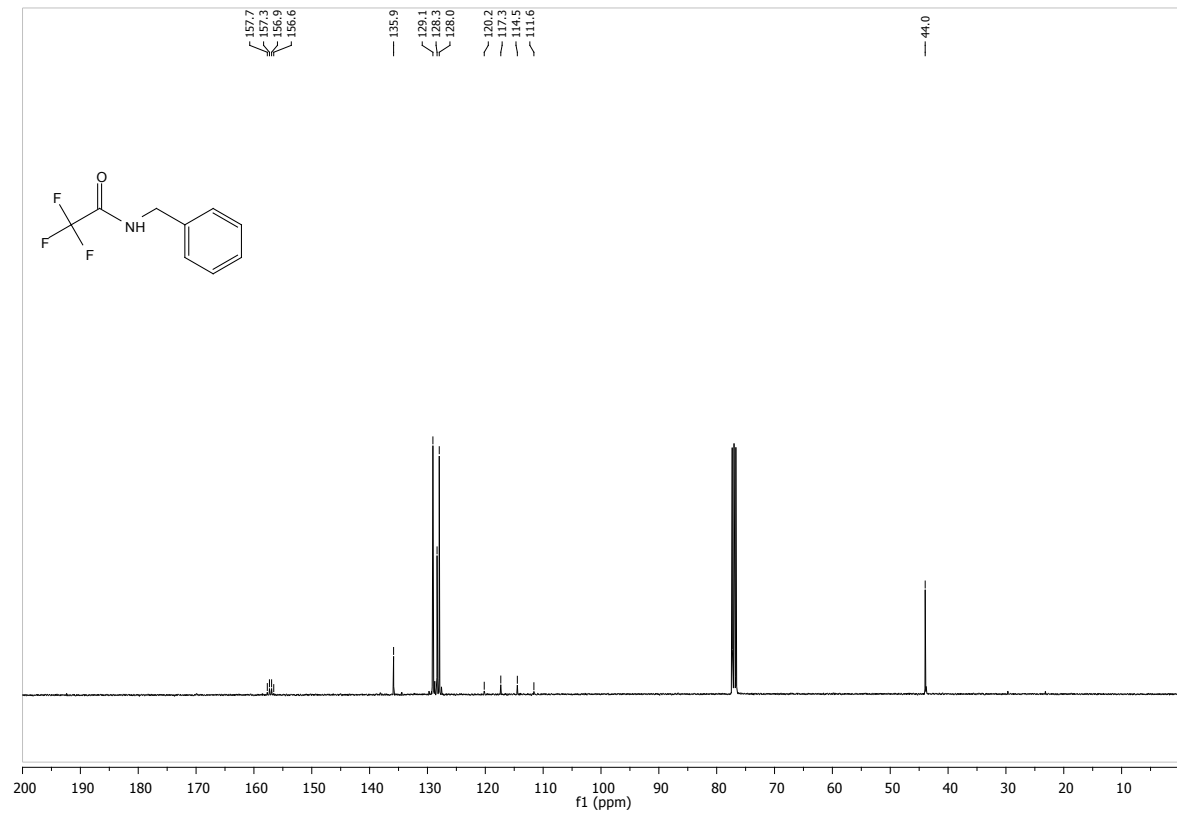
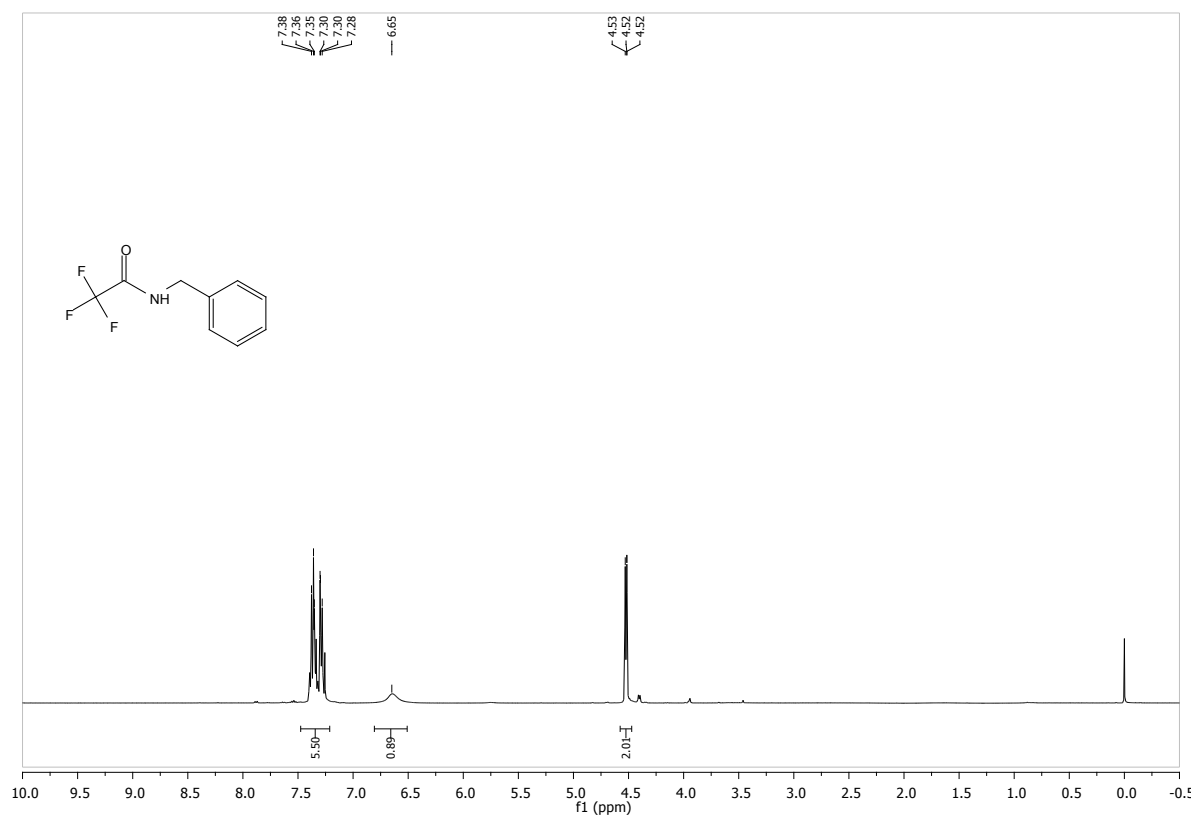
N-Benzyl-butyramide (7)



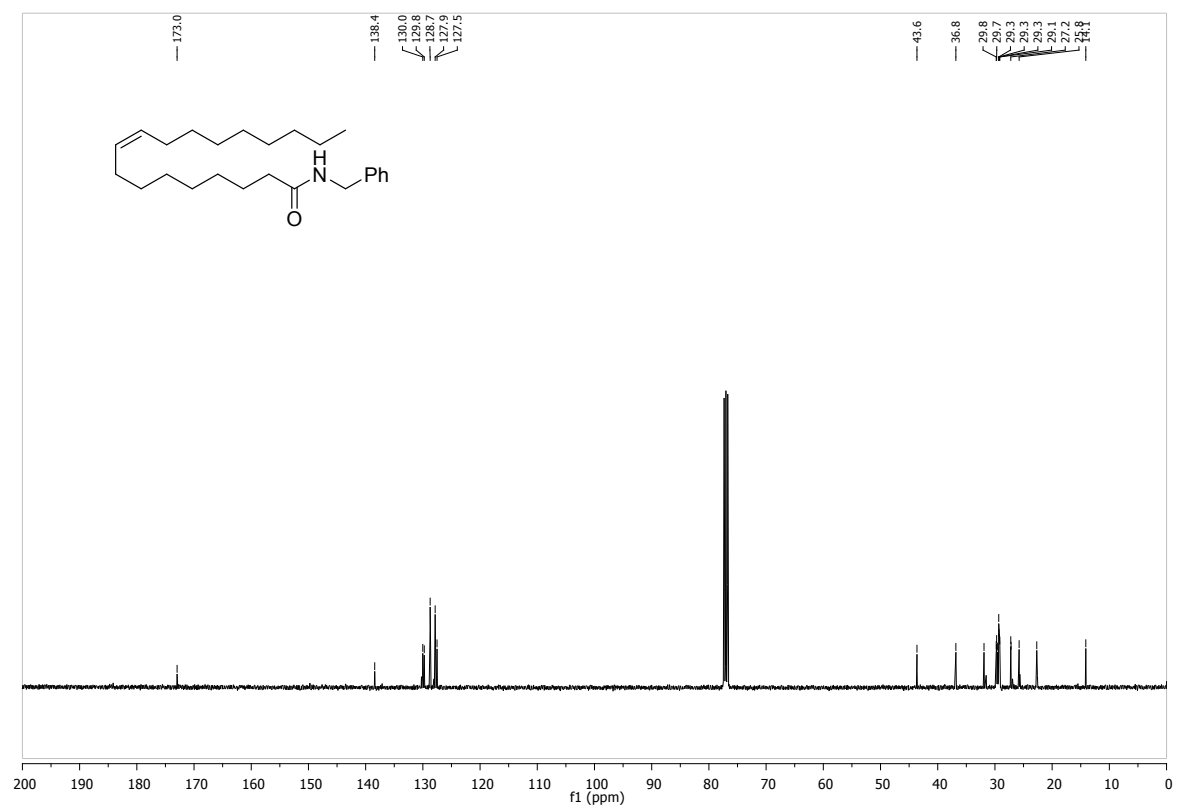
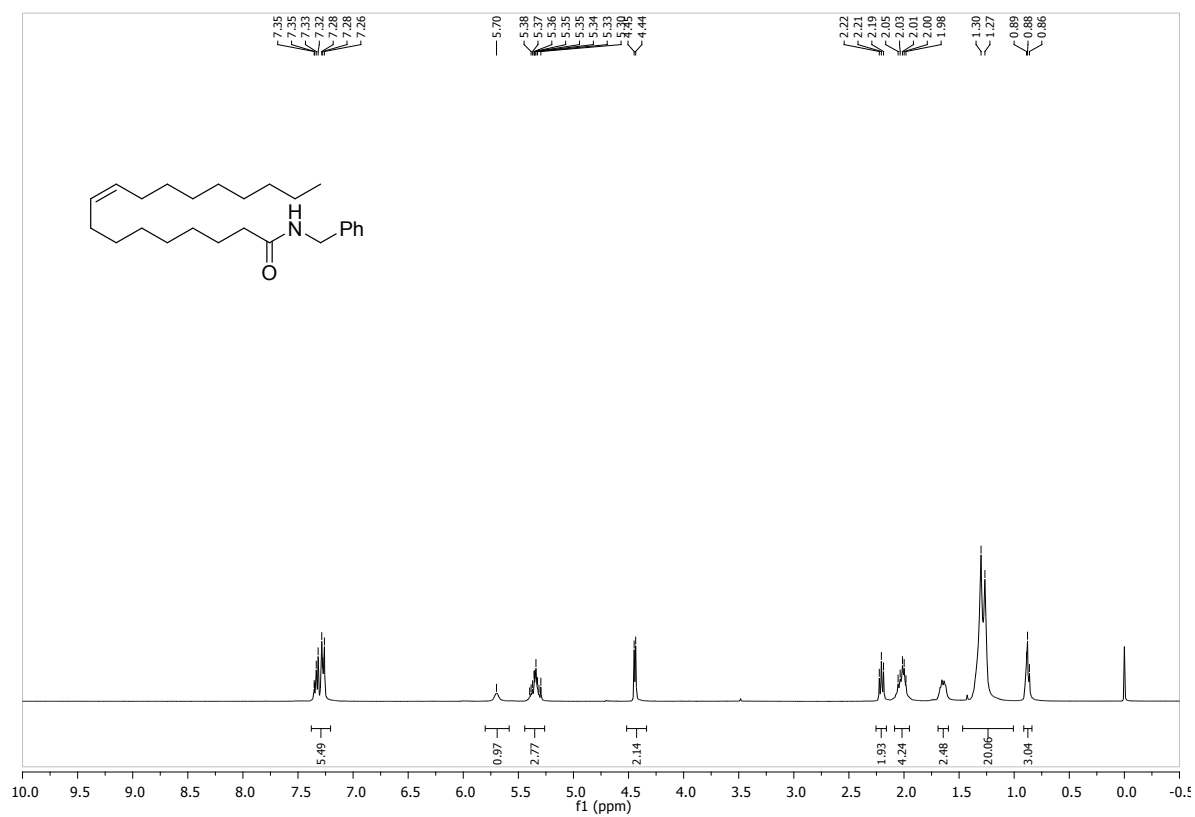
N-Benzyl-benzamide (8)



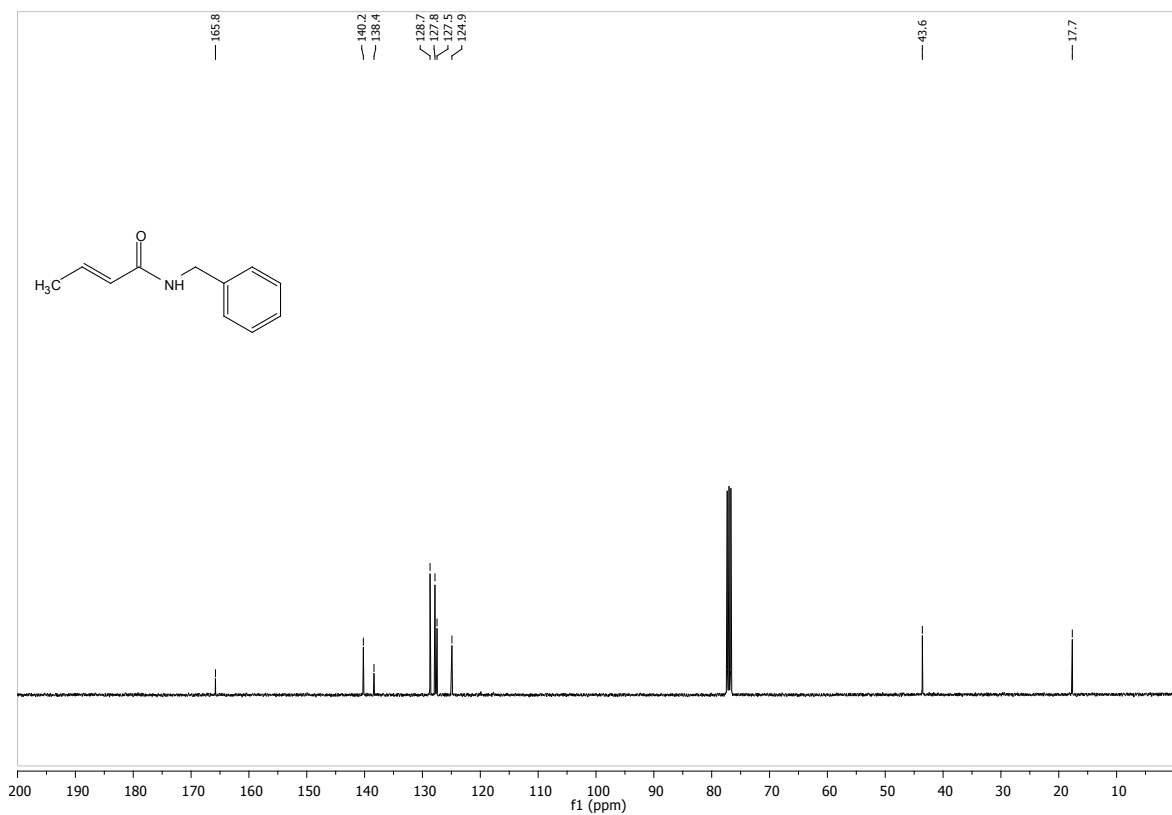
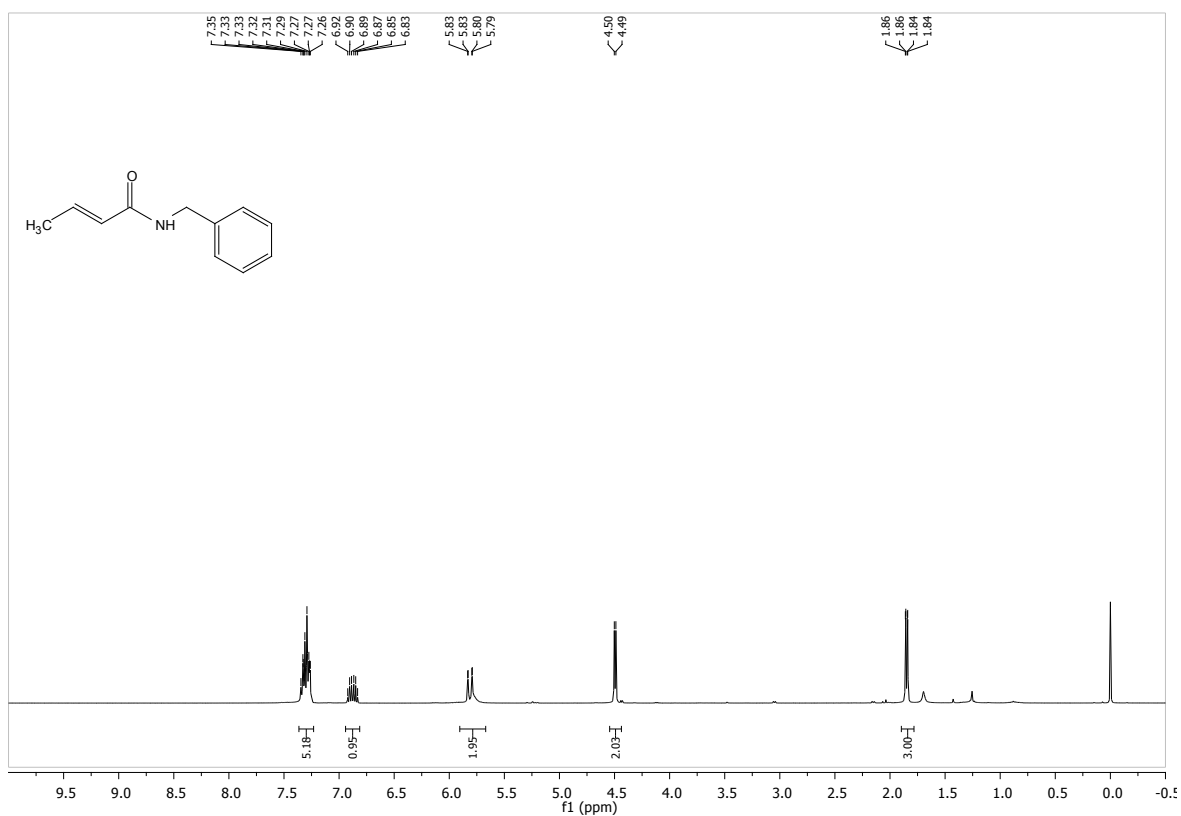
N-Benzyl-2,2,2-trifluoro-acetamide (9)



