

**Condensation of a carboxylic acid with an amine using the Boc₂O/DMAP
system under solvent-free conditions.**

Mourad Boukachabia*^a Merabet Khelassi Mounia ^a and Olivier Riant ^b

a. *Ecocompatible Asymmetric Catalysis Laboratory. (LCAE) Badji Mokhtar Annaba-University. B.P 12, 23000 Annaba, Algeria.*

* E-mail : mouradboukachabia@gmail.com / mourad.boukachabia@univ-annaba.dz (Mourad Boukachabia).

b. *Institute of Condensed Matter and Nanosciences, Molecules Solids and Reactivity (IMCN/MOST), Université Catholique de Louvain, Bâtiment Lavoisier, Pl. Louis Pasteur, 1, bte 3. 1348, Louvain La Neuve, Belgium.*

Table of contents

Contents	Page
1- NMR data of intermediates, by-products and amides	<u>S2</u>
2-NMR spectra of intermediates, by-products and amides	<u>S8</u>
3- References	<u>S33</u>

1- NMR data of intermediates, by-products and amides:

✓ Benzoic (*tert*-butyl carbonic) anhydride (1a):

Uncolored oil. ¹H NMR (600 MHz, CDCl₃) δ 7.35-7.35 (m, 2H_{arm}), 7.29-7.26 (m, 2H_{arm}), 7.02 (t, 1H_{arm}, *J* = 7.3 Hz), 1.51 (s, 9H). ¹³C NMR (151 MHz, CDCl₃) δ 152.8 (Ph-C=O), 138.4 (O-C=O), 129, 123.1, 119.7, 118.5, 80.58 (O-C-*t*Bu), 29.4, 28.4.

✓ *tert*-butyl benzoate (*t*-BB):

Uncolored oil. ¹H NMR (600 MHz, DMSO-*d*₆) δ 7.46 (d, 2H_{arm}, *J* = 7.8 Hz), 7.24 (t, 2H_{arm}, *J* = 7.9 Hz), 6.94 (t, 1H_{arm}, *J* = 7.3 Hz), 1.47 (s, 9H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 153.2 (Ph-C=O), 140.0 (O-C=O), 129.1, 122.4, 118.5, 79.4(O-C-*t*Bu), 28.6.

✓ *tert*-butyl phenylcarbamate (1c):

White crystalline solid. *mp*: 130 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 9.34 (bs, 1H, -NH), 7.46 (d, 2H_{arm}, *J* = 7.9 Hz), 7.32 – 7.11 (m, 2H_{arm}), 6.95 (t, 2H_{arm}, *J* = 7.4 Hz), 1.47 (s, 9H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 153.2 (HN-C=O), 140, 129.1, 122.4, 118.5, 28.6.

✓ *N*-phenylbenzamide (1b)¹:

Pale white crystalline solid. *mp*: 157 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 10.26 (s, 1H, -NH), 7.95 (d, 2H_{arm}, *J* = 7.8 Hz), 7.79 (d, 2H_{arm}, *J* = 7.8 Hz), 7.59 (m, 1H_{arm}), 7.53 (t, 2H_{arm}, *J* = 7.6 Hz), 7.35 (t, 2H_{arm}, *J* = 7.8 Hz), 7.10 (q, 1H, *J* = 7.1 Hz). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.0 (HN-C=O), 140.2, 139.7, 135.5, 132.0, 129.3, 129.1, 128.9, 128.1, 124.1, 122.2, 120.8, 118.6, 28.65.

✓ *N*-(*p*-tolyl) benzamide (1d)¹:

White crystalline solid. *mp*: 158 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 9.08 (s, 1H, -NH), 7.49 (d, 2H_{arm}, *J* = 8.6 Hz), 7.24 (d, 2H_{arm}, *J* = 6.6 Hz), 7.10 (d, 3H_{arm}, *J* = 9.0 Hz), 6.67 (d, 2H_{arm}, *J* = 8.9 Hz), 1.48 (s, 3H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 153.5 (HN-C=O), 153.3, 153.02, 152.0, 145.7, 137.4, 131.5, 122.0, 120.4, 119.3, 115.5, 27.7.

✓ *N*-(4-(trifluoromethyl) phenyl) benzamide (1e)²:

White powder. *mp*: 204 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 9.34 (s, 1H, -NH), 7.57 (dd, 1H_{arm}, *J* = 8.5, 1.0 Hz), 7.46 (d, 3H_{arm}, *J* = 7.9 Hz), 7.41 (dd, 1H_{arm}, *J* = 8.4, 1.1 Hz), 7.24 (dd, 3H_{arm}, *J* = 8.5, 7.5 Hz), 6.95 (dd, 1H_{arm}, *J* = 7.8, 6.9 Hz). ¹³C NMR (151 MHz, DMSO-*d*₆) δ

167.3 (HN-C=O), 153.2, 140.0, 139.4, 136.4, 129.5, 129.3, 129.1, 128.5, 126.5, 124.0, 122.4, 119.6, 118.5.

✓ ***N*-(4-nitrophenyl) benzamide (1f)¹:**

Deep yellow solid. *mp*: 195 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 10.78 (s, 1H), 8.24 (d, *J* = 8.8 Hz, 2H), 8.18 (d, *J* = 8.7 Hz, 3H), 8.03 (d, *J* = 8.8 Hz, 2H), 7.94 (d, *J* = 7.6 Hz, 2H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.86 (HN-C=O), 146.27, 146.05, 142.05, 132.74, 129.08, 128.47, 125.69, 118.52.

✓ ***N*-(*o*-tolyl) benzamide (1g)¹:**

Beige solid. *mp*: 139 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 9.84 (s, 1H), 7.94 (d, *J* = 8.5 Hz, 2H), 7.63 – 7.44 (m, 3H), 7.38 – 7.13 (m, 4H), 3.30 (s, 3H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 165.8 (HN-C=O), 136.9, 135.07, 134.2, 132.07, 130.8, 128.9, 128.1, 127.1, 126.5, 18.4.

✓ ***N*-(*m*-tolyl) benzamide (1h)¹:**

White solid. *mp*: 141 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 9.87 (s, 1H), 7.93 (s, 2H), 7.63 – 7.41 (m, 3H), 7.35 – 7.04 (m, 4H), 3.30 (s, 2H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 165.81 (HN-C=O), 136.96, 135.07, 134.27, 132.06, 130.85, 128.96, 128.16, 127.17, 126.54, 18.44.

✓ ***N*-(3-methoxyphenyl) benzamide (1i)¹:**

Beige solid. *mp*: 110 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 10.21 (s, 1H), 7.91 (d, *J* = 8.3 Hz, 2H), 7.65 – 7.40 (m, 4H), 7.40 – 7.13 (m, 2H), 6.65 (d, *J* = 5.6 Hz, 1H), 3.74 (s, 3H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.12 (HN-C=O), 135.51, 132.12, 129.92, 128.93, 128.18, 109.68, 106.54, 55.54.

✓ ***N*-(3-acetylphenyl) benzamide (1j)³:**

Beige crystalline solid. *mp*: 102 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 10.42 (s, 1H), 8.34 (s, 1H), 8.04 (d, *J* = 9.3 Hz, 1H), 7.97 – 7.93 (m, 2H), 7.68 (d, *J* = 7.7 Hz, 1H), 7.60 – 7.55 (m, 1H), 7.54 – 7.45 (m, 3H), 2.55 (s, 3H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.27 (HN-C=O), 140.11, 137.79, 135.14, 132.32, 129.60, 128.99, 128.24, 125.32, 124.20, 120.19, 27.31.

✓ ***N*-benzyl benzamide (1k)³:**

White crystalline solid. *mp*: 104 °C. ¹H NMR (300 MHz, CDCl₃) δ= 7.80 (dd, *J* = 8.3 and 1.2 Hz, 2H_{arm}), 7.45-7.50 (m, 1H_{arm}), 7.35-7.41 (m, 2H_{arm}), 7.27-7.33 (m, 5H_{arm}), 6.89 (bs, 1H, -NH), 4.59 (d, *J* = 5.7 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃) δ= 167.5, 138.3, 134.4, 131.5, 128.7, 128.5, 127.8, 127.5, 127.1, 44.0.

✓ **(S)-N-(1-phenylethyl) benzamide (1l)³:**

Pale yellow crystalline solid. *mp*: 119 °C. ¹H NMR (300 MHz, CDCl₃) δ = 7.75-7.95 (m, 2H_{arm}); 7.3-7.5 (m, 8H_{arm}); 6.4-6.5 (d, *J* = 6.94 Hz, 1H, -NH); 5.26-5.4 (m, 1H); 1.6-1.7 (d, *J* = 6.9 Hz, 3H).

✓ **N-phenyl-2-(3-(trifluoromethyl) phenyl) acetamide (1m)⁴:**

White powder. *mp*: 137 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 8.24 (d, 2H_{arm}, *J* = 8.8 Hz), 8.05 (d, 2H_{arm}, *J* = 8.9 Hz), 7.96 (d, 2H_{arm}, *J* = 8.2 Hz), 7.65 – 7.54 (m, 3H_{arm}). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.8 (HN-C=O), 146.4, 146.0, 142.9, 141.9, 134.7, 132.6, 129.0, 128.4, 126.9, 125.6, 125.4, 125.3, 120.3, 118.4, 112.8, 55.4.