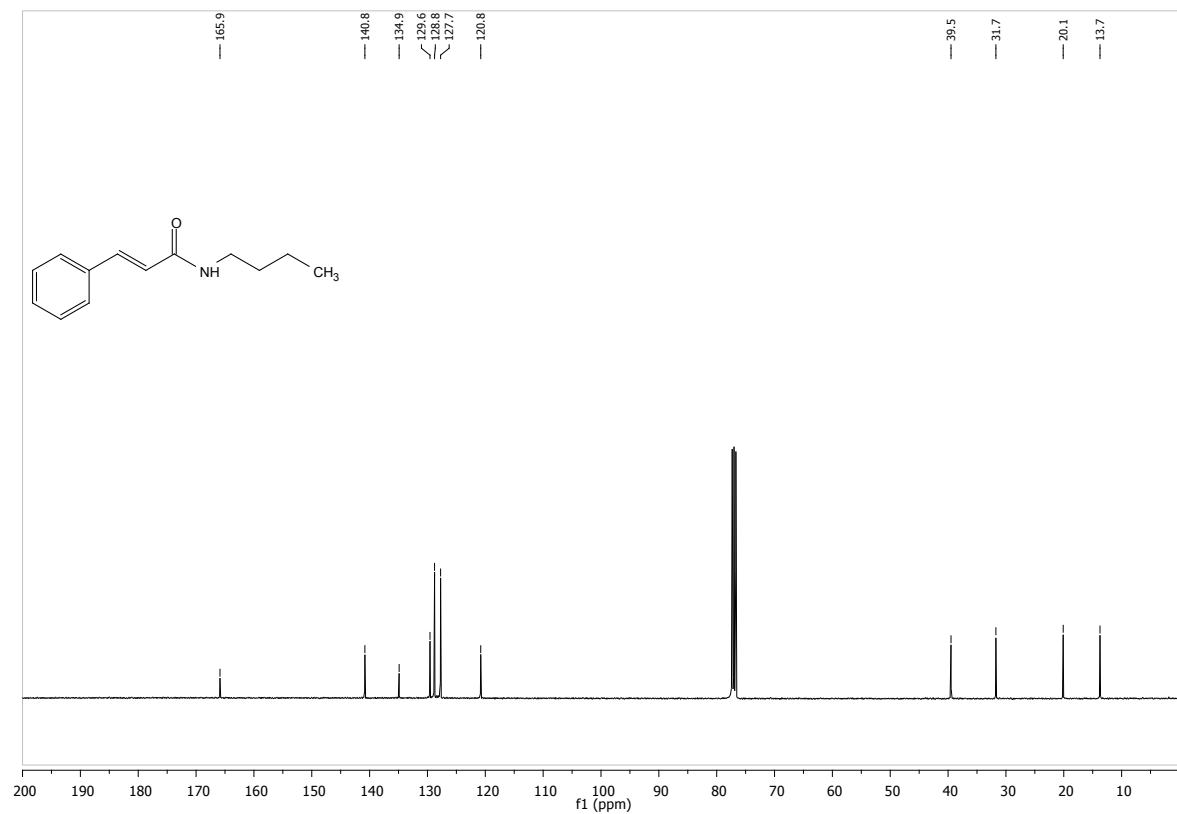
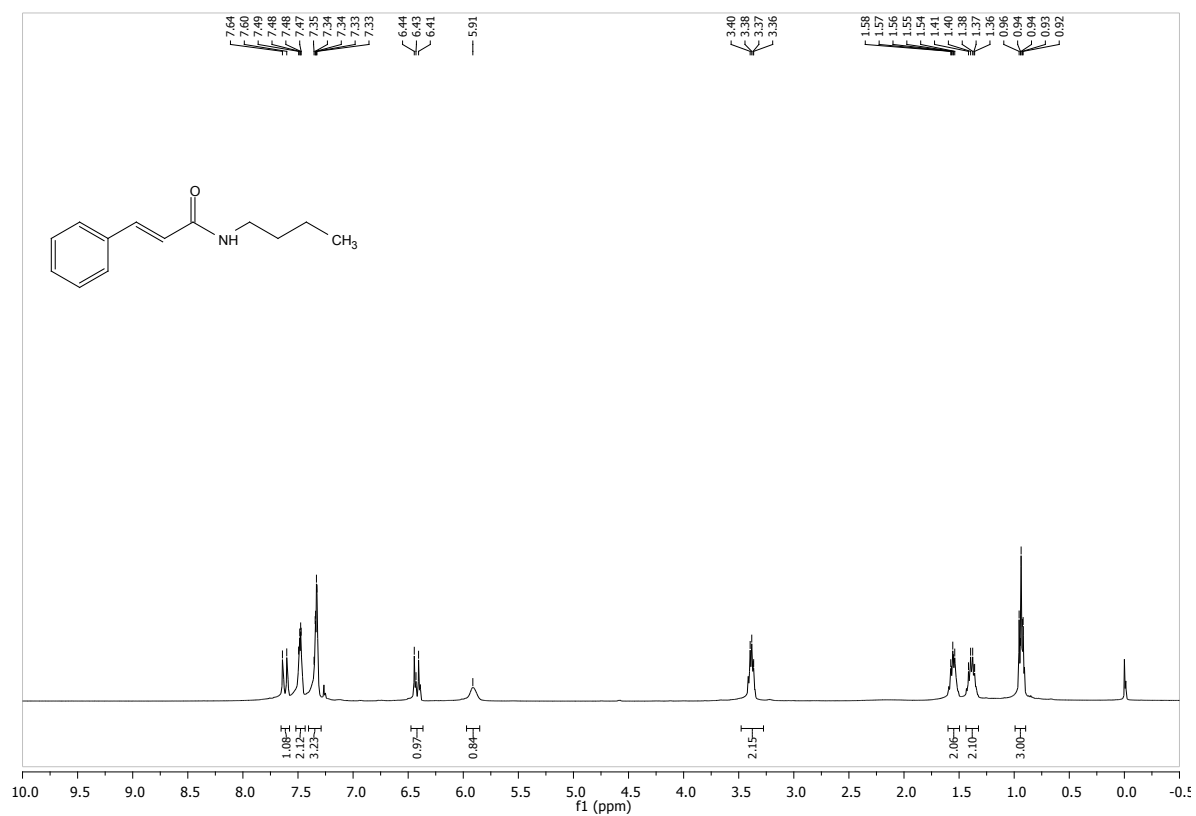
N-benzyleamide (10)



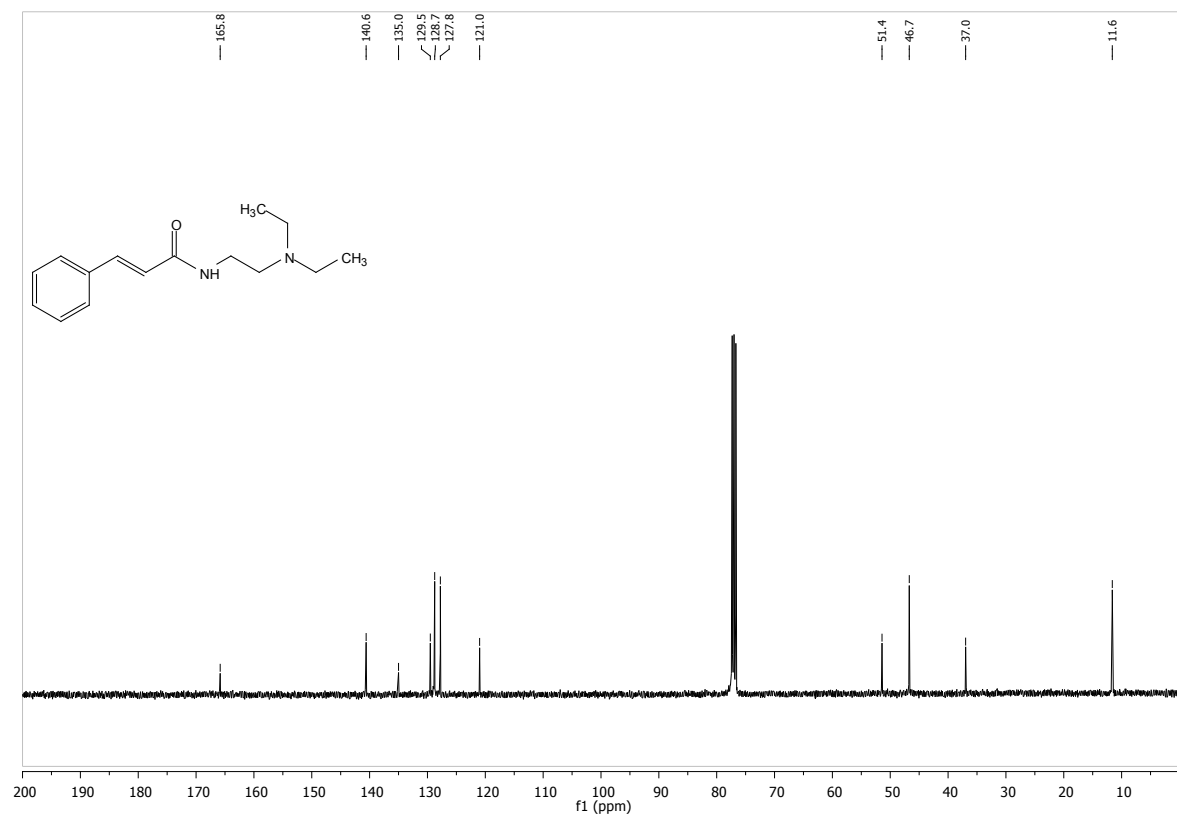
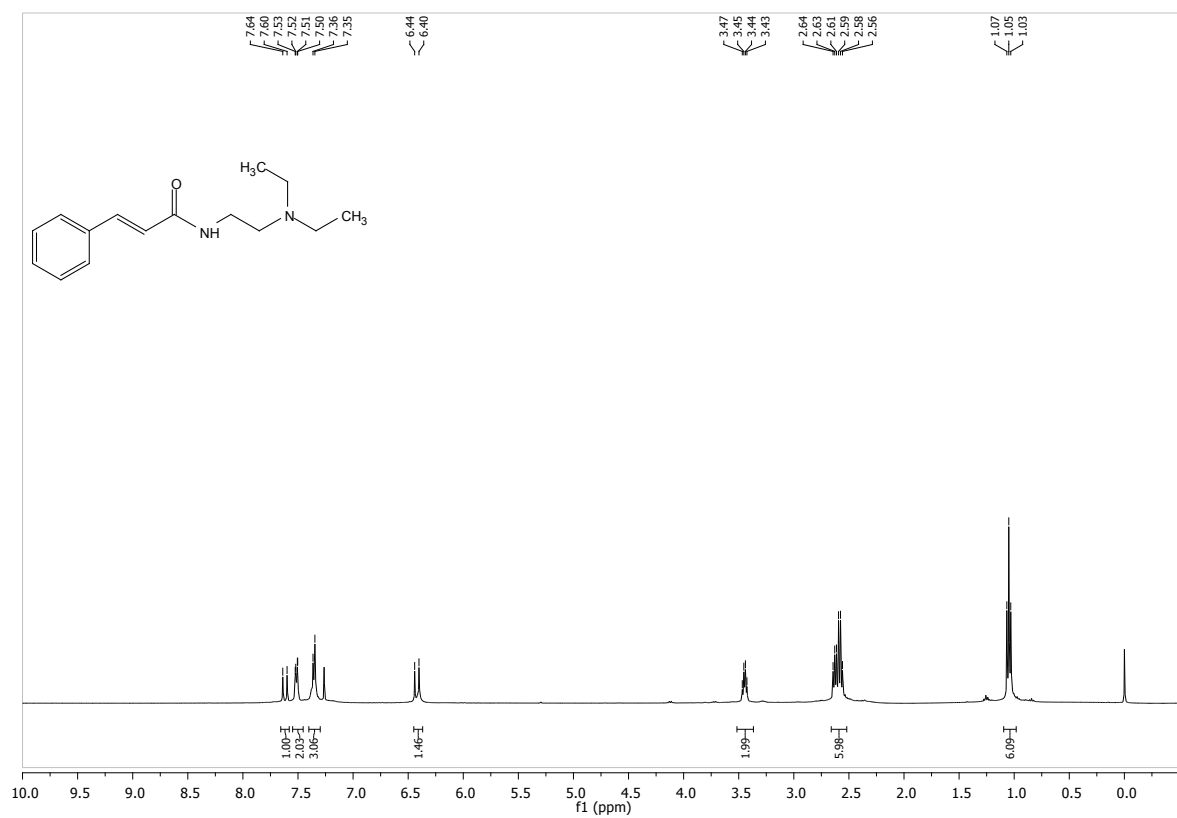
2-butenic acid benzylamide (11)



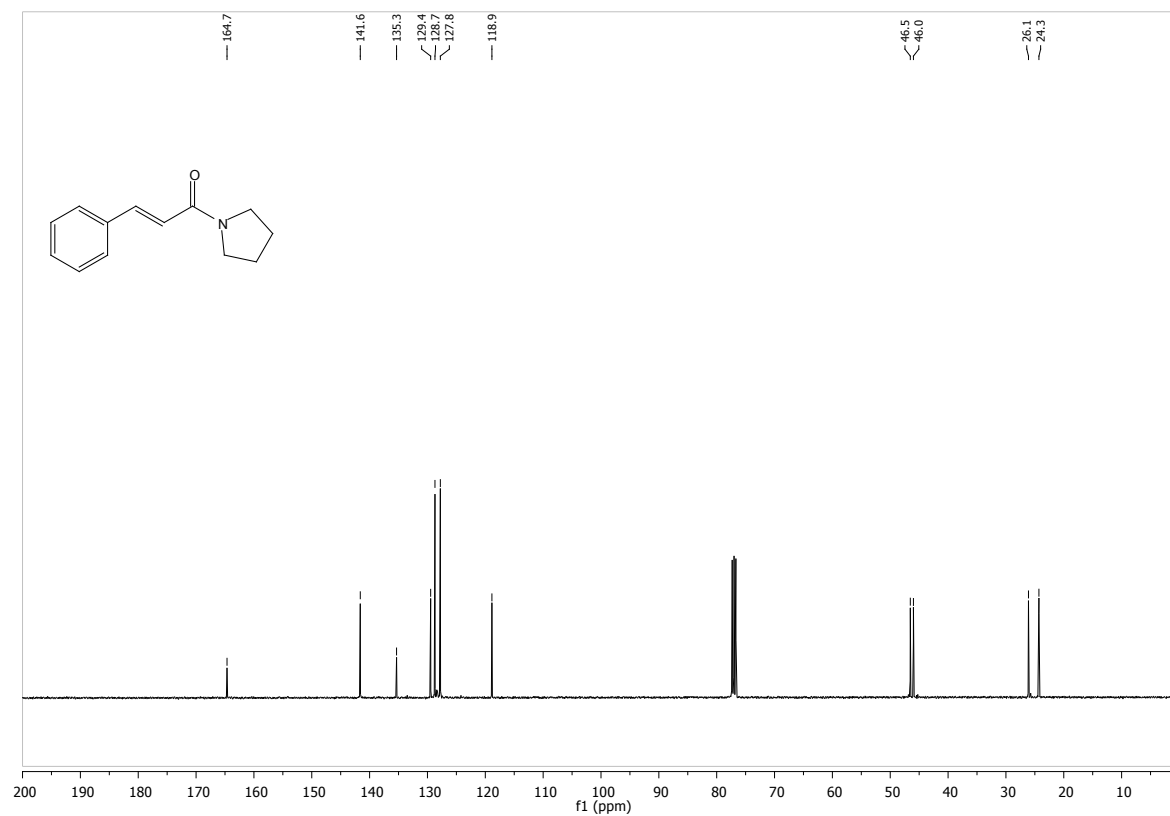
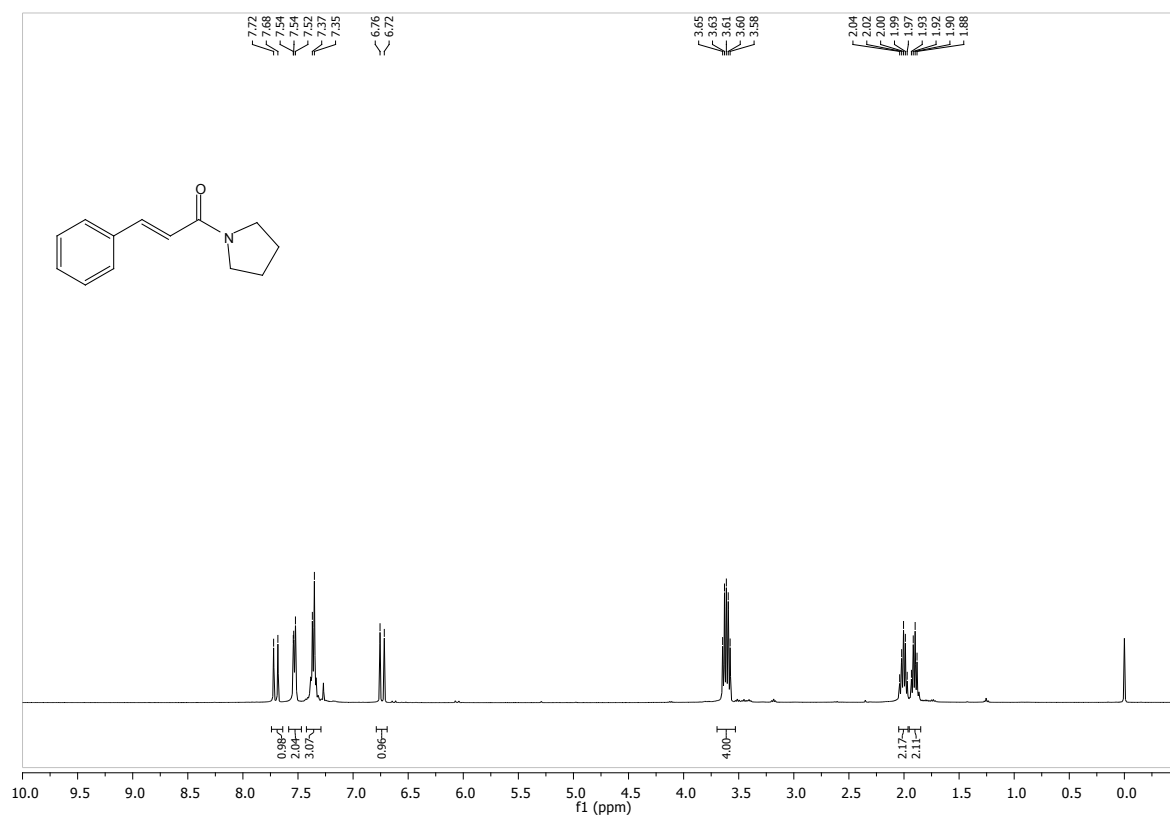
N-Butyl-3-phenyl-acrylamide (12)



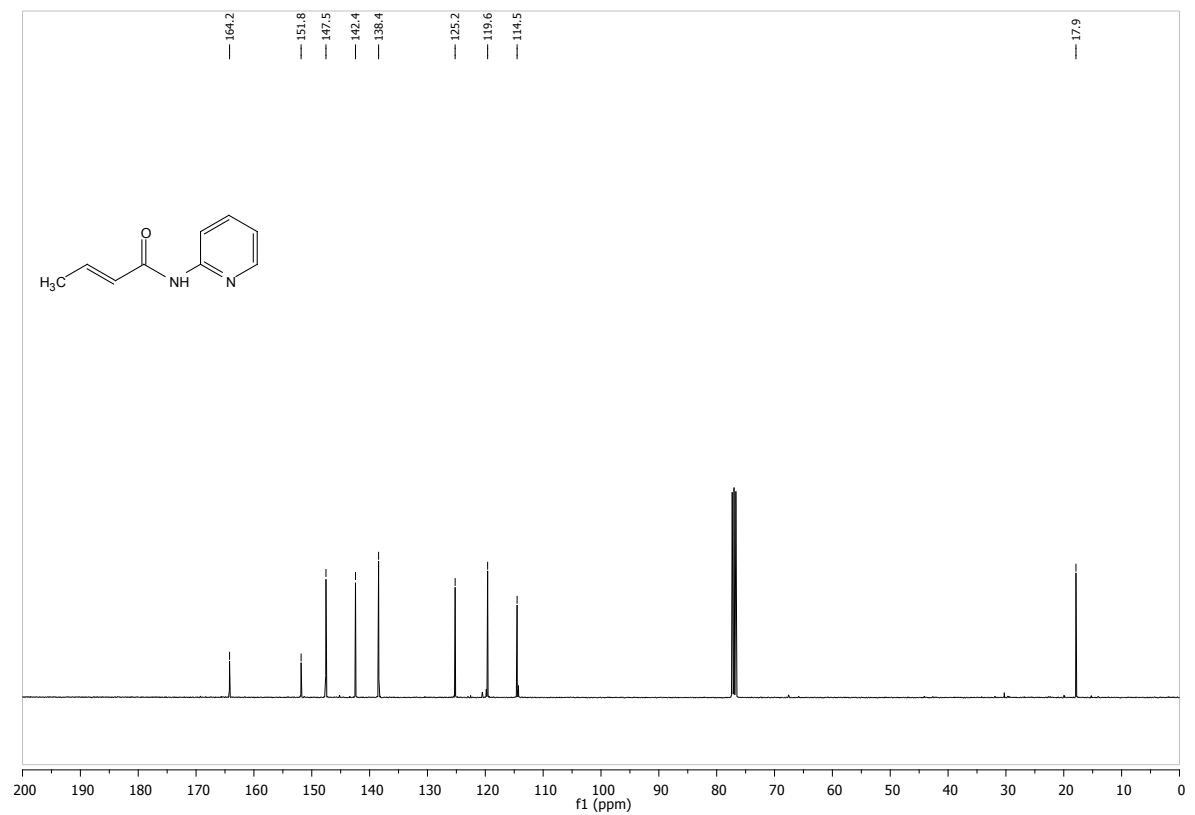
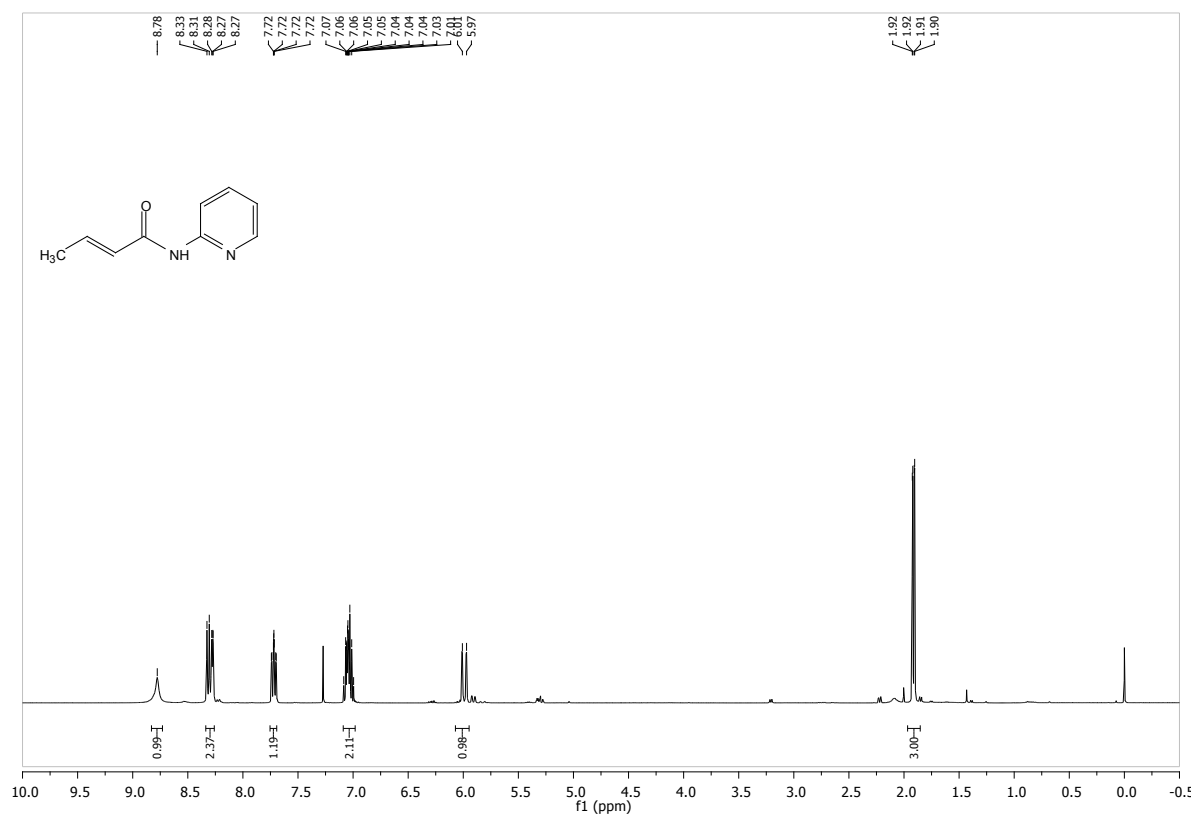
N-(2-Diethylamino-ethyl)-3-phenyl-acrylamide (13)



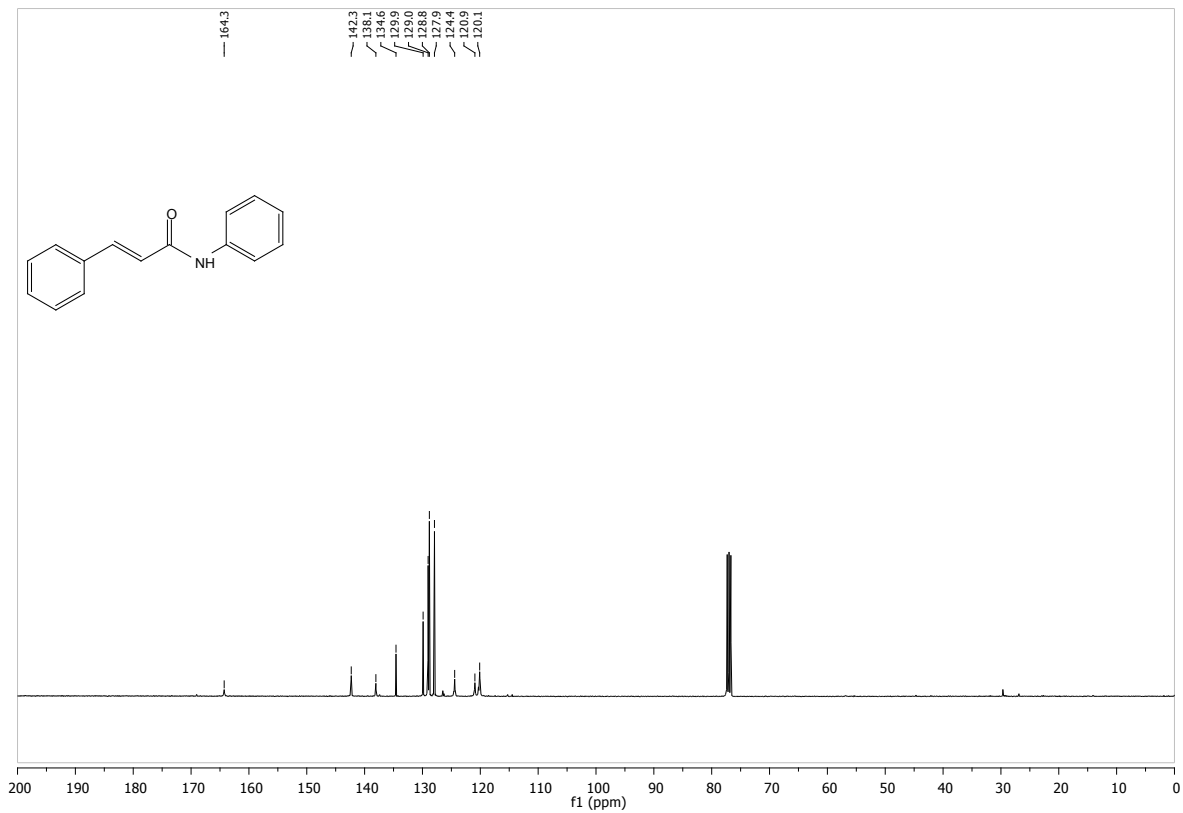
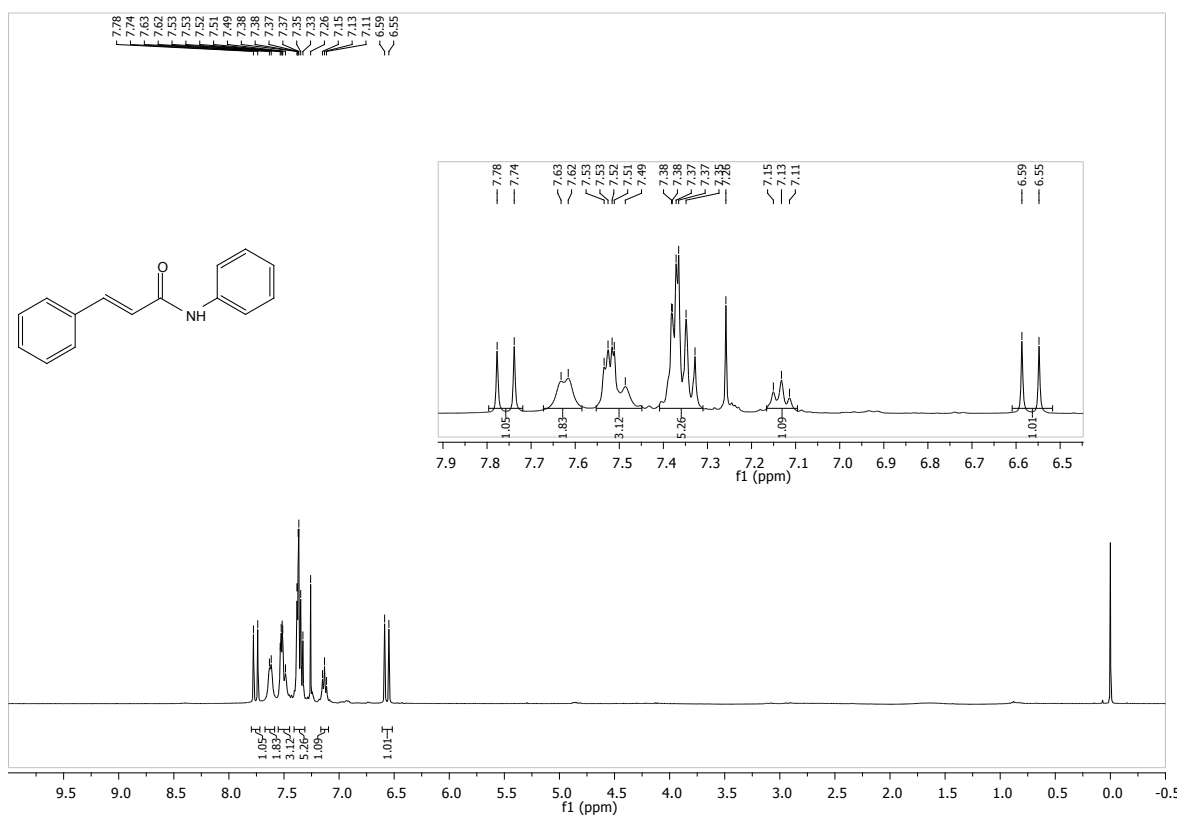
N-pyrrolidyl-1-cinnamoylamide (14)



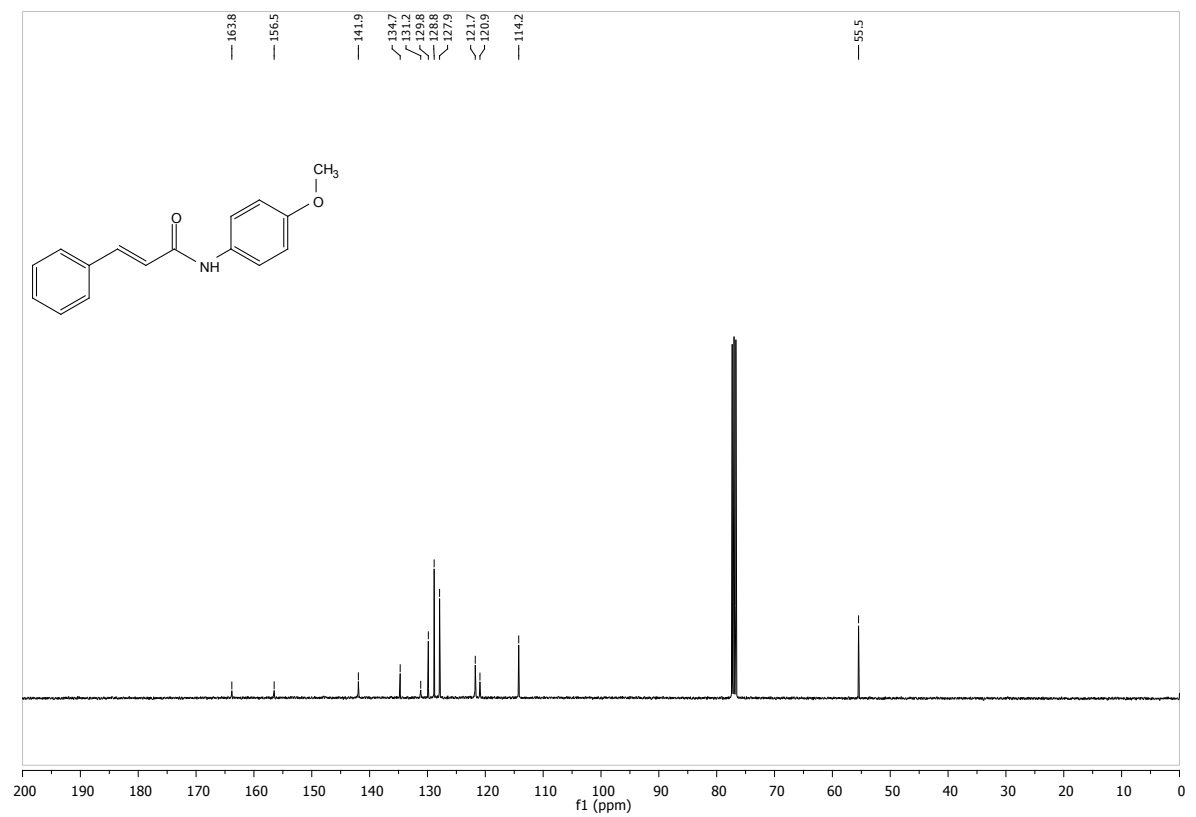
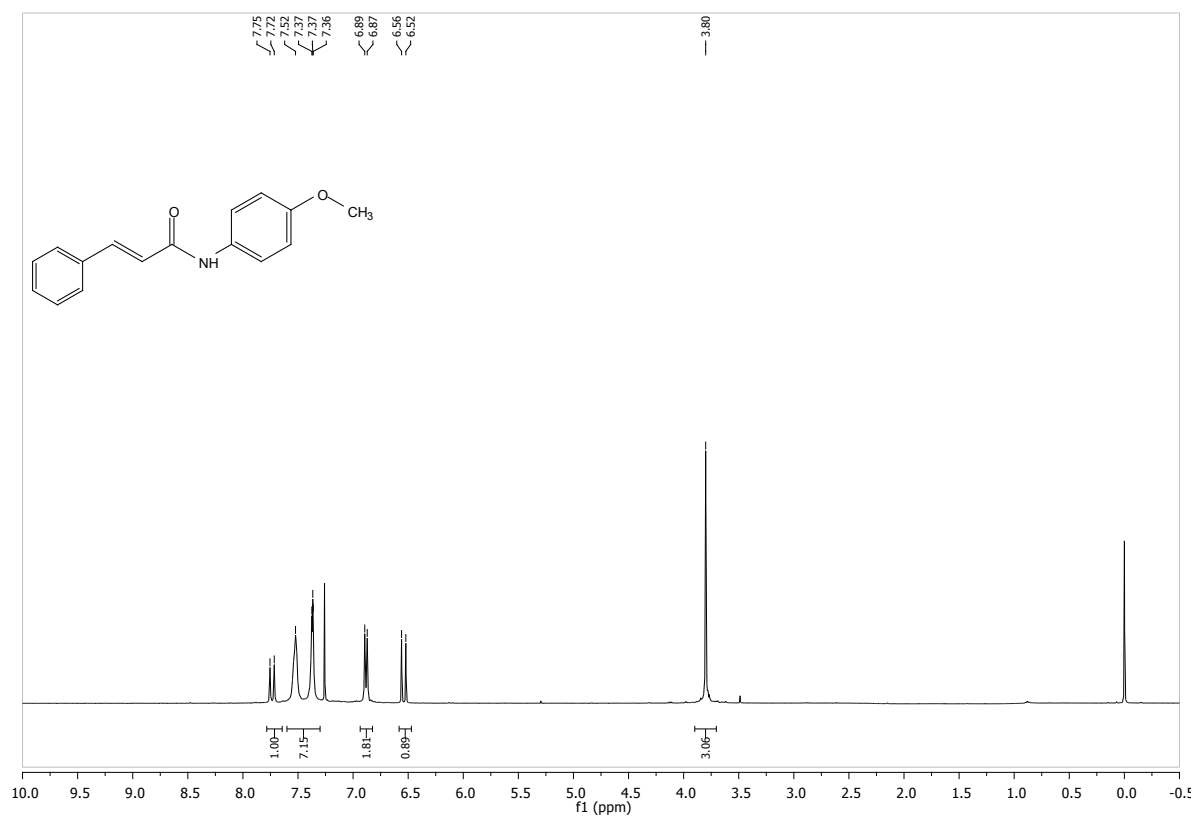
(E)-N-(pyridin-2-yl)but-2-enamide (15)