✓ **N-tosylbenzamide (1n)⁵:**

Yellow oil. ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.50 (s, 1H, -NH), 7.88 (d, 2H_{arm}, *J* = 8.3 Hz), 7.85 (dd, 2H_{arm}, *J* = 8.4, 1.2 Hz), 7.64-7.58 (m, 1H_{arm}), 7.50-7.46 (m, 2H_{arm}), 7.44 (dd, 2H_{arm}, *J* = 8.5, 0.6 Hz), 2.39 (s, 3H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 165.9 (HN-C=O), 144.7, 137.1, 133.7, 132.0, 130.0, 129.8, 129.1, 128.9, 128.2, 126.1, 21.6.

✓ **N-phenyl-2-naphthamide (1o)¹:**

Beige crystalline solid. *mp*: 166 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 10.43 (s, 1H, -NH), 8.56 (s, 1H_{arm}), 8.07-8.03 (m, 4H_{arm}), 7.83 (d, 2H_{arm}, *J* = 7.7 Hz), 7.62-7.57 (m, 2H_{arm}), 7.35 (t, 2H_{arm}, *J* = 7.8 Hz), 7.09 (t, 1H_{arm}, *J* = 7.4 Hz). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.1 (HN-C=O), 140.3, 139.8, 134.80, 132.8, 132.6, 129.4, 129.3, 129.1, 128.5, 128.3, 128.2, 127.3, 125.0, 124.2, 122.5, 122.2, 120.1, 118.7, 28.6.

✓ **2-phenoxy-N-phenylpropanamide (1p)⁶:**

Beige powder. *mp*: 120 °C. ¹H NMR (300 MHz, CDCl₃) δ 8.13 (bs, 1H, -NH), 7.59 – 7.41 (m, 2H_{arm}), 7.25 (t, *J* = 7.8 Hz, 4H_{arm}), 6.99-6.90 (m, 4H_{arm}), 4.71 (q, *J* = 6.8 Hz, 1H), 1.59 (d, *J* = 6.8 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 170.2 (HN-C=O), 156.7, 137.0, 129.9, 129.0, 124.7, 122.4, 119.9, 115.7, 75.5, 18.7.

✓ **N-(pentan-2-yl) benzamide (1q)³:**

Uncolored crystalline oil. ¹H NMR (250 MHz, CDCl₃) δ = 7.75-7.9 (m, 2H_{arm}), 7.3 -7.6 (m, 3H_{arm}), 6.1 (d, *J* = 7.19 Hz, 1H), 4.1 – 4.3 (m, 1H), 1.3 – 1.7 (m, 4H), 1.25 (d, *J* = 6.58 Hz, 3H), 0.92 (t, *J* = 7.08 Hz, 3H).

✓ ***N*-(2-methylbutyl) benzamide (1r)³:**

Uncolored oil. ¹H NMR (250 MHz, CDCl₃) δ 7.75-7.95 (m, 2H_{arm}), 7.3-7.5 (m, 8H_{arm}), 6.5 (d, *J* = 6.94 Hz, 1H), 5.26-5.4 (m, 1H), 1.6 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (62.9 MHz, CDCl₃) δ=167.7 (HN-C=O), 134.8, 131.2, 128.4, 126.8, 45.6, 34.9, 27.0, 17.1, 11.3.

✓ ***N*-(*tert*-butyl) benzamide (1s)⁷:**

White crystalline solid. *mp*: 129 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 8.13 (bs, 1H, -NH), 7.96 (dd, 1H_{arm}, *J* = 5.2, 3.3 Hz), 7.79 (dd, 1H_{arm}, *J* = 8.5, 0.9 Hz), 7.45 (d, 1H_{arm}, *J* = 7.9 Hz), 7.27 – 7.15 (m, 1H_{arm}), 7.02 – 6.89 (m, 1H_{arm}), 1.47 (s, 9H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.0 (HN-C=O), 153.2, 140.0, 139.7, 135.5, 132.0, 129.1, 128.9, 128.1, 124.1, 122.5, 120.8, 118.5, 79.4, 28.6.

✓ ***N*-(thiophen-2-ylmethyl) benzamide (1u)³:**

Beige crystalline solid. *mp*: 120 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 9.11 (t, *J* = 5.9 Hz, 1H), 7.83 (d, *J* = 7.0 Hz, 2H), 7.56 – 7.44 (m, 2H), 7.44 – 7.31 (m, 2H), 7.05 – 6.88 (m, 2H), 4.59 (d, *J* = 6.8 Hz, 2H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.55 (HN-C=O), 143.26, 134.65, 131.88, 128.88, 127.78, 127.17, 125.90, 125.49, 38.29.

✓ ***N*-(furan-2-ylmethyl) benzamide (1v)³:**

Orange crystalline solid. *mp*: 93 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 8.94 (t, *J* = 5.8 Hz, 1H), 7.87 – 7.81 (m, 2H), 7.52 (d, *J* = 30.2 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 2H), 6.36 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.23 (d, *J* = 3.3 Hz, 1H), 4.43 (d, *J* = 5.7 Hz, 2H). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 166.60 (HN-C=O), 152.99, 142.54, 134.65, 128.84, 127.82, 111.01, 36.57.

✓ ***N*-phenyldecanamide (2a)⁸:**

White crystalline solid. *mp*: 136 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 7.59 (d, 1H_{arm}, *J* = 7.7 Hz), 7.51 – 7.42 (m, 1H_{arm}), 7.31 – 7.22 (m, 2H_{arm}), 6.98 (dt, 1H_{arm}, *J* = 27.5, 7.4 Hz), 3.36 (s, 1H, -NH), 2.28 (t, 2H, *J* = 7.4 Hz), 1.60 – 1.50 (m, 1H), 1.23 (s, 13H), 0.85 (t, 3H, *J* = 7.0 Hz). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 171.7 (HN-C=O), 153.0, 140.2, 139.9, 129.2, 129.1, 123.3, 122.2, 119.4, 118.6, 36.9, 31.8, 29.6, 29.5, 29.4, 29.3, 29.2, 25.6, 22.6, 14.4.

✓ ***N*-benzyldecanamide (2b)⁸:**

Pale yellow solid. *mp*: 65 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 8.27 (bs, 1H, -NH), 7.33 – 7.28-7.25 (m, 2H_{arm}), 7.21 – 7.18 (m, 2H_{arm}), 4.21 (d, 2H, *J* = 6.0 Hz), 2.08 (t, 2H, *J* = 7.4 Hz), 1.48-1.46 (m, 2H), 1.23 (s, 12H), 0.82 (t, 3H, *J* = 6.9 Hz). ¹³C NMR (151 MHz, DMSO-

***d*₆**) δ 172.6 (HN-C=O), 140.3, 128.7, 127.8, 127.6, 127.3, 127.1, 60.0, 35.8, 31.8, 29.4, 29.3, 29.2, 29.1, 28.9, 28.1, 27.2, 25.8, 23.5, 22.6, 14.5.

✓ ***N*-benzyltetradecanamide (2c)⁹:**

Beige solid. *mp*: 78 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 7.41 (t, 1H, -NH, *J* = 6.0 Hz), 7.30 (dd, 2H_{arm}, *J* = 13.2, 5.8 Hz), 7.28 – 7.18 (m, 3H_{arm}), 4.21 (d, 2H, *J* = 6.2 Hz), 2.16-2.13 (m, 1H), 1.99 (d, 1H, *J* = 5.3 Hz), 1.49 – 1.41 (m, 2H), 1.41 – 1.39 (m, 10H), 1.32 – 1.25 (m, 10H), 0.86 (t, 3H, *J* = 6.8 Hz). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 172.6 (HN-C=O), 156.3, 140.7, 140.3, 130.0, 128.6, 127.6, 127.4, 127.1, 127.0, 78.1, 35.8, 35.3, 31.8, 29.7, 29.6, 29.4, 29.3, 29.2, 29.1, 29.0, 28.9, 28.7, 28.5, 28.2, 27.2, 27.1, 25.9, 25.1, 22.6, 14.4.

✓ ***N*-oleoylethanolamide¹⁰:**

White wax. ¹H NMR (600 MHz, DMSO-*d*₆) δ 5.30 (d, 2H, *J* = 5.6 Hz), 3.37 (t, 2H, *J* = 6.2 Hz), 3.09 (q, 2H, *J* = 6.1 Hz), 2.04 (t, 2H, *J* = 7.5 Hz), 1.97 (dd, 4H, *J* = 12.6, 6.7 Hz), 1.46 (t, 2H, *J* = 7.0 Hz), 1.38 – 1.37 (m, 2H), 1.28 – 1.15 (m, 20H), 0.84 (t, 3H, *J* = 7.0 Hz). ¹³C NMR (75 MHz, CDCl₃) δ 172.6 (HN-C=O), 130.1 (HC=CH), 60.5 (H₂C-OH), 41.9 (H₂C-NH), 35.8, 35.2, 32.5, 31.8, 31.4, 29.7, 29.6, 29.4, 29.3, 29.2, 29.1, 28.7, 28.1, 27.9, 27.8, 27.1, 25.8, 25.7, 25.1, 22.6, 22.5, 14.4, 14.3, 13.9.

✓ **2-(6-methoxynaphthalen-2-yl)-*N*-(*p*-tolyl) propanamide (3a)¹¹:**

White solid. *mp*: 140 °C. ¹H NMR (300 MHz, CDCl₃) δ 7.77 (t, *J* = 7.6 Hz, 3H_{arm}), 7.45 (dd, *J* = 8.5, 1.7 Hz, 1H_{arm}), 7.31 (s, 4H_{arm}), 7.21-7.16 (m, 2H_{arm}), 7.18 (dd, *J* = 10.0, 5.6 Hz, 1H), 7.07 (d, *J* = 8.3 Hz, 2H_{arm}), 3.95 (s, 3H), 3.86 (q, *J* = 7.1 Hz, 1H), 2.29 (s, 3H), 1.69 (d, *J* = 7.1 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 172.6 (HN-C=O), 157.2, 133.8, 129.3, 129.2, 127.8, 126.3, 126.2, 119.6, 119.3, 105.6, 55.3, 48.0, 20.8.

✓ ***N*-(4-chlorophenyl)-2-(6-methoxynaphthalen-2-yl) propanamide (3b)¹¹:**

White solid. *mp*: 160 °C. ¹H NMR (300 MHz, CDCl₃) δ 7.71 (t, *J* = 8.6 Hz, 3H_{arm}), 7.37 (dd, *J* = 8.4, 1.8 Hz, 3H_{arm}), 7.18-7.05 (m, 4H_{arm}), 6.97 (s, 1H, -NH), 3.89 (s, 3H), 3.80 (q, *J* = 7.1 Hz, 1H), 1.63 (d, *J* = 7.1 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 178.1 (HN-C=O), 135.6, 134.7, 131.8, 129.2, 128.0, 126.4, 126.0, 121.1, 119.4, 105.6, 55.3, 48.1, 18.2.

✓ **2-(2-fluoro-[1,1'-biphenyl]-4-yl)-*N*-(4-methoxyphenyl) propanamide (3c)¹²:**

White crystalline solid. *mp*: 153 °C. ¹H NMR (300 MHz, CDCl₃) δ 7.53 – 7.49 (m, 2H_{arm}), 7.41 – 7.29 (m, 5H_{arm}), 7.18 – 7.11 (m, 2H_{arm}), 7.05 (bs, 1H, -NH), 6.78 – 6.75 (m, 2H), 3.71

(s, 3H), 3.65 (q, $J = 7.2$ Hz, 1H), 1.56 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 171.1 (HN-C=O), 156.2, 142.1, 135.6, 135.3, 131.2, 130.7, 128.9, 128.5, 127.8, 123.6, 121.7, 115.5, 114.1, 77.4, 77.0, 76.6, 55.4, 47.4, 18.6.

✓ ***N*-benzyl-2-(2-fluoro-[1,1'-biphenyl]-4-yl) propanamide (3d)¹³:**

White crystalline solid. *mp*: 126 °C. ^1H NMR (300 MHz, CDCl_3) δ 7.48 – 7.20 (m, 10H_{arm}), 7.14 – 7.04 (m, 4H_{arm}), 5.62 (bs, 1H, -NH), 4.44 – 4.30 (m, 2H), 3.54 (q, $J = 7.1$ Hz, 1H), 1.52 (d, $J = 7.1$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 173.3 (HN-C=O), 142.6, 138.1, 135.3, 131.0, 128.9, 128.7, 128.4, 127.7, 127.6, 127.5, 123.6, 115.4, 115.1, 46.7, 43.7, 32.5, 18.6.

✓ **2-(3-benzoylphenyl)-*N*-phenylpropanamide (3e)⁶:**

White powder. *mp*: 92 °C. ^1H NMR (300 MHz, CDCl_3) δ 7.82–7.06 (m, 14H_{arm}+ -NH), 3.78(q, $J = 7.1$ Hz, 1H), 1.60 (d, $J = 7.1$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 196.6 (C=O Benzoyl-), 171.7(HN-C=O), 153.8, 141.8, 141.6, 138.1, 137.8, 137.3, 132.6, 132.5, 131.4, 131.3, 130.08, 130.03, 129.3, 129.2, 129.1, 128.9, 128.8, 128.7, 128.3, 128.3, 124.3, 119.8, 50.1, 47.9, 45.2, 32.7, 32.5, 31.1, 30.6, 26.1, 26.1, 25.4, 25.2, 24.7, 20.5, 18.8.

✓ **2-(3-benzoylphenyl)-*N*-(4-methoxyphenyl) propanamide (3f)¹³:**

White powder. *mp*: 128 °C. ^1H NMR (300 MHz, CDCl_3) δ 7.80 – 7.66 (m, 2H_{arm} + -NH), 7.66 – 7.48 (m, 3H_{arm}), 7.47 – 7.33 (m, 3H_{arm}), 7.34 – 7.23 (m, 3H_{arm}), 6.72 (d, 2H_{arm}, $J = 9.0$ Hz, 1H), 3.93 – 3.44 (s+q, 4H), 1.51 (d, $J = 7.1$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 196.6 (C=O Benzoyl-), 171.5 (HN-C=O), 156.4, 141.7, 138.0, 137.3, 132.6, 131.5, 130.8, 130.0, 129.3, 129.2, 128.9, 128.3, 121.7, 114.0, 55.4, 47.7, 18.8.

✓ **2-(4-Isobutylphenyl)-*N*-phenylpropanamide (3g)⁶:**

White powder. *mp*: 151 °C. ^1H NMR (300 MHz, CDCl_3) δ 7.34 (d, $J = 7.7$ Hz, 2H_{arm}), 7.22–7.15 (m, 3H_{arm}+ -NH), 7.08–6.95 (m, 4H_{arm}), 3.64 (q, $J = 7.2$ Hz, 1H), 2.39 (d, $J = 7.2$ Hz, 2H), 1.83–1.72 (m, 1H), 1.51(d, $J = 7.2$ Hz, 3H), 0.81(d, $J = 6.6$ Hz, 6H). ^{13}C NMR (75 MHz, CDCl_3) δ 172.6 (HN-C=O), 141.1, 138.0, 137.9, 129.8, 128.9, 127.4, 124.1, 119.6, 47.7, 45.0, 30.2, 22.4, 18.5.

✓ **2-(4-Isobutylphenyl)-*N*-(4-(trifluoromethyl)phenyl) propanamide (3e)⁶:**

White crystalline solid. *mp*: 140 °C. ^1H NMR (300 MHz, CDCl_3) δ 7.45 (q, $J = 9$ Hz, 4H_{arm}), 7.32 (bs, 1H, -NH), 7.19–7.03 (m, 4 H_{arm}), 3.65 (q, $J = 7.1$ Hz, 1H), 2.40 (d, $J = 7.2$ Hz, 2H), 1.84–1.75 (m, 1H), 1.53–1.50 (d, $J = 7.2$ Hz, 3H), 0.83 (d, $J = 6.6$ Hz, 6H). ^{13}C NMR (75

MHz, CDCl₃) δ 171.9 (HN-C=O), 140.9, 139.9, 136.5, 128.9, 128.2, 126.3, 126, 125.1, 125, 121.2, 118.1, 124.6, 59.6, 46.7, 43.9, 29.1, 21.3, 17.4, 13.

✓ **(S)-N-phenylpyrrolidine-2-carboxamide (4a)¹⁴:**

Pale yellow solid. *mp*: 72 °C. **¹H NMR (250 MHz, CDCl₃)** δ 9.8 (s, 1H - NH), 7.65 (m, 5H_{arm}), 3.9 (t, 1H), 3.05 (m, 2H), 2.9 (s, 1H), 2.2 (m, 2H), 1.8 (m, 2H). **¹³C NMR (90.56 MHz, CDCl₃)** δ 173.4 (HN-C=O), 137.8, 128.9, 119.4, 60, 47.3, 30.7, 26.5.

✓ **(S)-N-(p-tolyl) pyrrolidine-2-carboxamide (4b)¹⁴:**

White crystalline solid. *mp*: 103 °C. **¹H NMR (360 MHz, CDCl₃)** δ 9.6 (s, 1H), 7.5 (dd, 4H_{arm}), 3.9 (t, 1H), 2.99 (m, 2H), 2.37 (s, 3H), 2.25 (m, 2H), 2.22 (m, 1H), 1.8 (m, 2H). **¹³C NMR (90.56 MHz, CDCl₃)** δ 173.0 (HN-C=O), 135.3, 133.4, 129, 129.2, 61, 47.3, 30.7, 26.2, 20.8.

✓ **(S)-N-(4-(trifluoromethyl) phenyl) pyrrolidine-2-carboxamide (4c)¹⁵:**

Pale yellow solid. *mp*: 110 °C. **¹H NMR (250 MHz, CDCl₃)** δ 10.01 (s, 1H), 7.65 (dd, 4H_{arm}), 3.9 (t, 1H), 2.3 (m, 3H), 2.07 (m, 2H), 1.8 (m, 2H). **¹³C NMR (75 MHz, CDCl₃)** δ 173.8 (HN-C=O), 140.7, 126, 118.7, 60.9, 47.2, 30.6, 26.2.

✓ **(S)-N-benzylpyrrolidine-2-carboxamide (4d)¹⁴:**

Pale yellow solid. *mp*: 119 °C. **¹H NMR (300 MHz, CDCl₃)** δ 8.44 (s, 1H), 7.51 (m, 5H_{arm}), 4.4 (m, 2H), 4.25 (m, 1H), 3.1 (m, 2H), 2.2 (m, 1H), 1.9 (m, 4H). **¹³C NMR (75 MHz, CDCl₃)** δ 171.3 (HN-C=O), 138.0, 128.6, 127.5, 127.4, 59.9, 46.6, 43.4, 30.4.