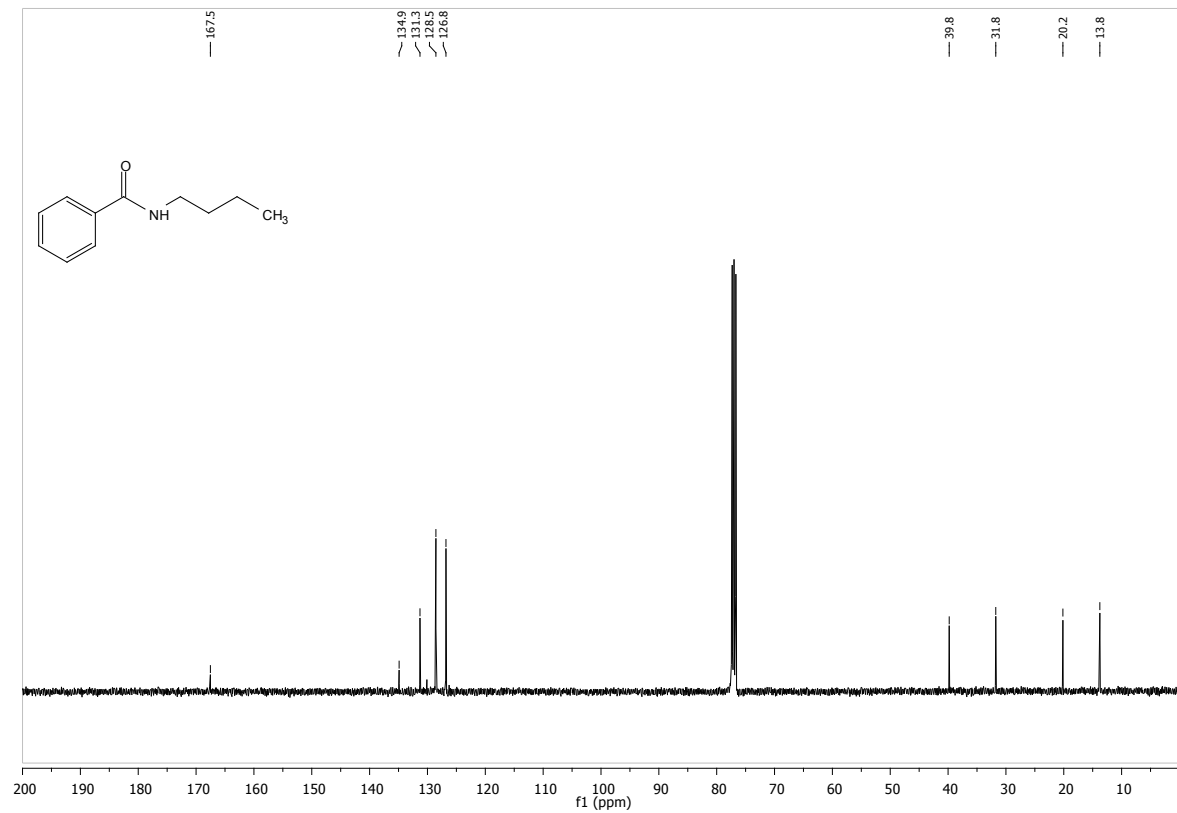
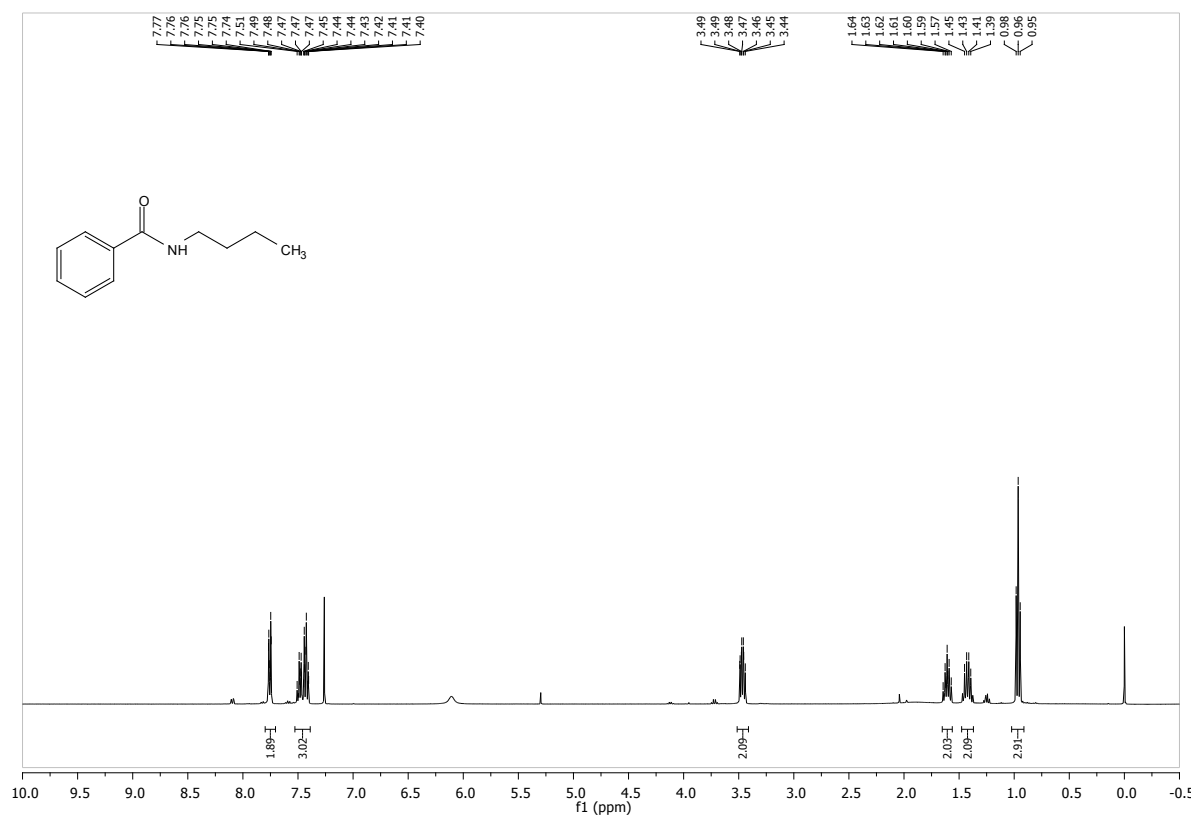
3-N-Diphenyl-acrylamide (16)



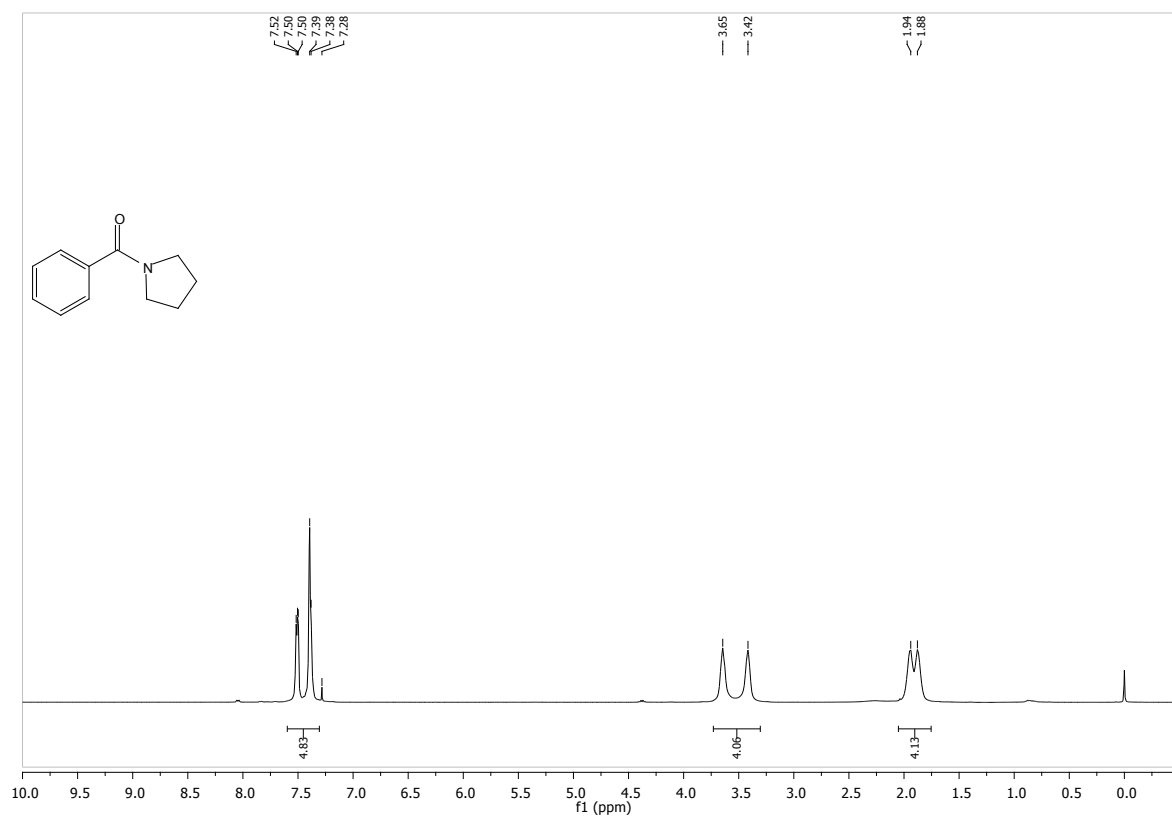
***N*-(4-Methoxy-phenyl)-3-phenyl-acrylamide (17)**

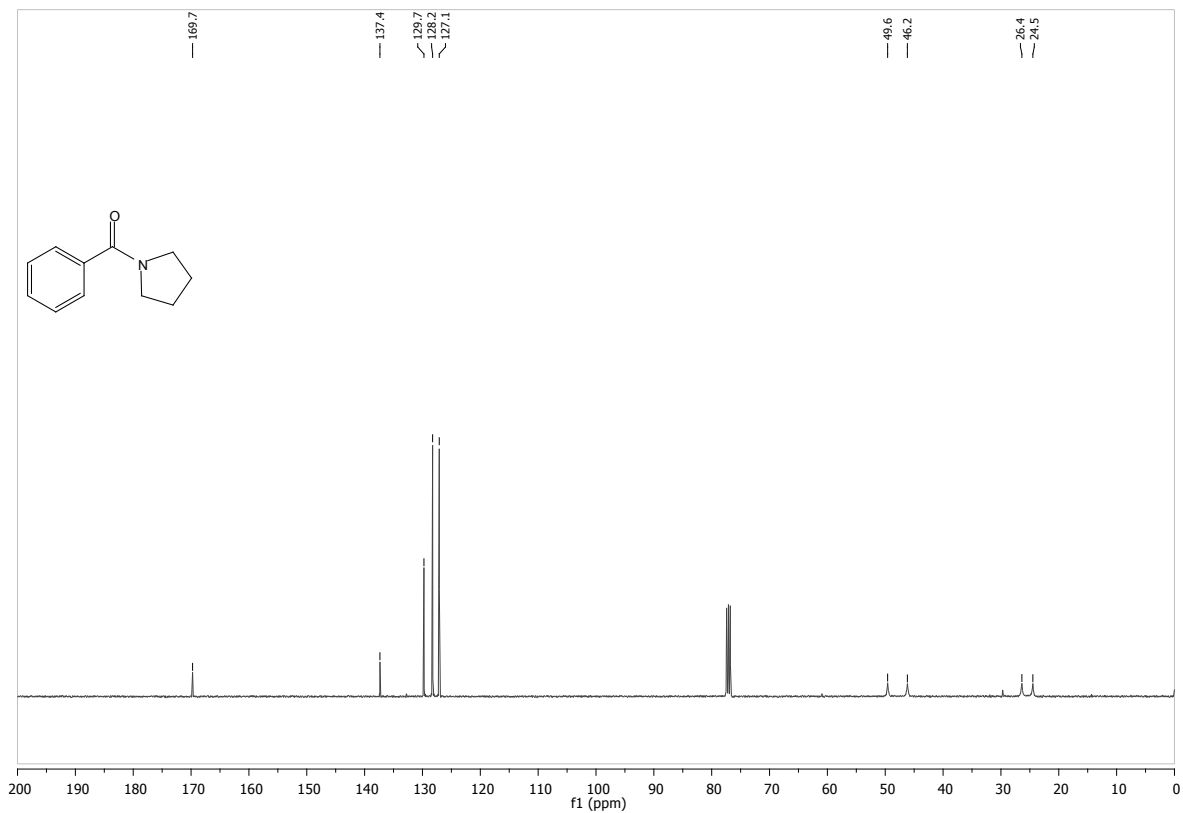


N-Butyl-benzamide (18)

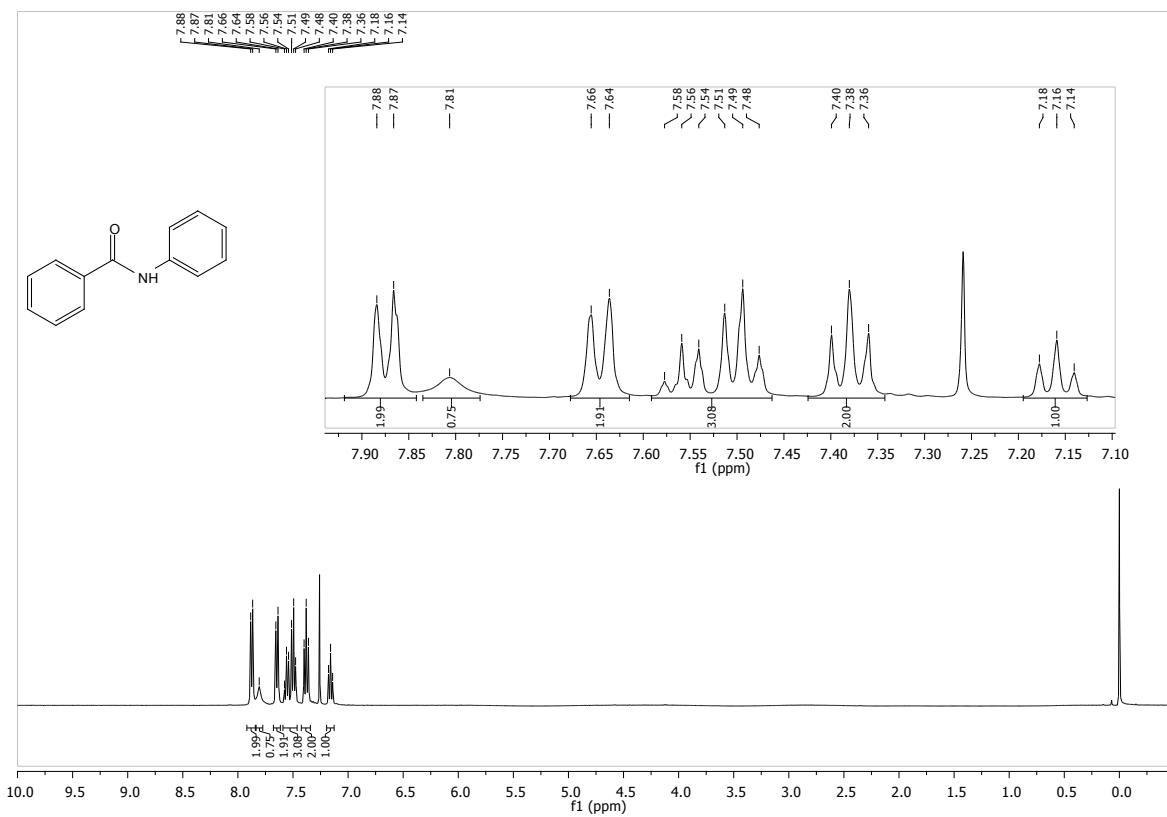


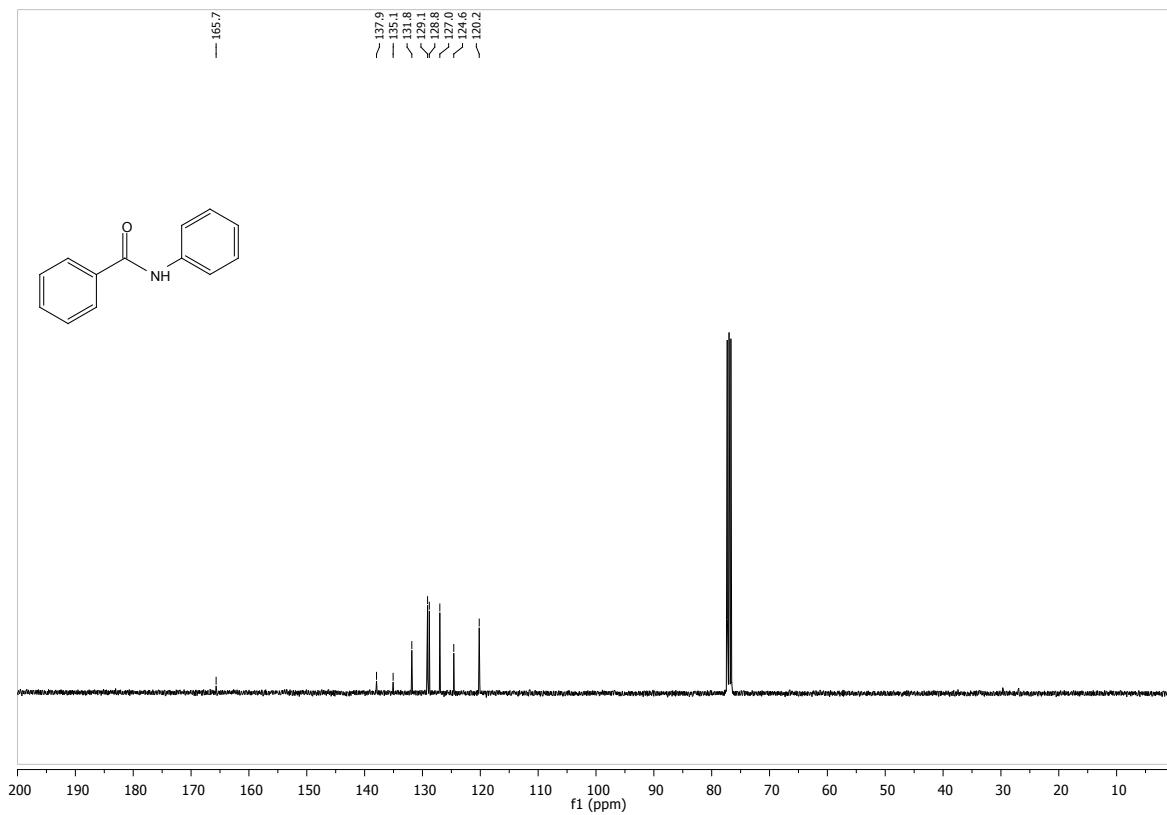
***N*-benzoylpyrrolidine (19)**



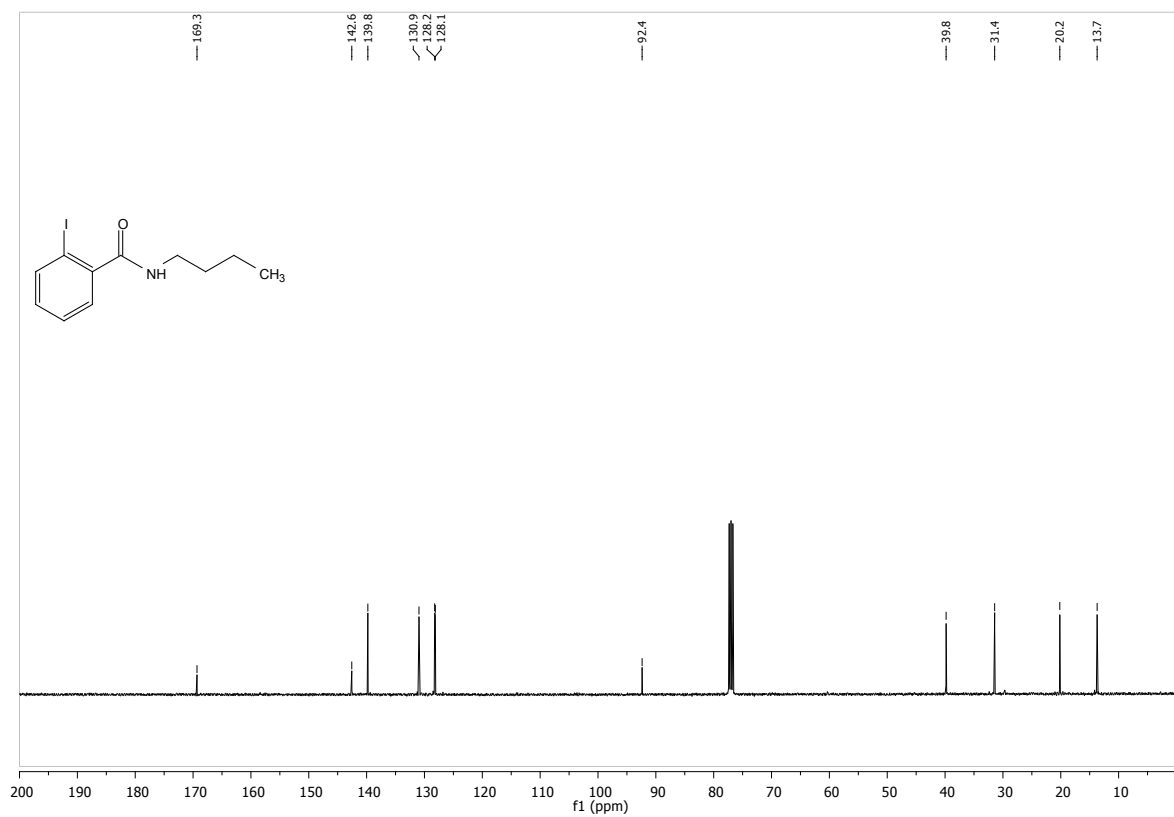
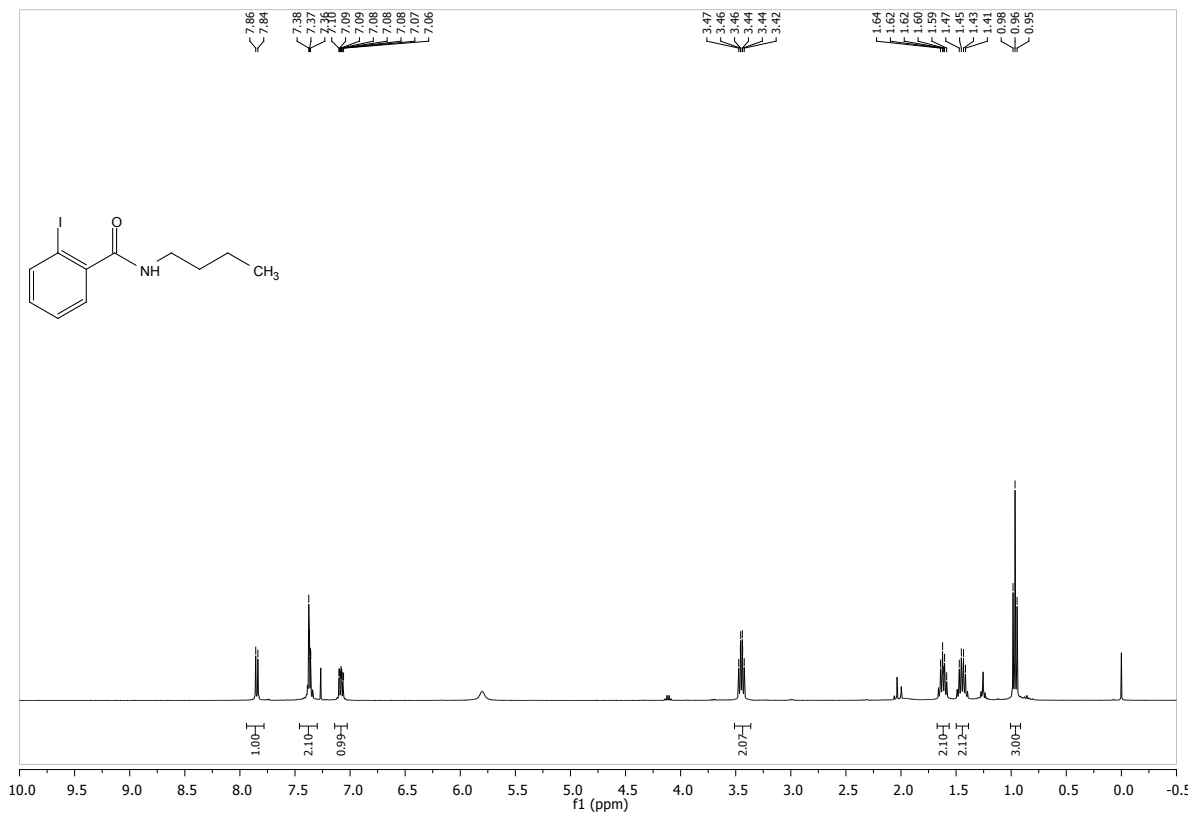


N-Phenyl-benzamide (20)

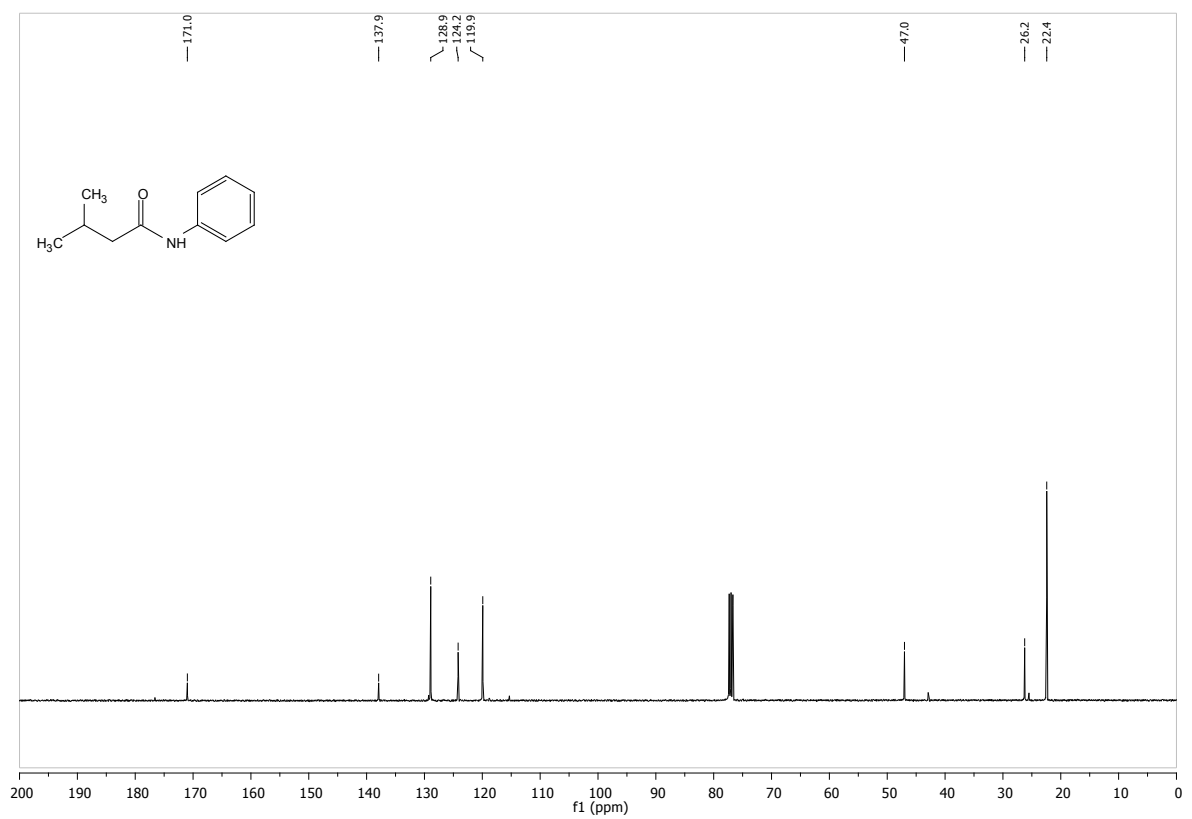
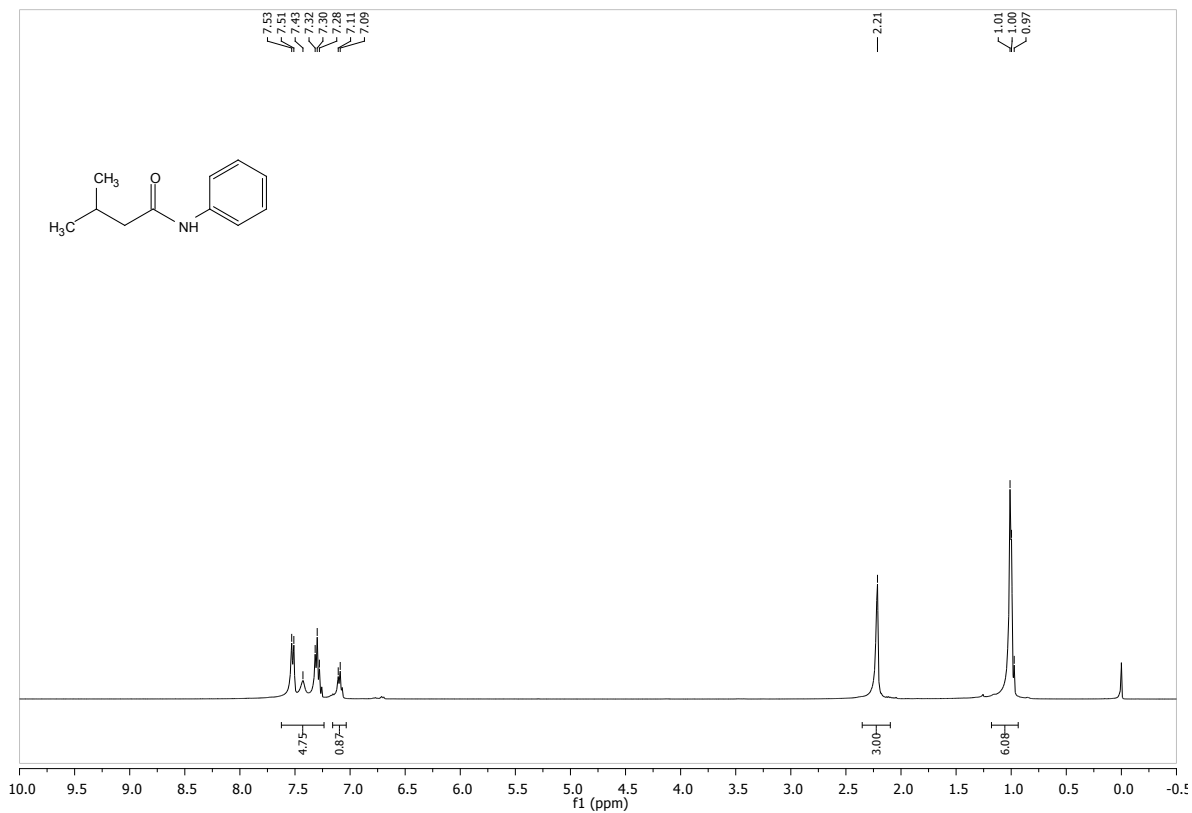




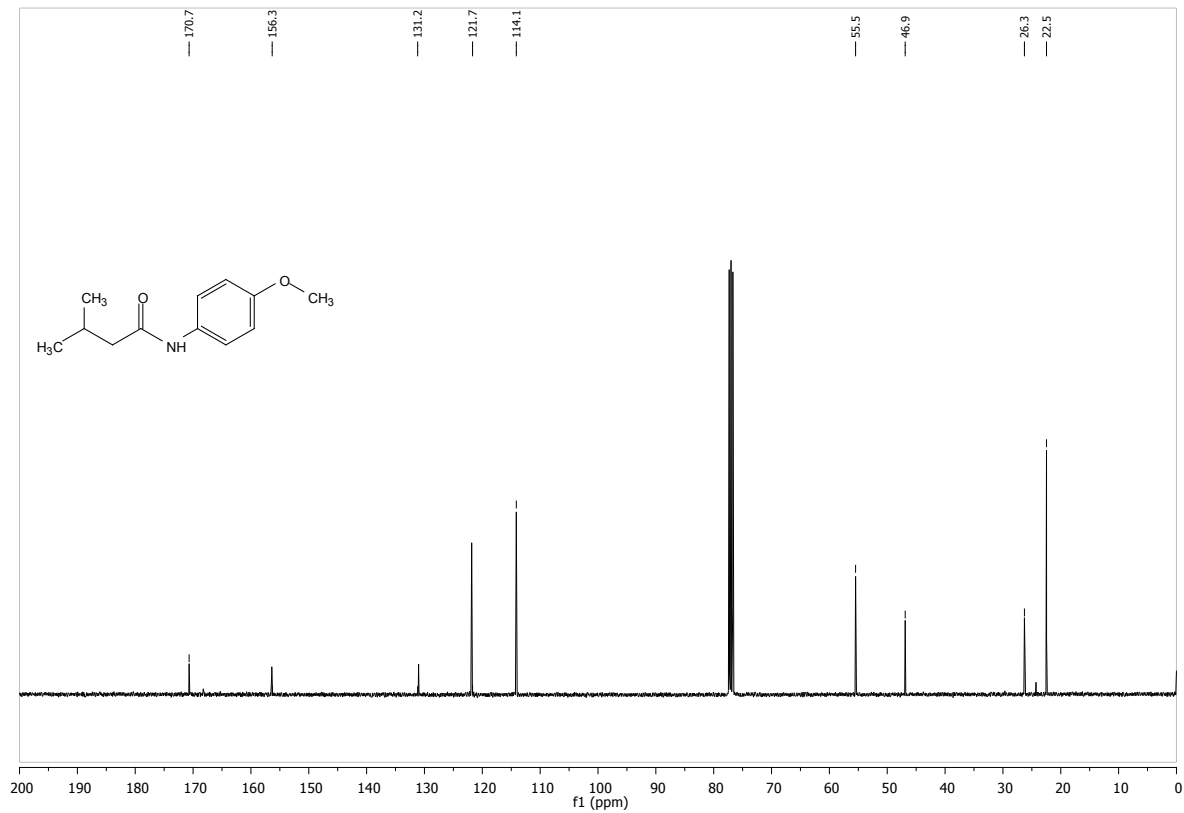
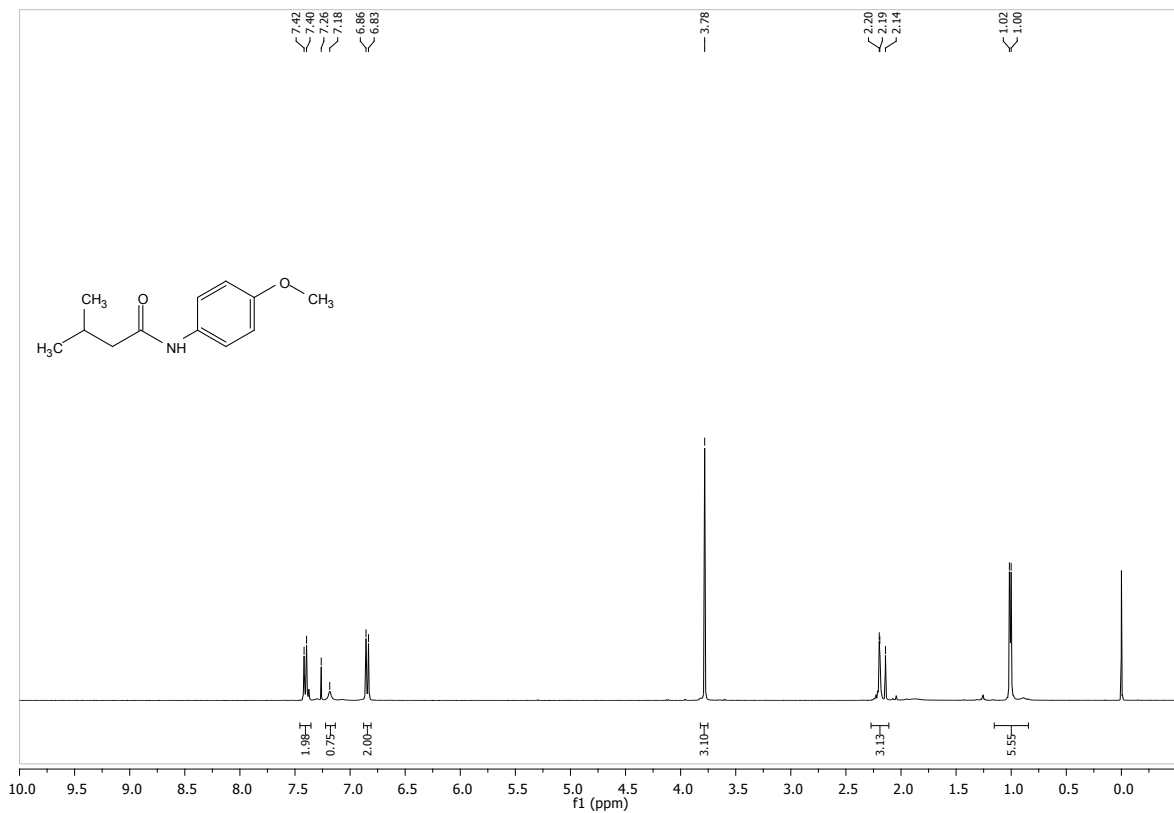
N-Butyl-2-iodo-benzamide (21)