✓ **(S)-N-((R)-1-phenylethyl) pyrrolidine-2-carboxamide (4e)¹⁴:**

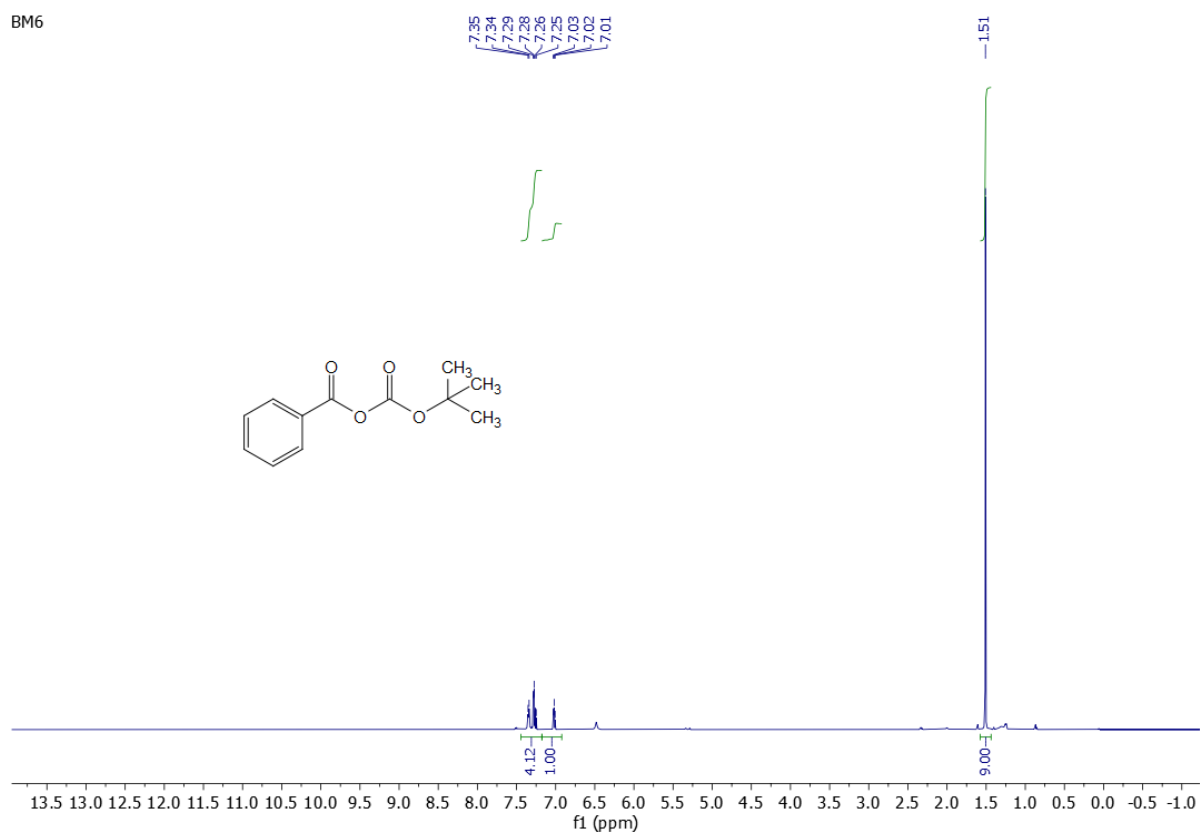
White solid. *mp*: 125 °C. **¹H NMR (360 MHz, CDCl₃)** δ 8.4 (s, 1H), 7.65 (m, 5H_{arm}), 5.05 (t, 1H), 3.69 (m, 1H), 3.3 (s, 1H), 2.8 (m, 2H), 1.7 (m, 2H), 1.6 (m, 2H), 1.5 (t, 3H). **¹³C NMR (75 MHz, CDCl₃)** δ : 173.6 (HN-C=O), 128.6, 128.5, 127.0, 126, 125.8, 60, 47.3, 30.7, 26.0, 24.9, 21.2.

✓ **(S)-N-(tert-butyl) pyrrolidine-2-carboxamide (4f)¹⁶:**

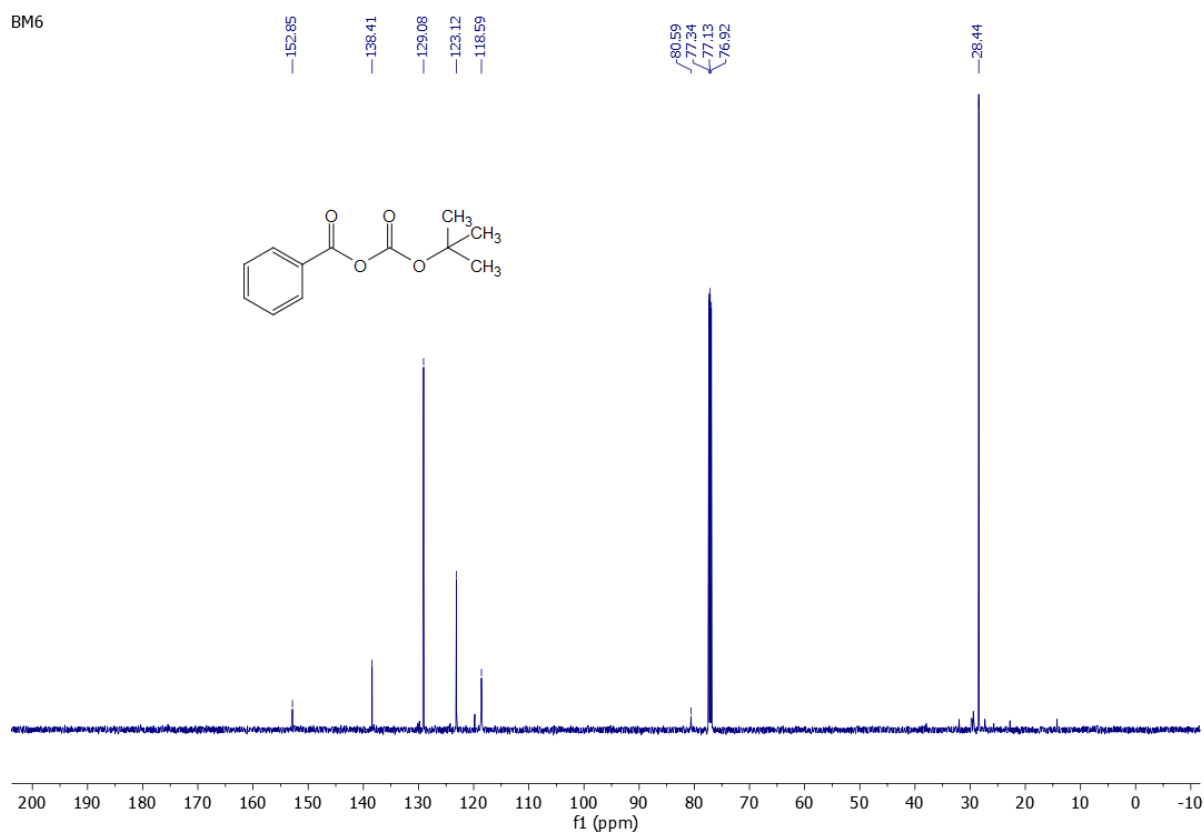
White solid. *mp*: 95 °C. **¹H NMR (250 MHz, CDCl₃)** δ 3.9 (t, 1H), 3.05 (m, 2H), 2.35 (m, 2H), 2.26 (m, 3H), 1.3 (s, 9H). **¹³C NMR (62.9 MHz, CDCl₃)** δ 173.6 (HN-C=O), 60.0, 50.1, 47.0, 30.5, 28.6, 26.0.

2- NMR spectra of intermediates, by-products and amides:

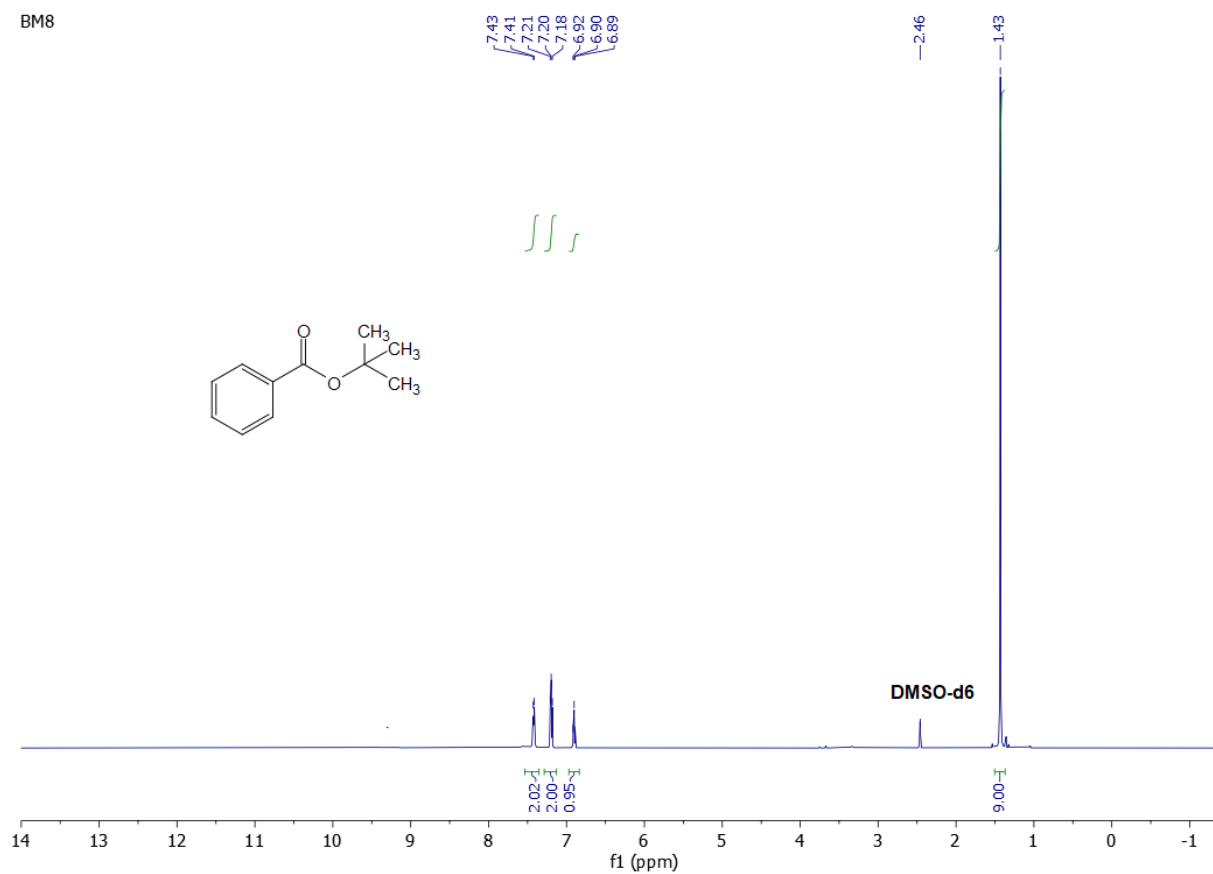
BM6



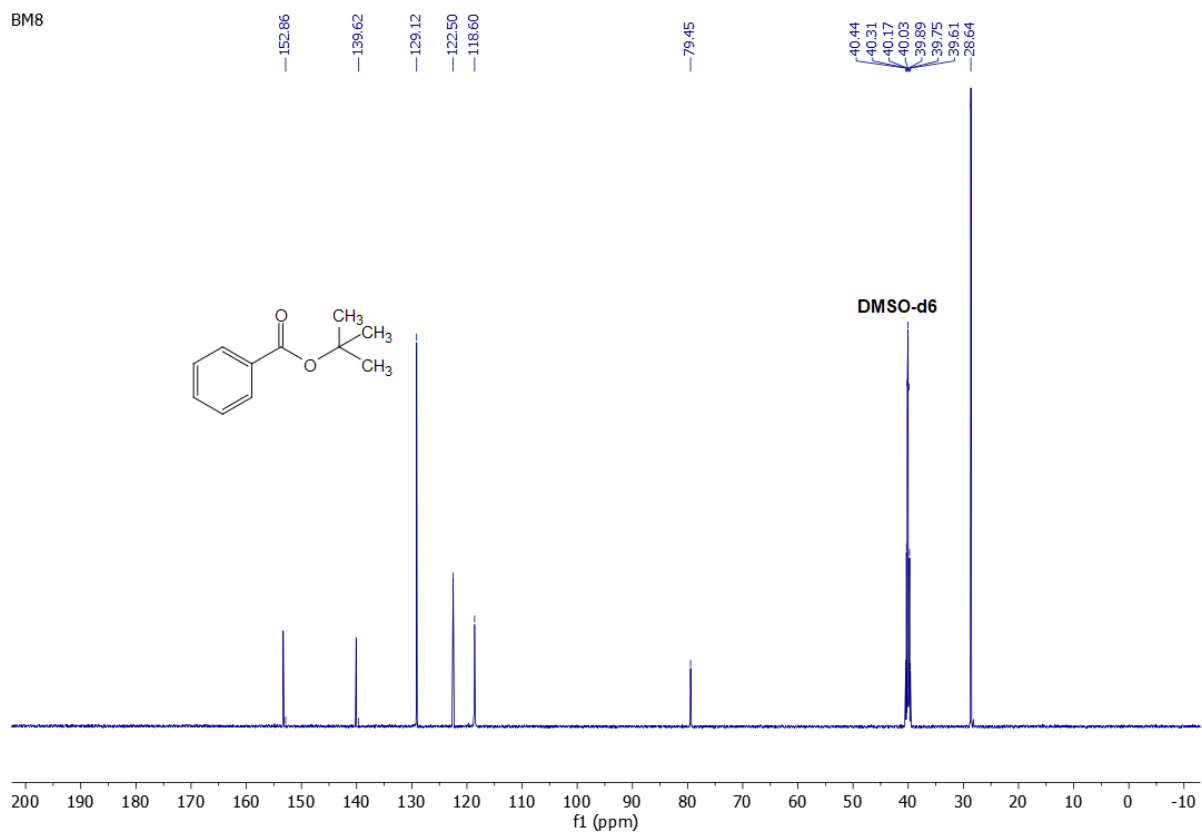
BM6



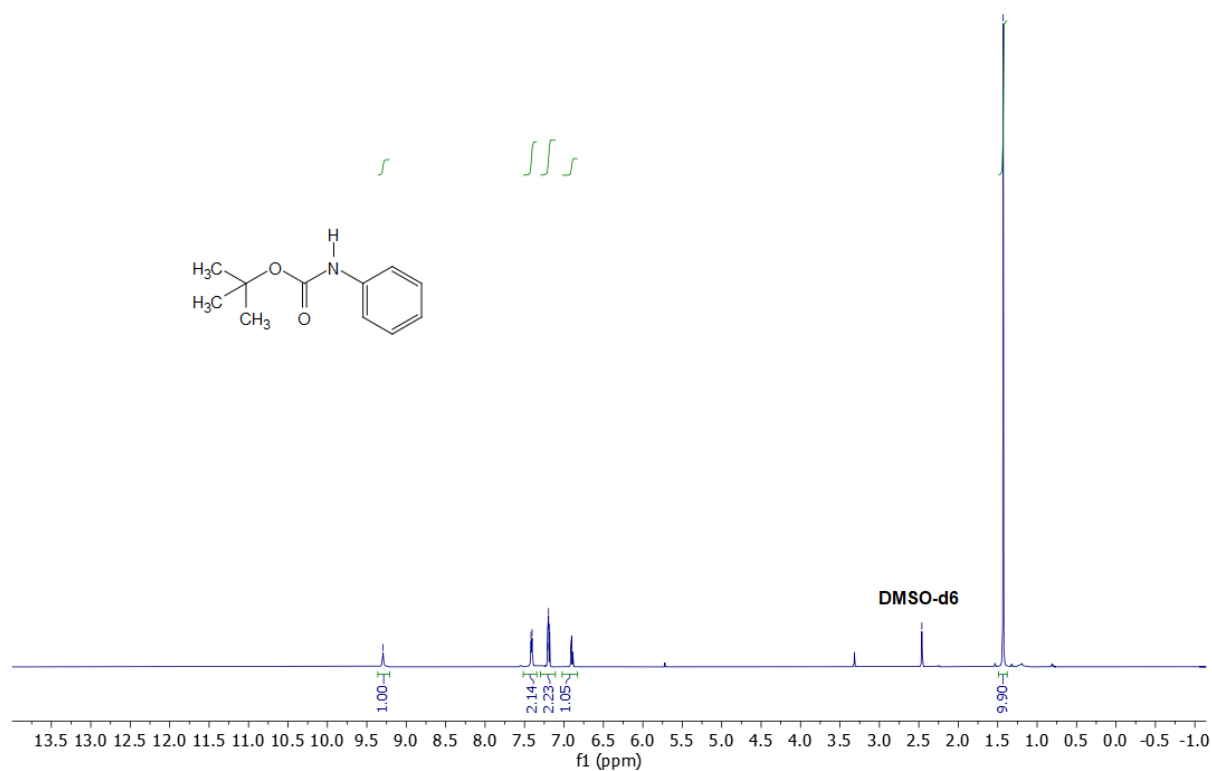
BM8



BM8



— 9.29



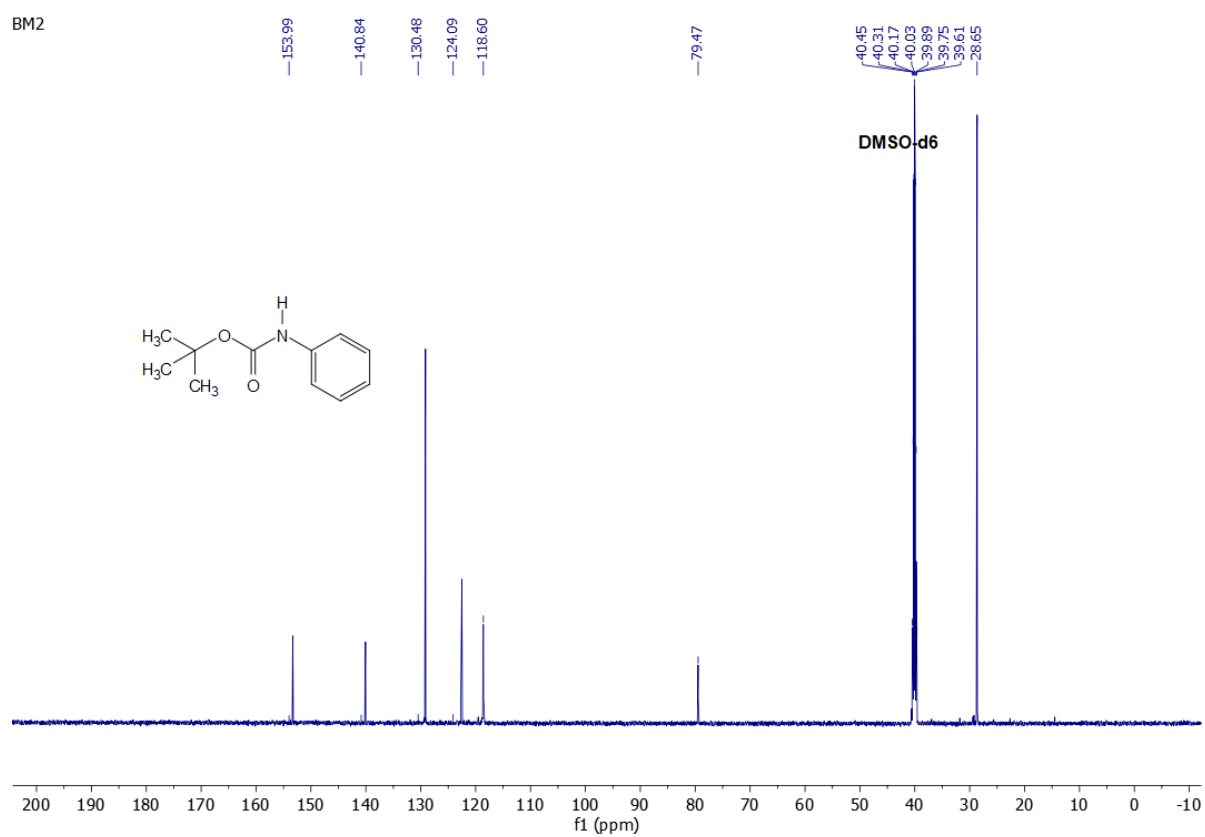
—153.99

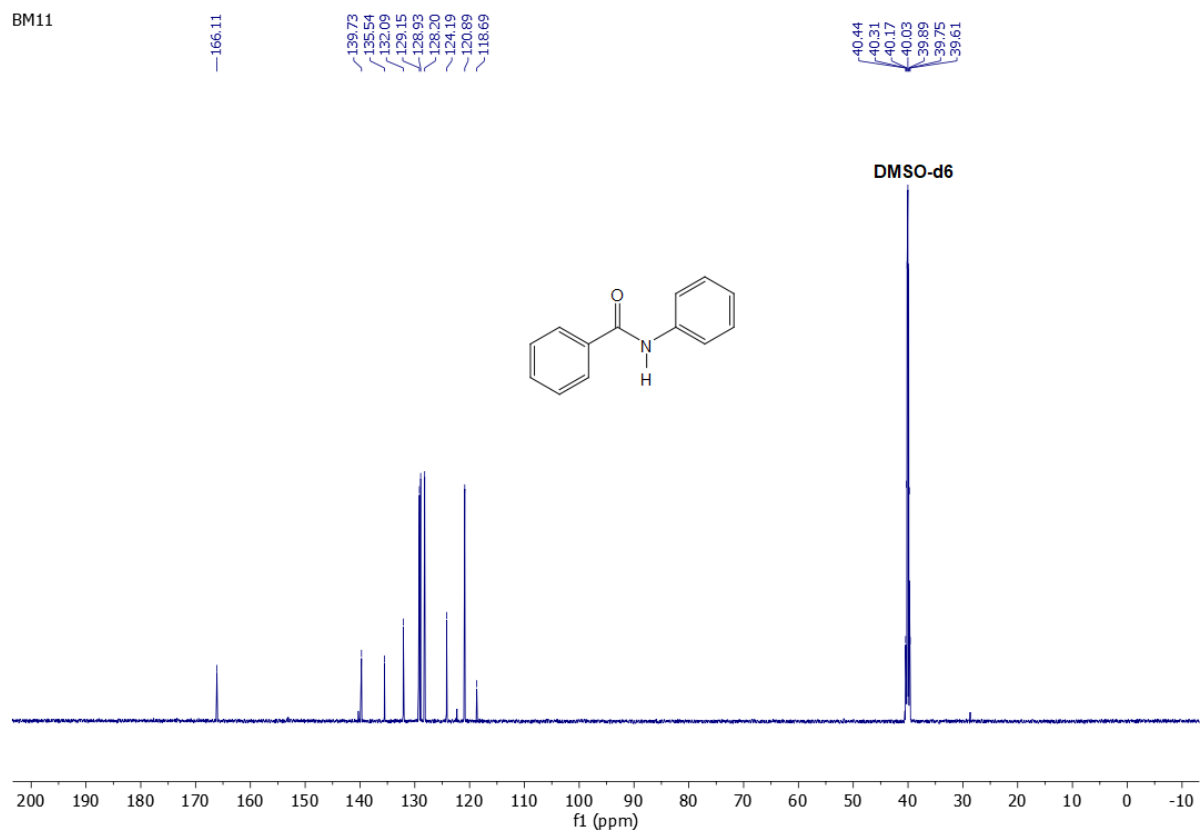
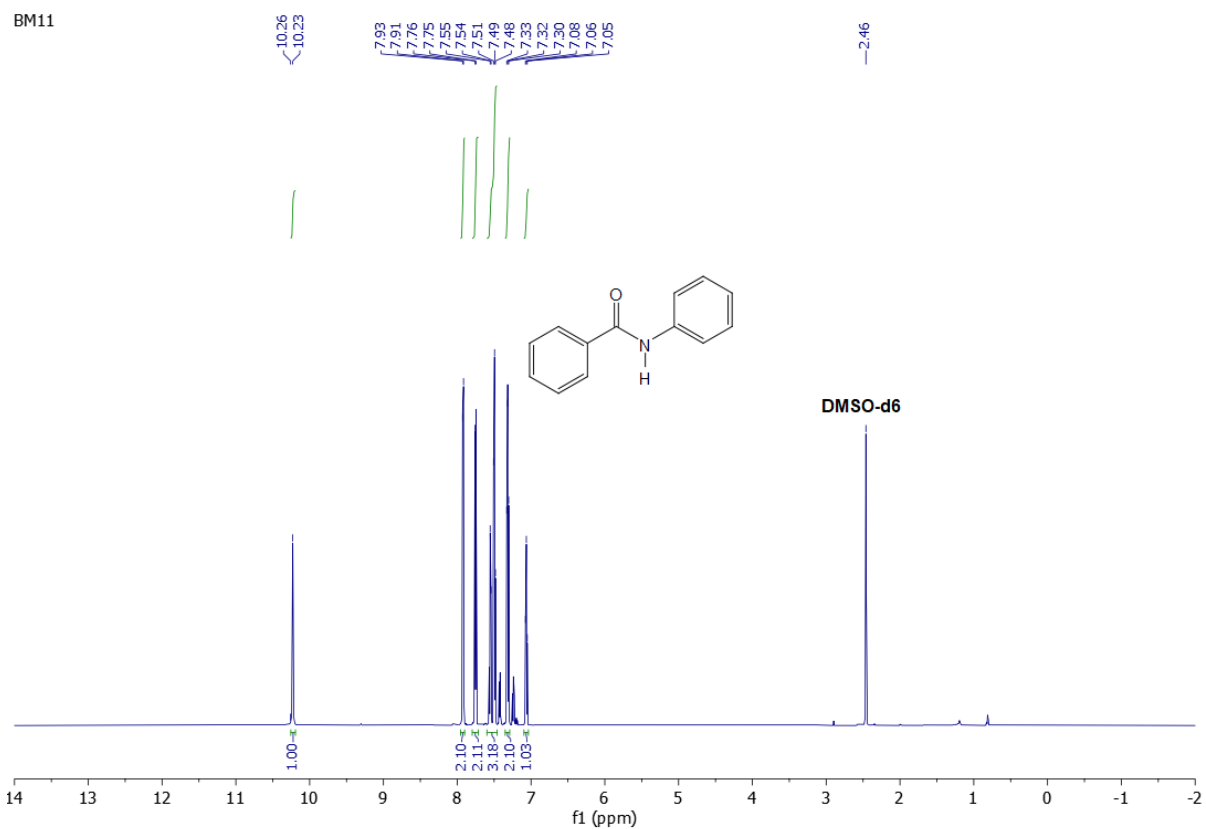
—140.84