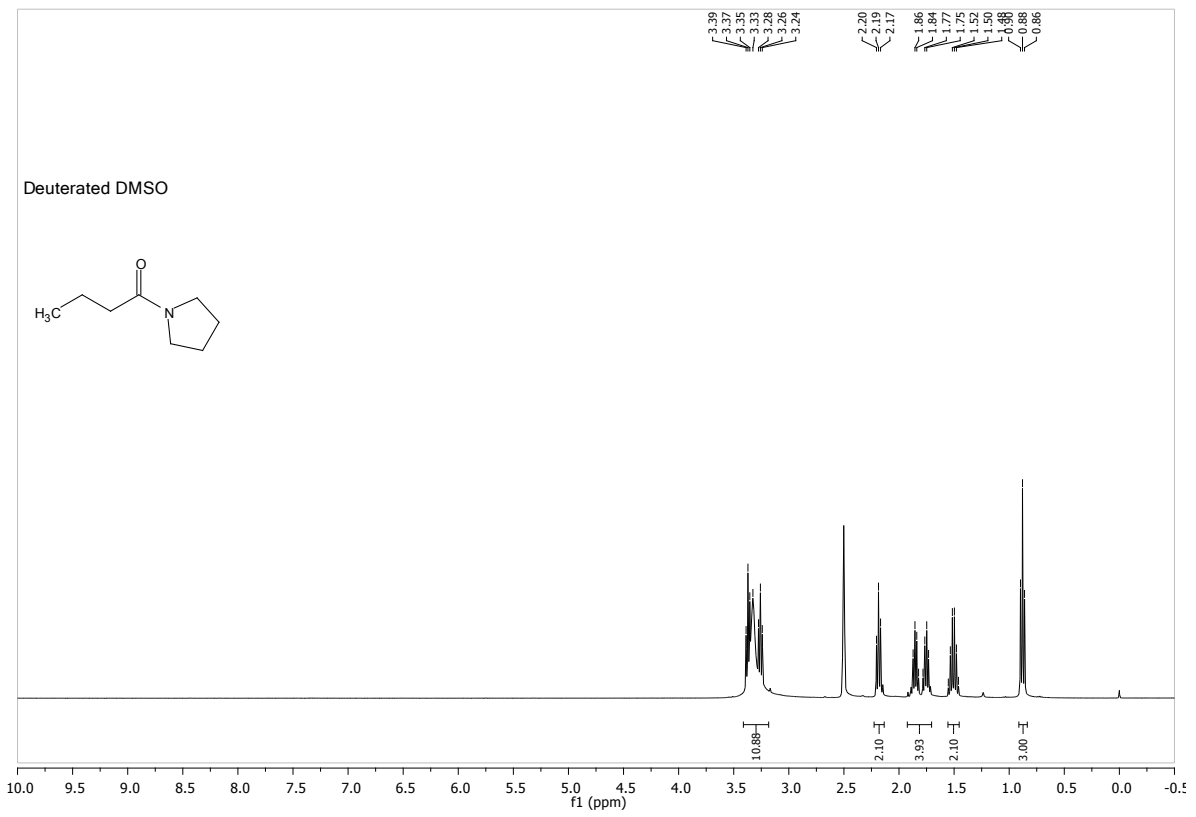
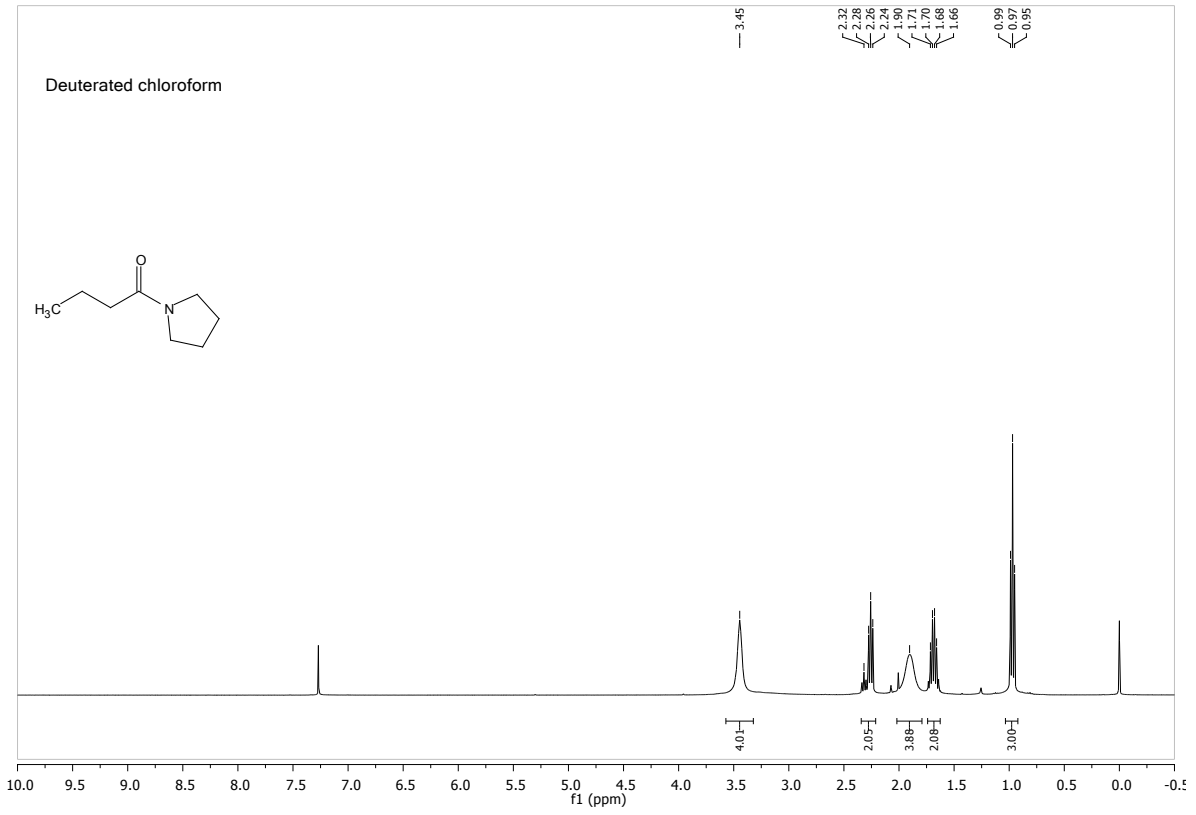
3-Methyl-N-phenyl-butanamide (22)

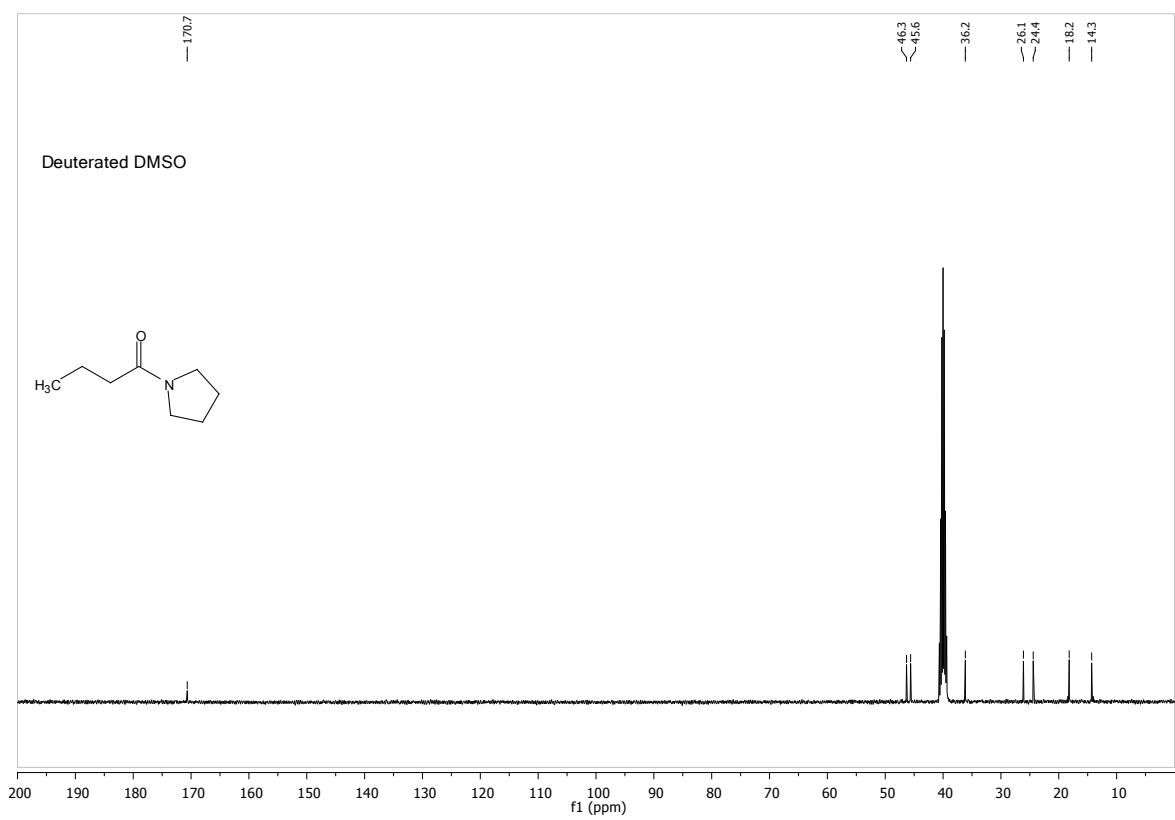
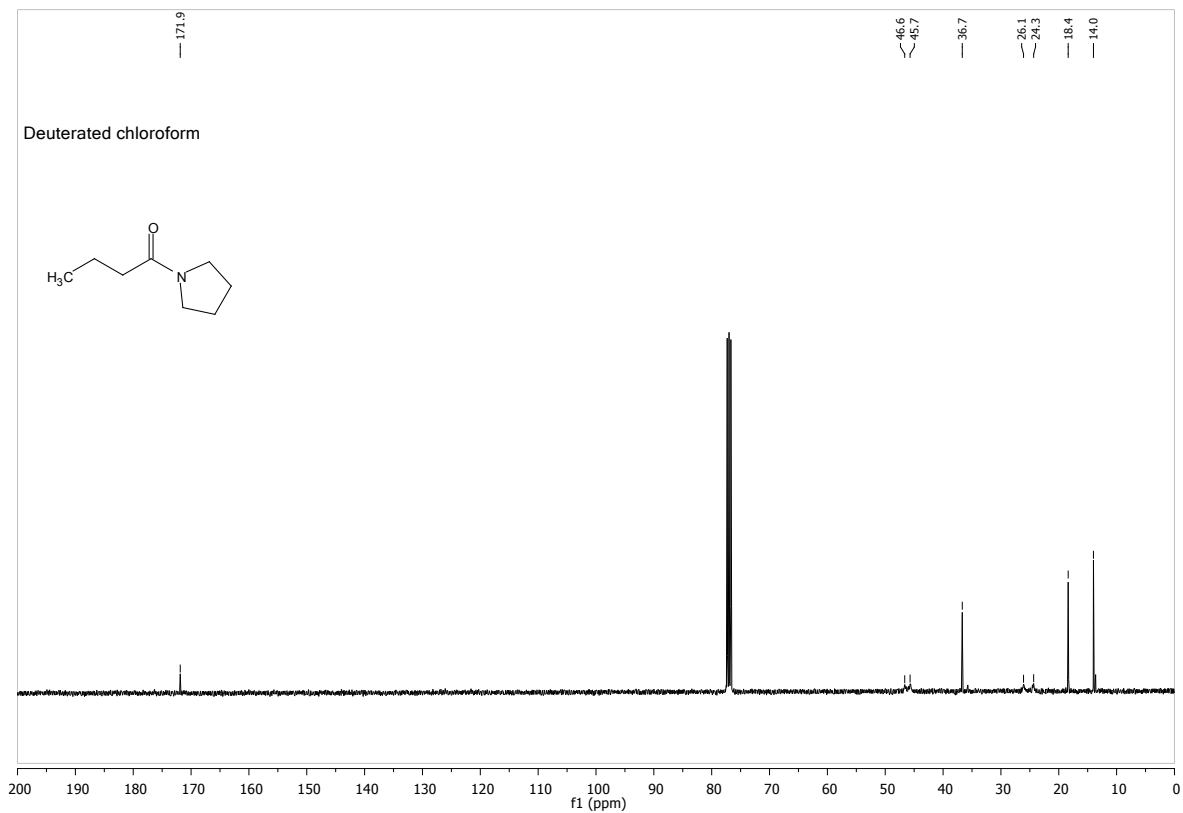


N-(4-Methoxy-phenyl)-3-methyl-butamide (23)

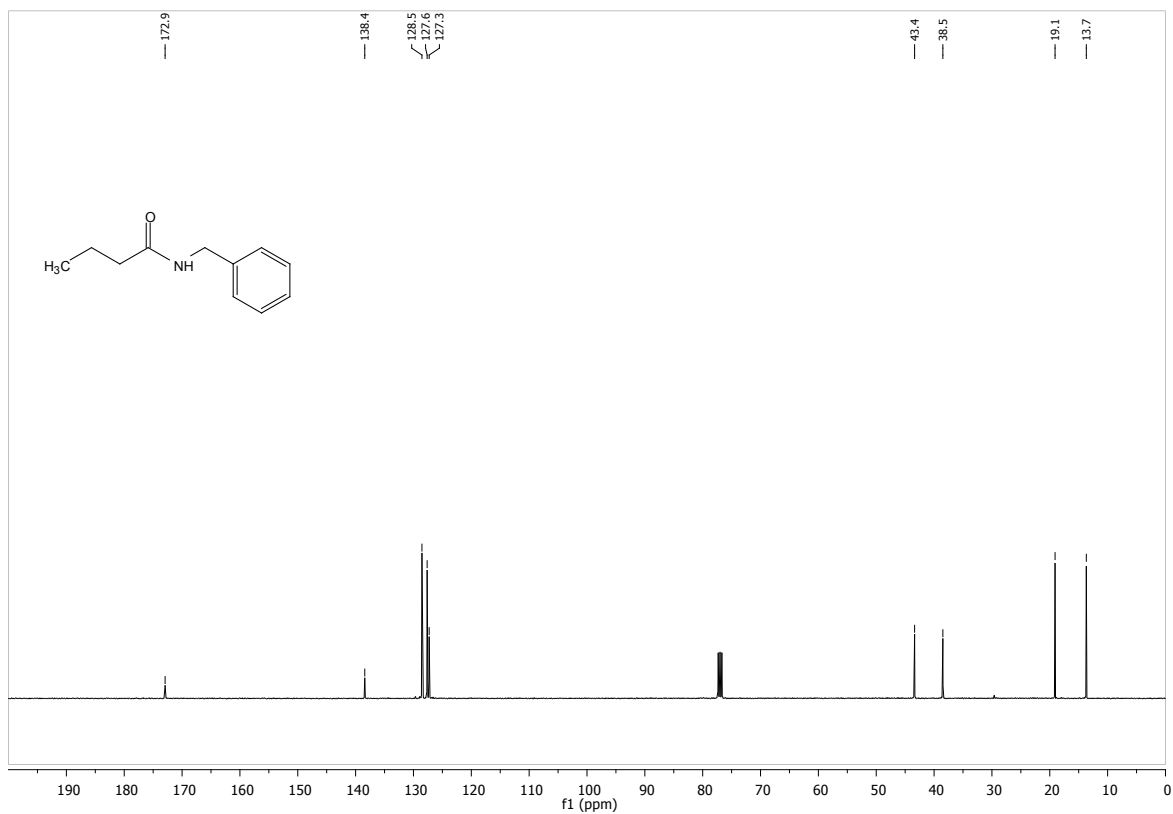
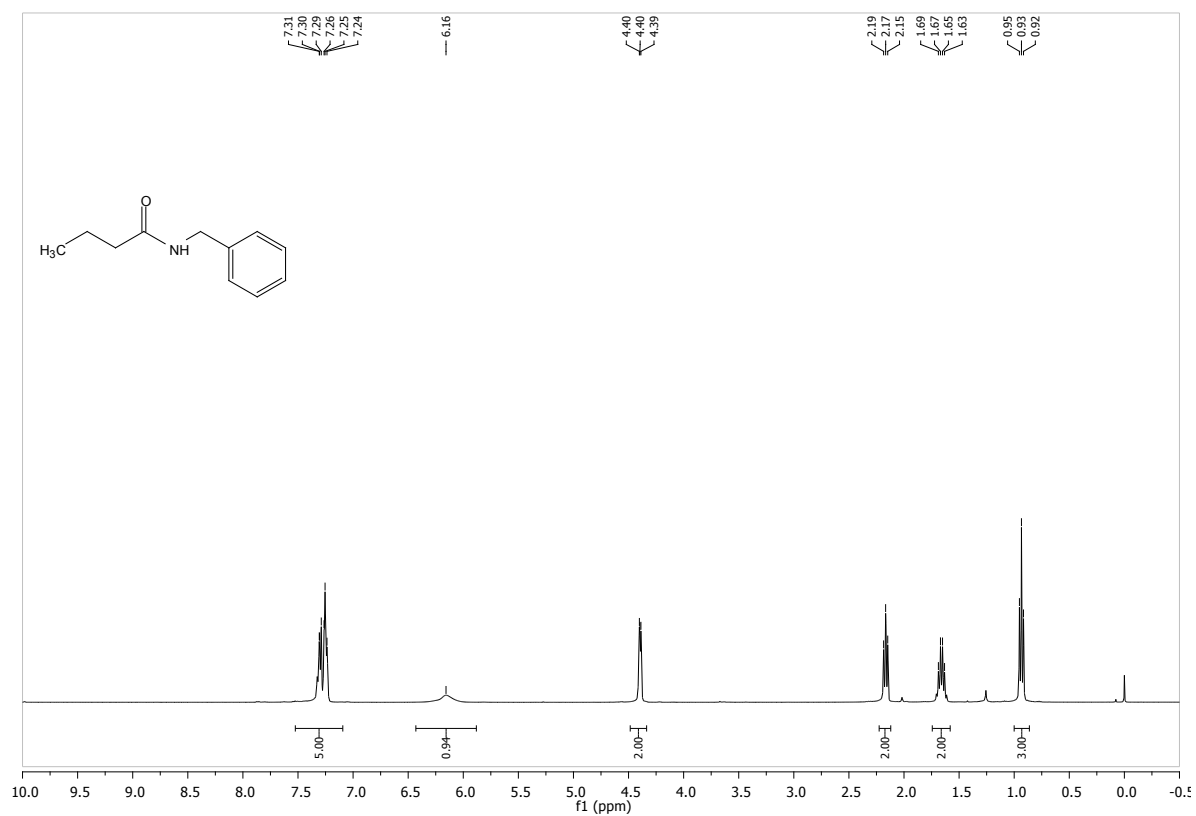