—130.48

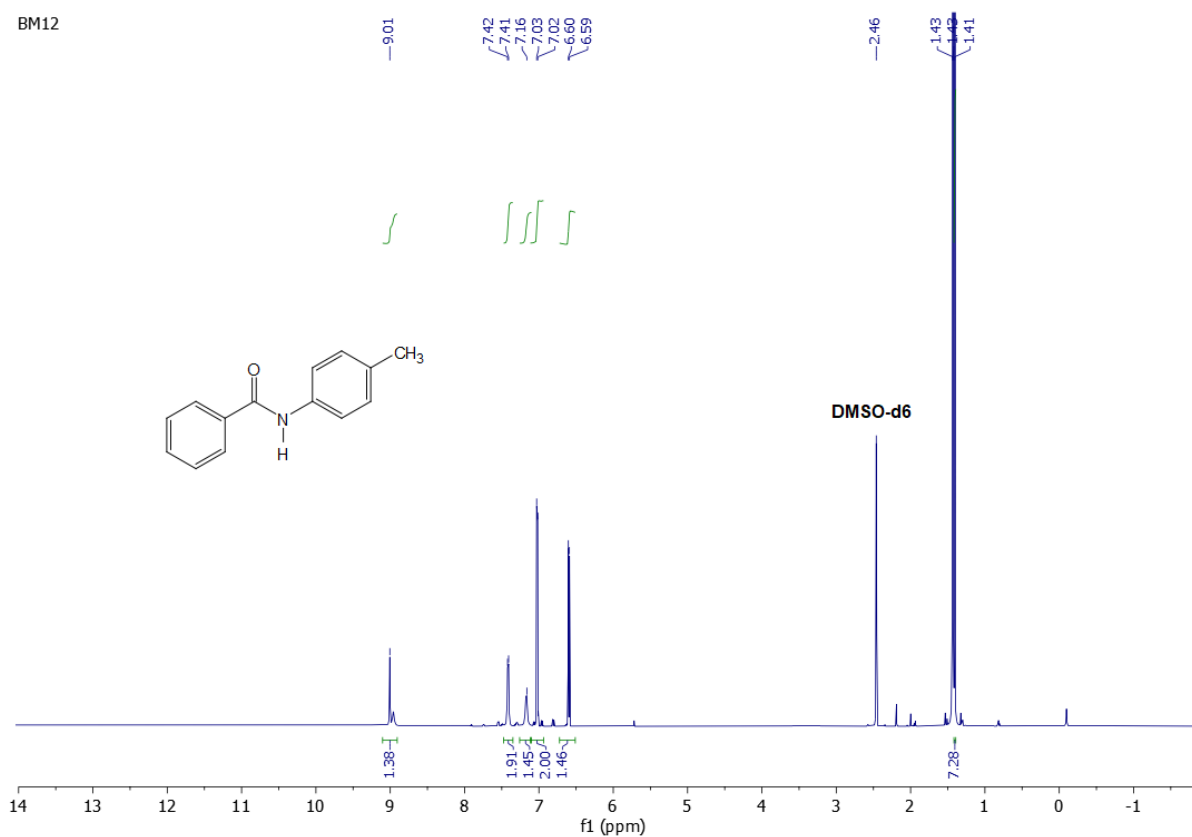
—124.09

—118.60

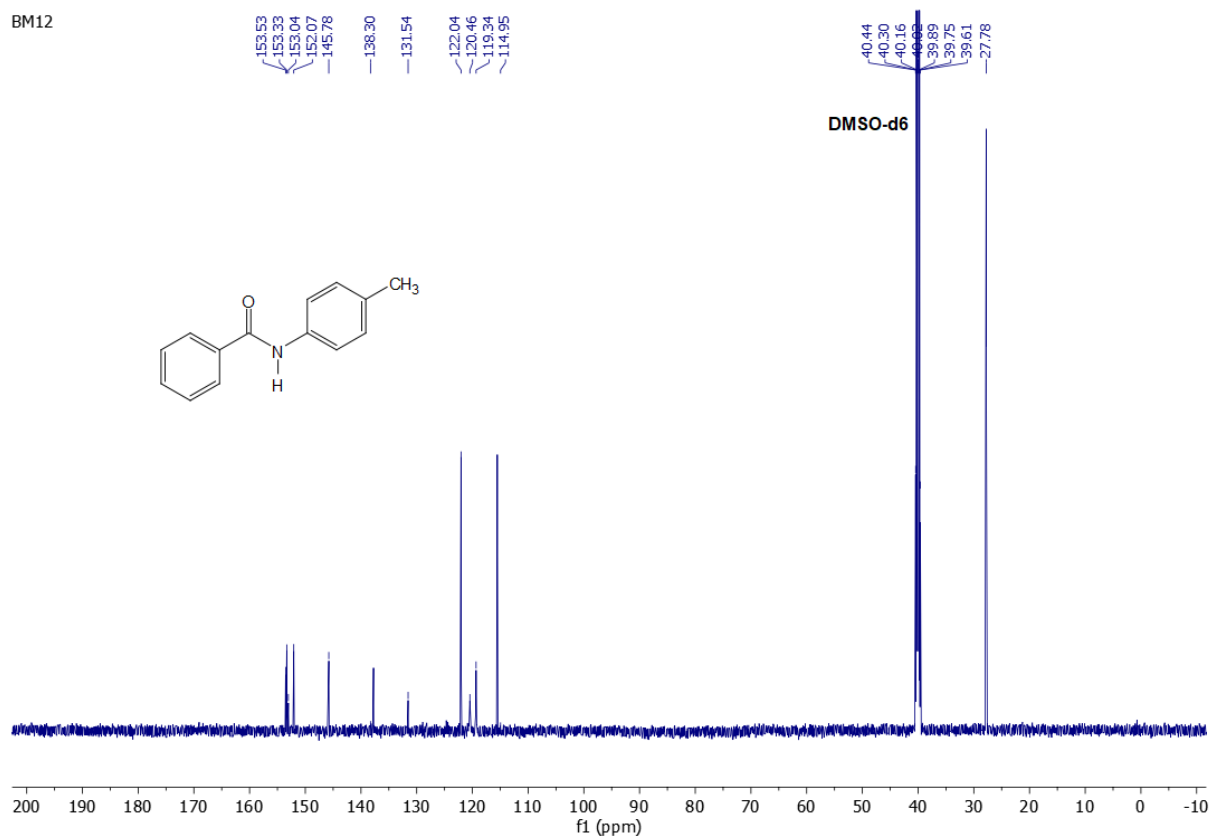


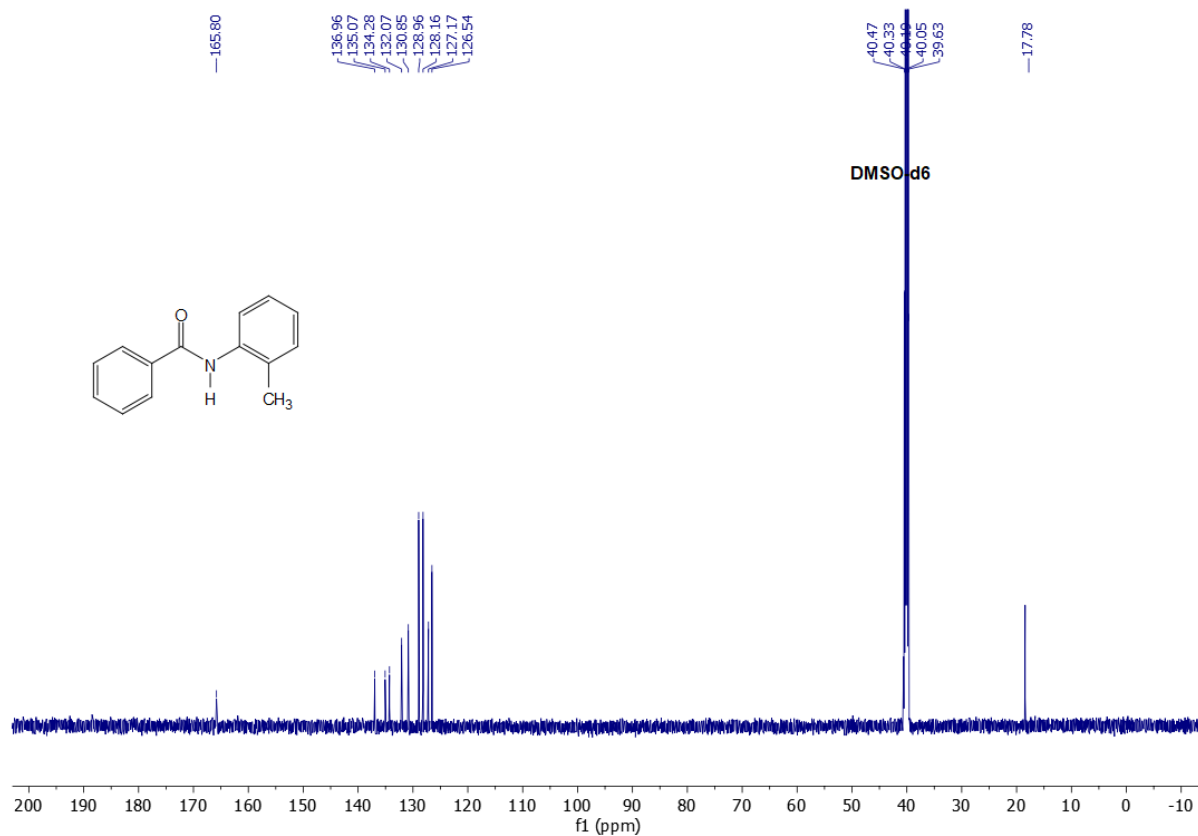
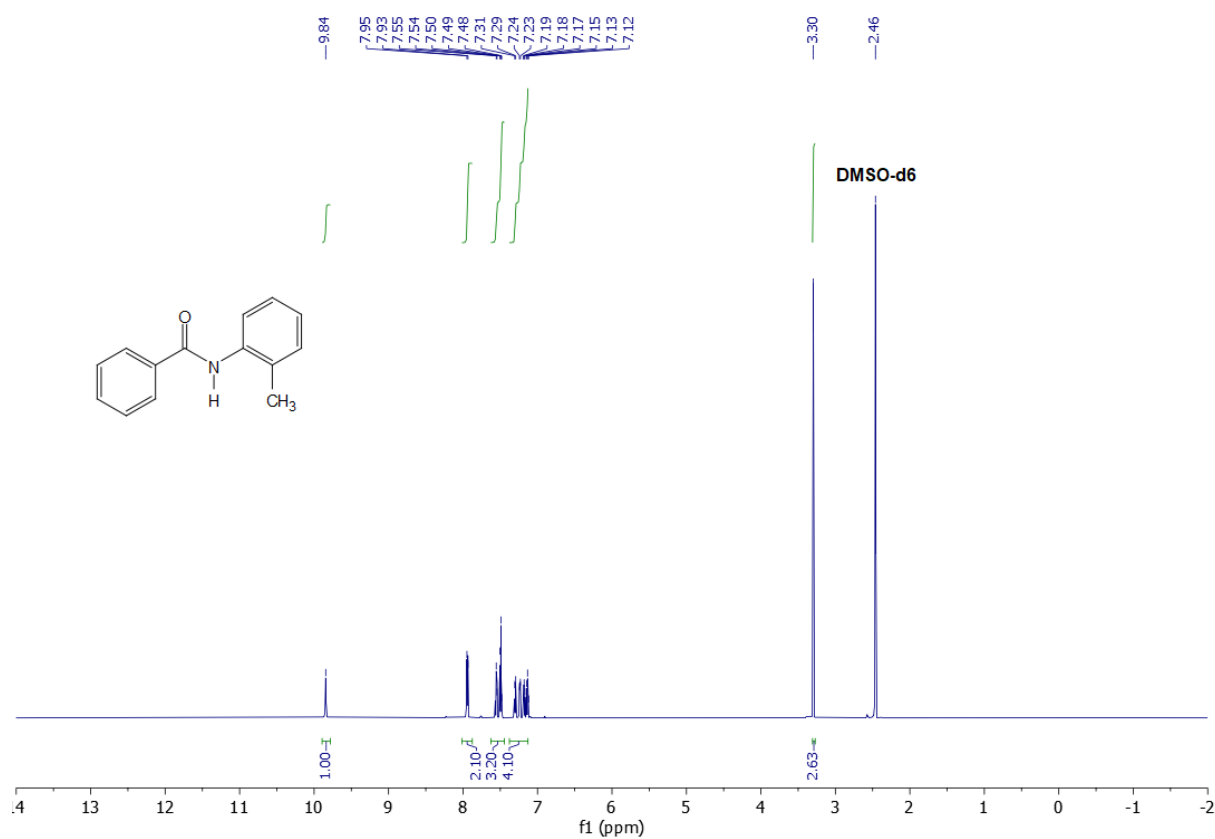