***N*-butanoylpyrrolidine (24)**

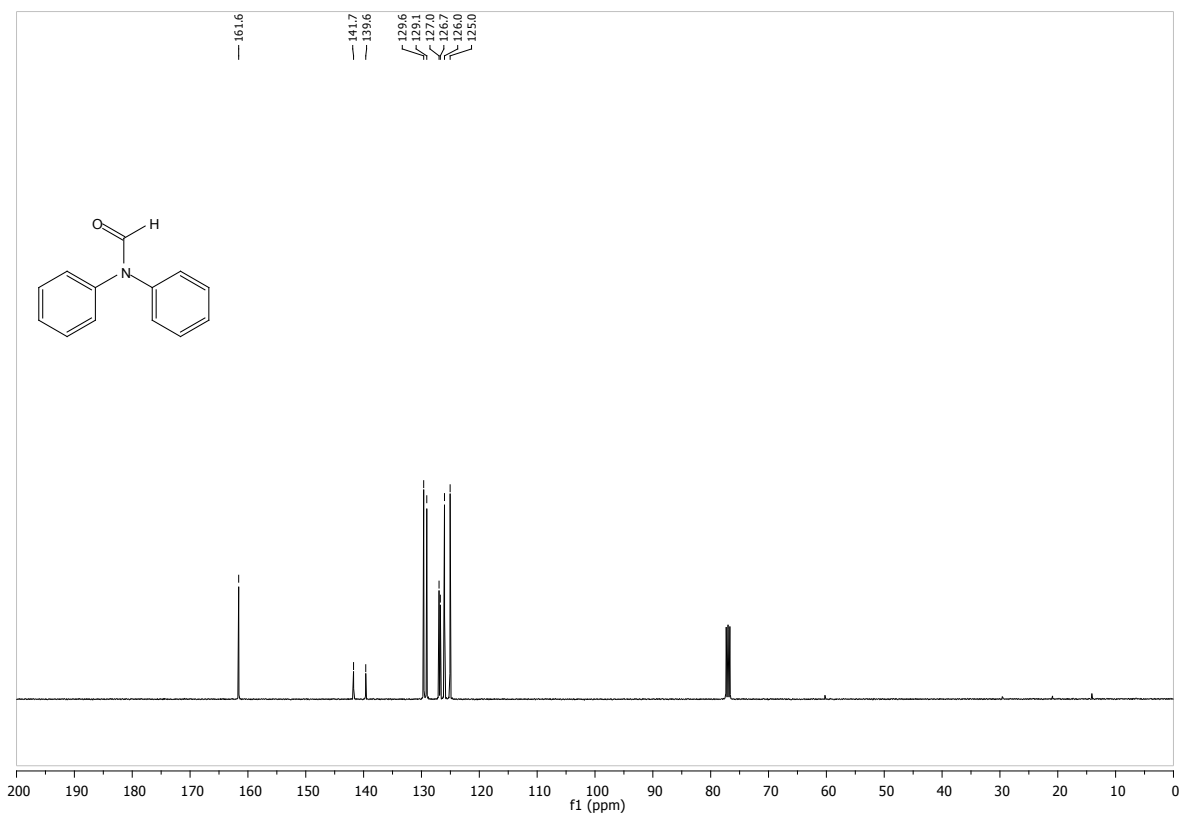
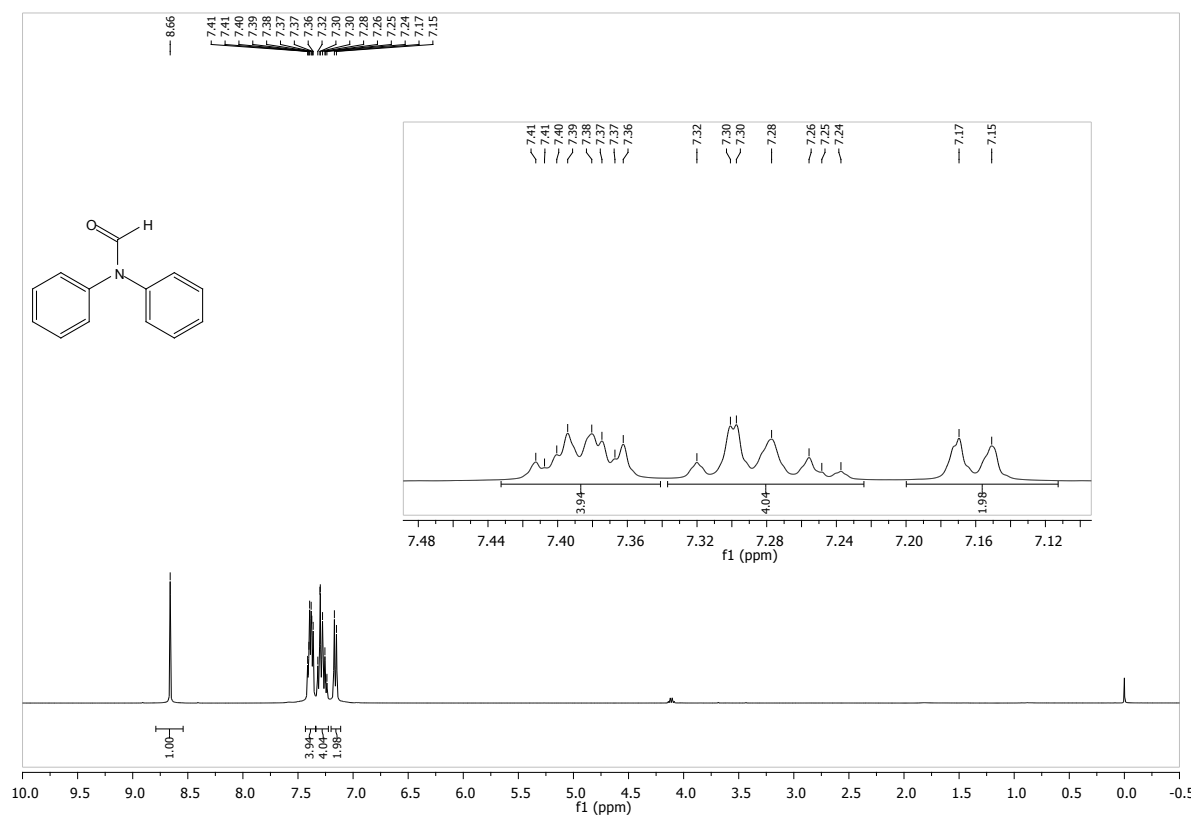




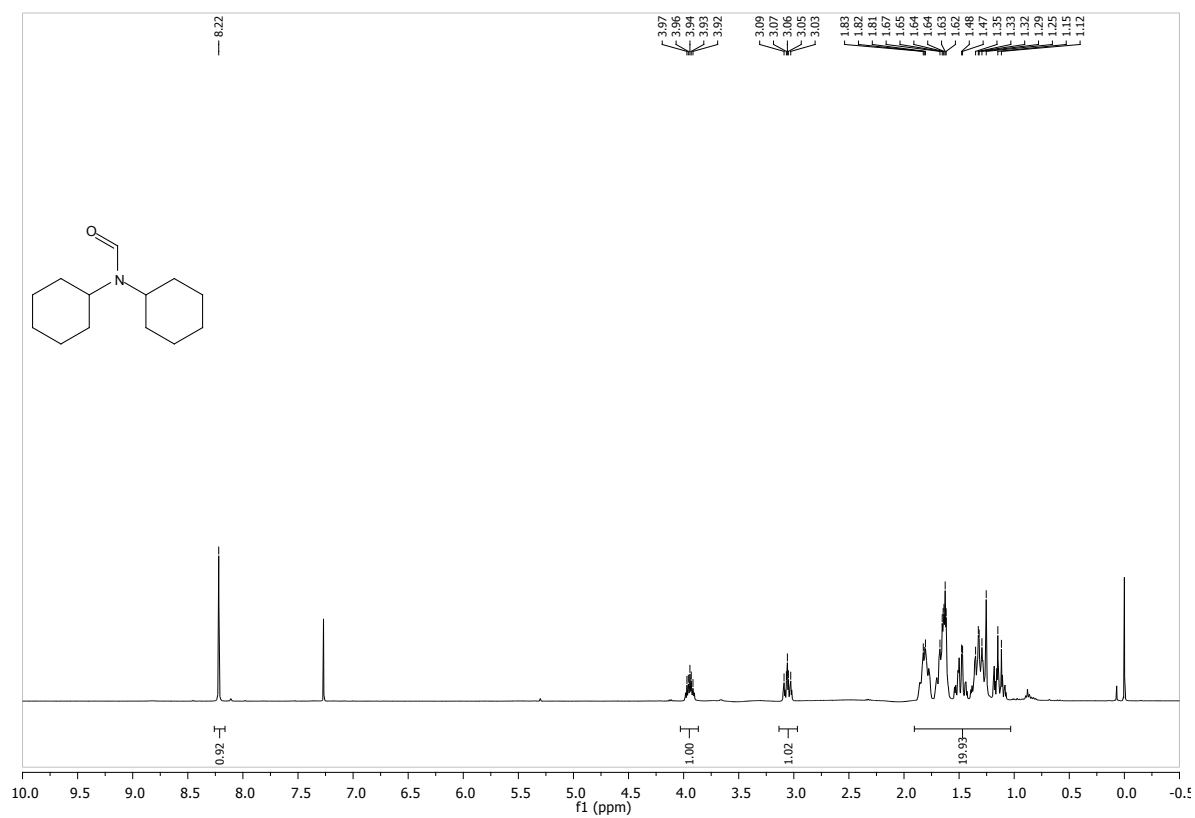
N-Phenyl-butylamide (25)



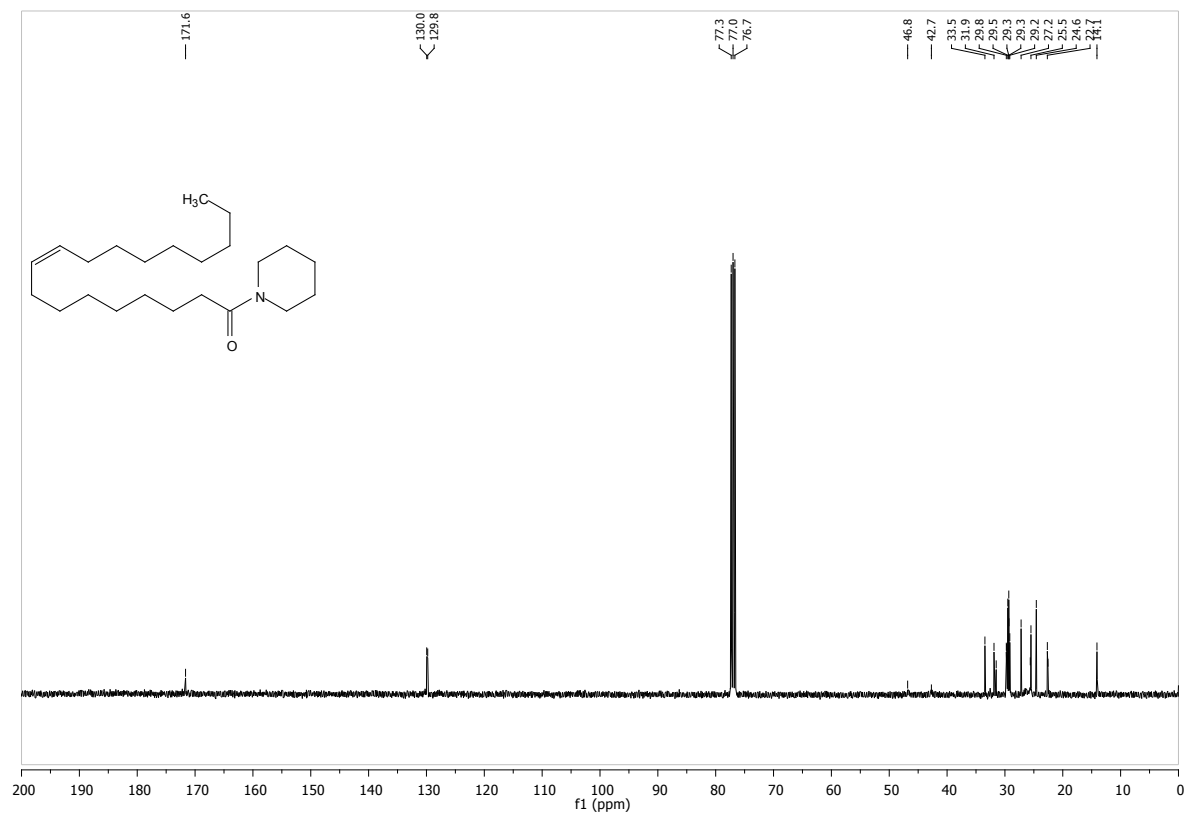
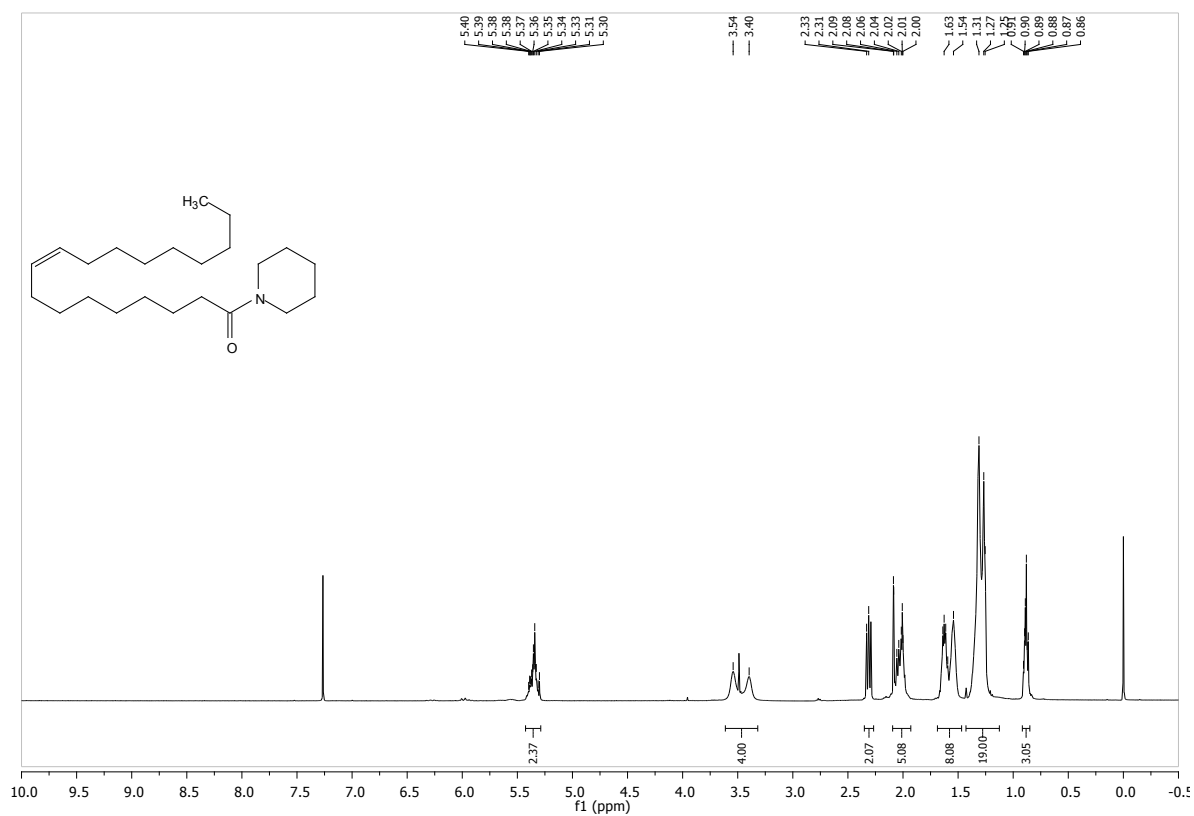
N,N-Diphenyl-formamide (26)



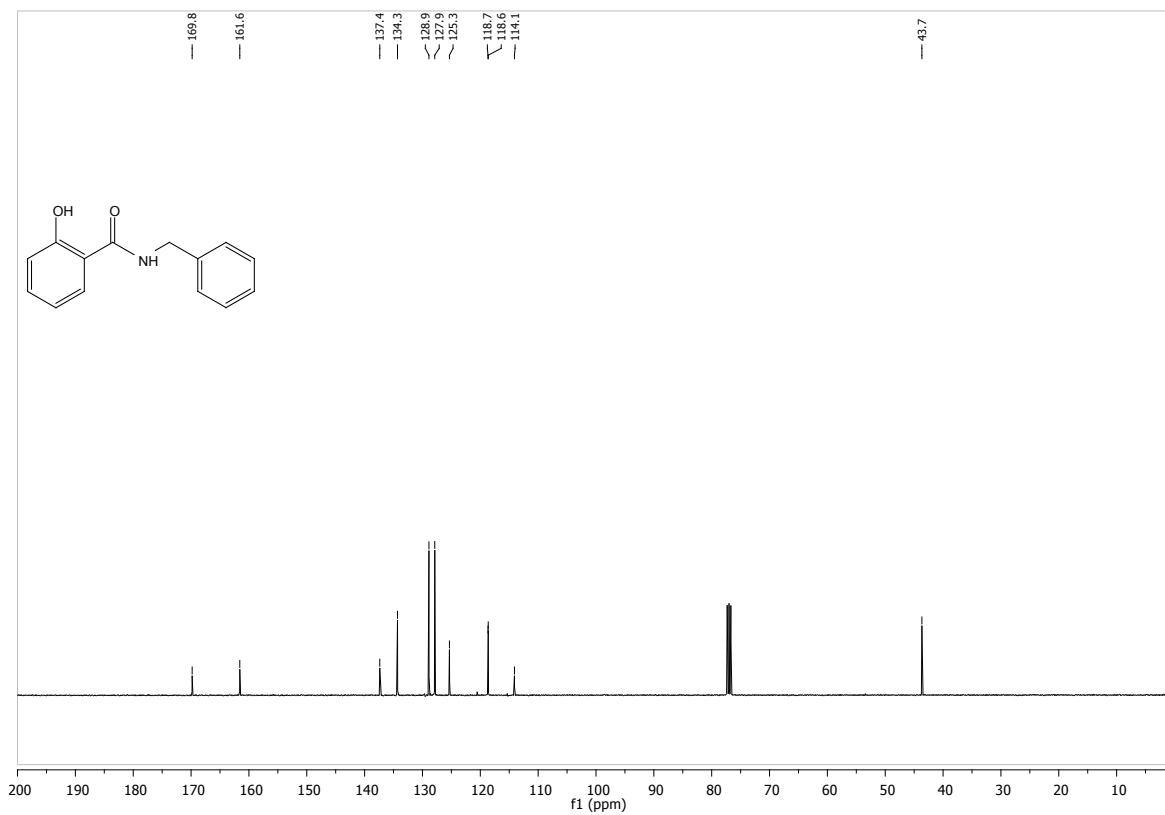
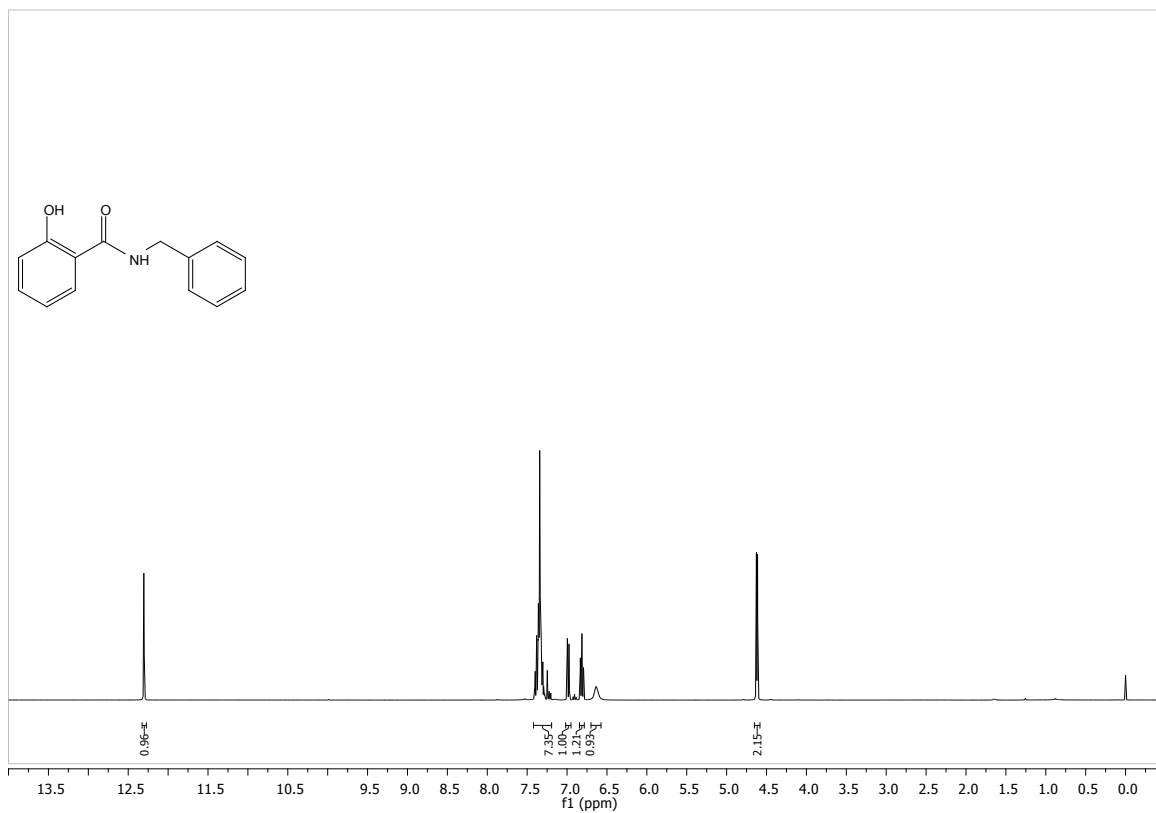
N,N-Dicyclohexyl-formamide (27)



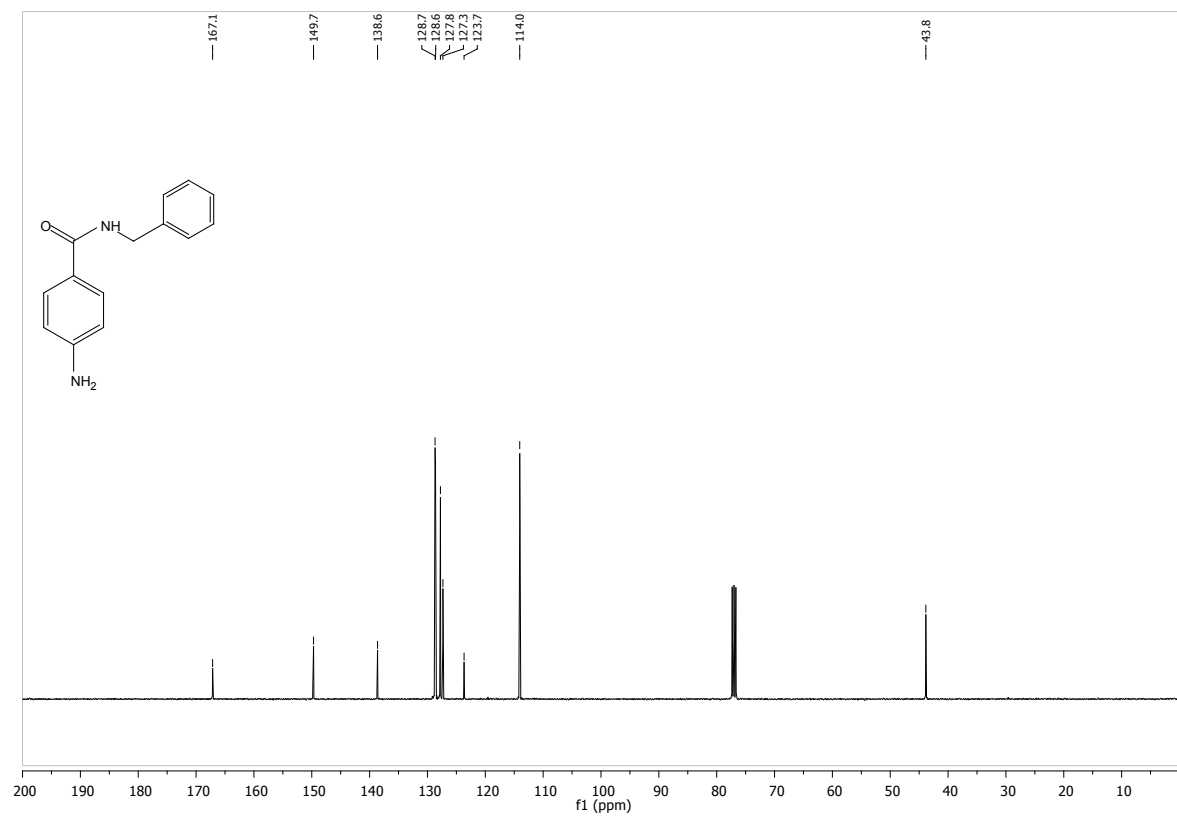
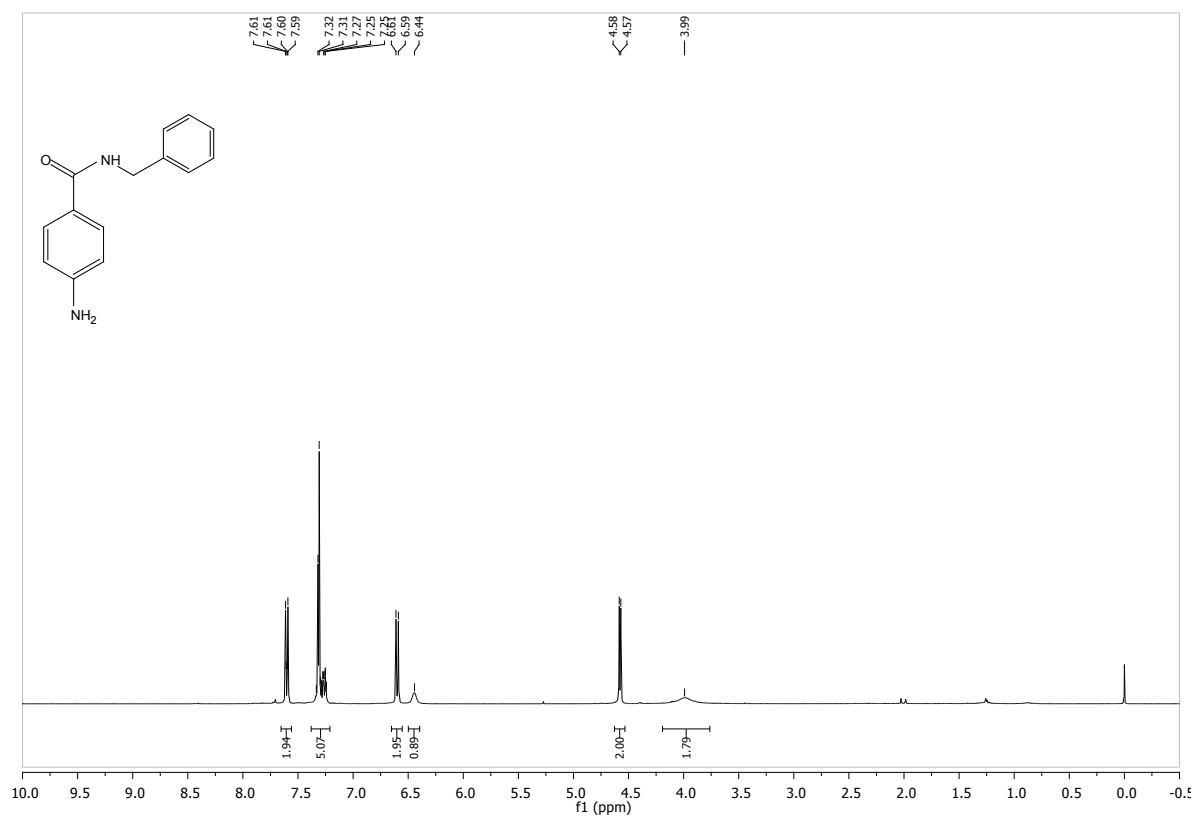
(Z)-1-(piperidin-1-yl)octadec-9-en-1-one (28)



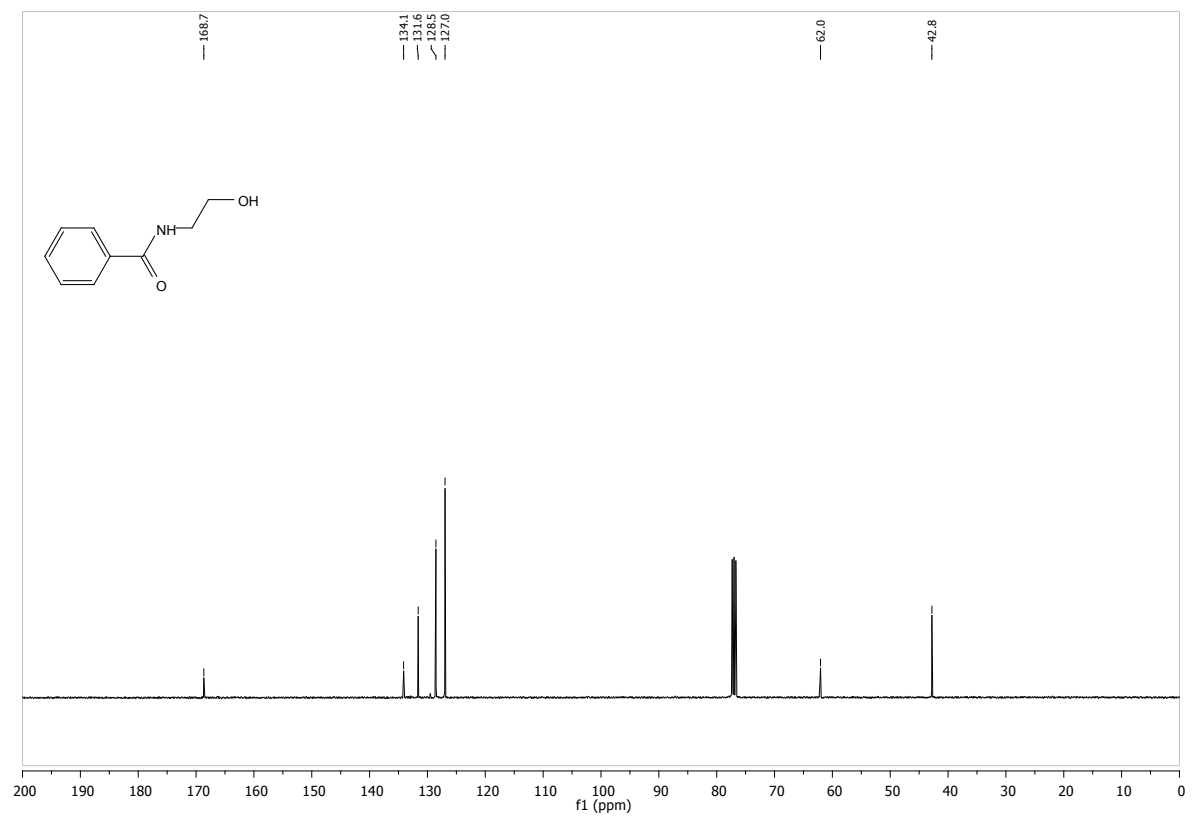
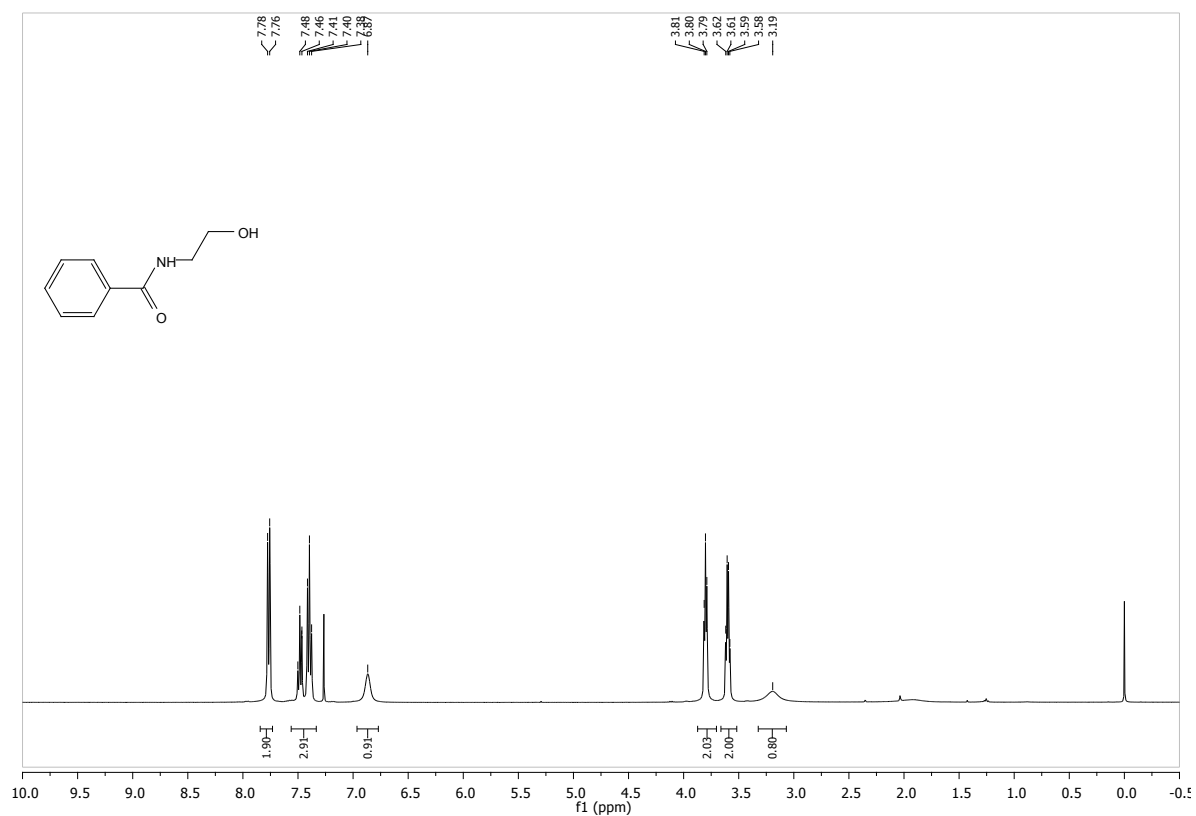
N-Benzyl-2-hydroxy-benzamide (32)



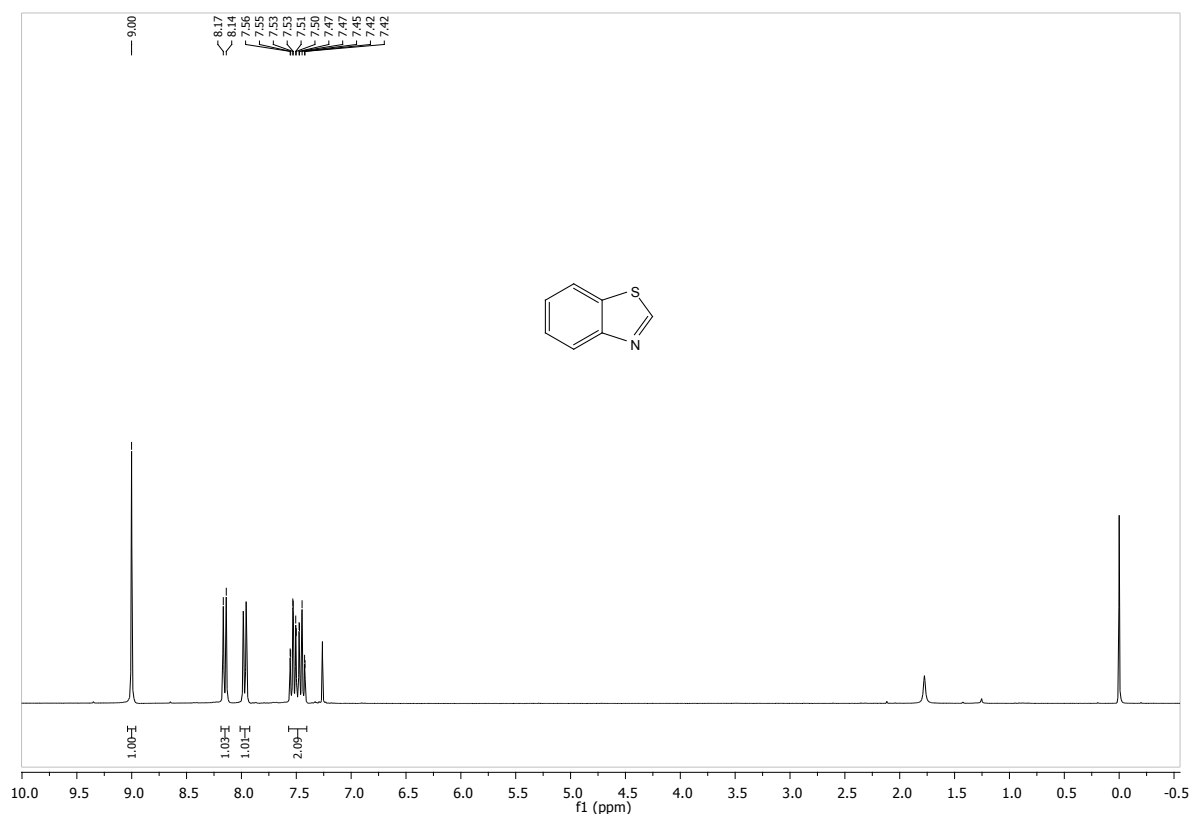
4-Amino-N-benzyl-benzamide (33)



N-(2-Hydroxy-ethyl)-benzamide (40)



Benzothiazole (43)



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