BM12

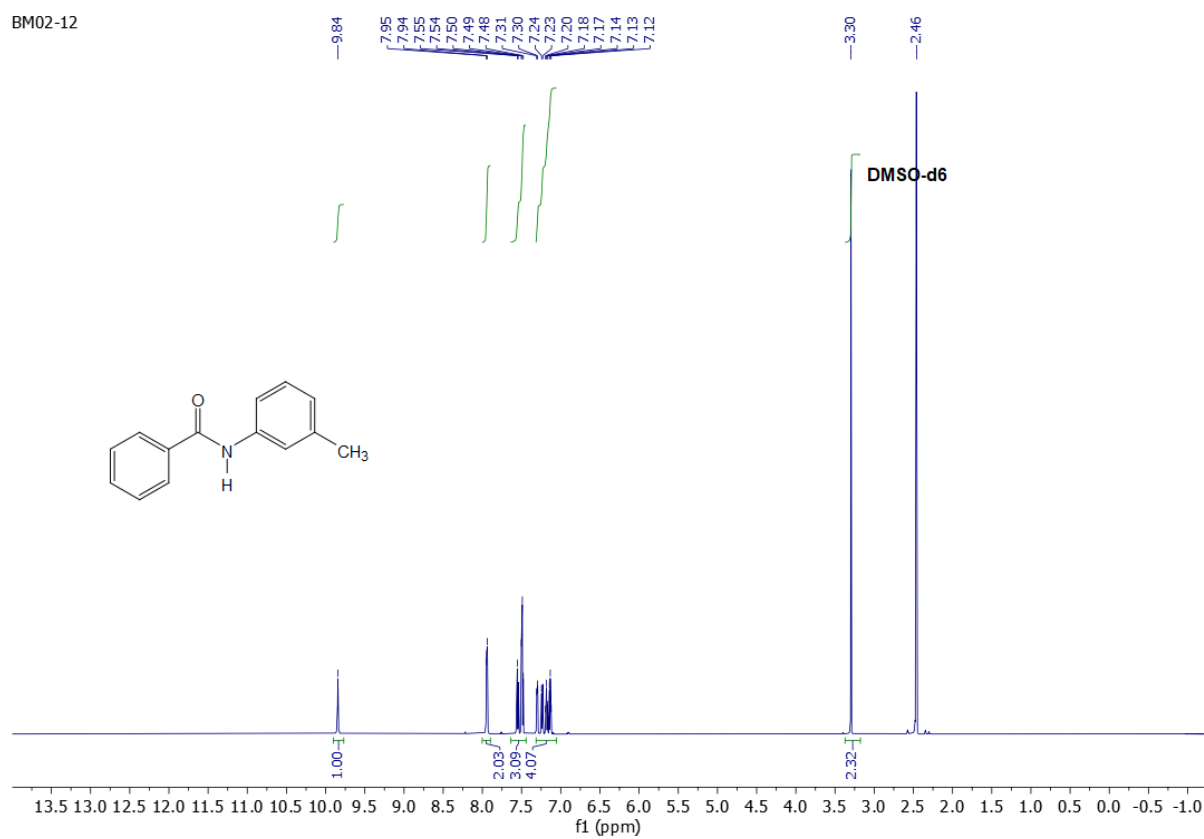


BM12

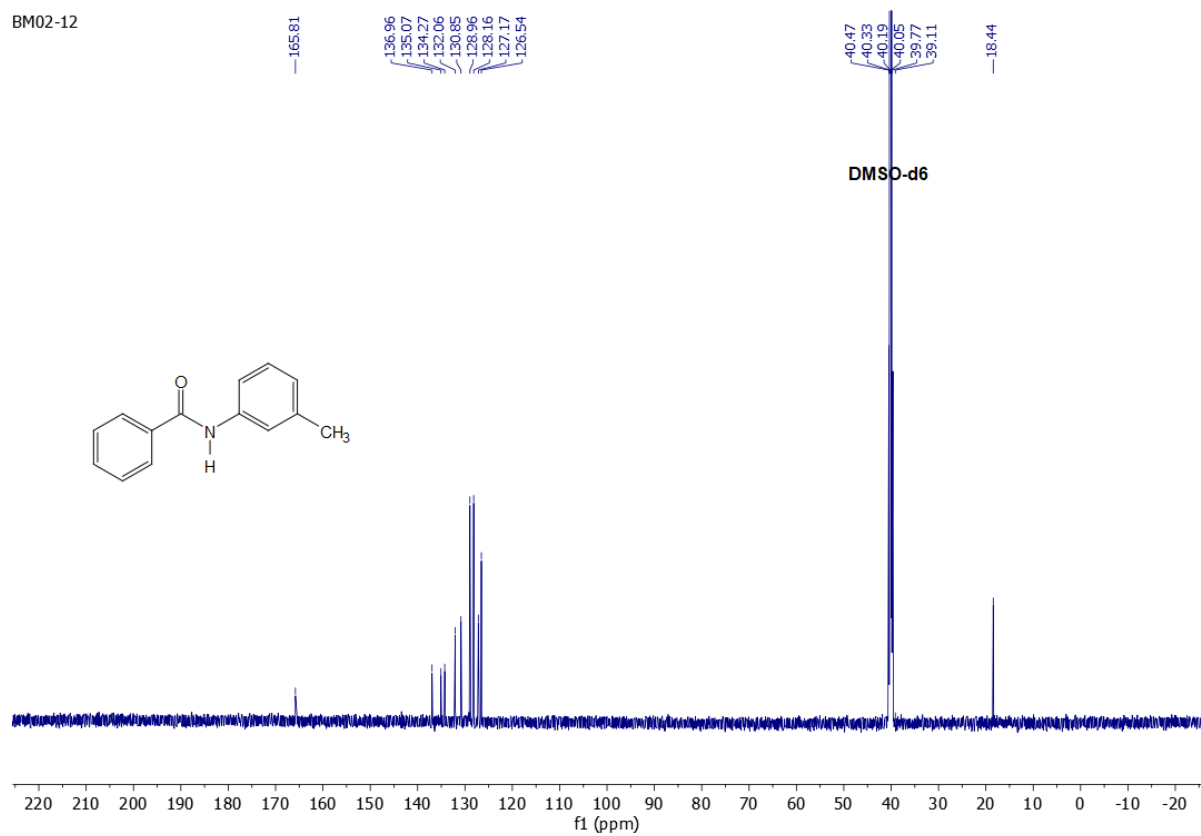




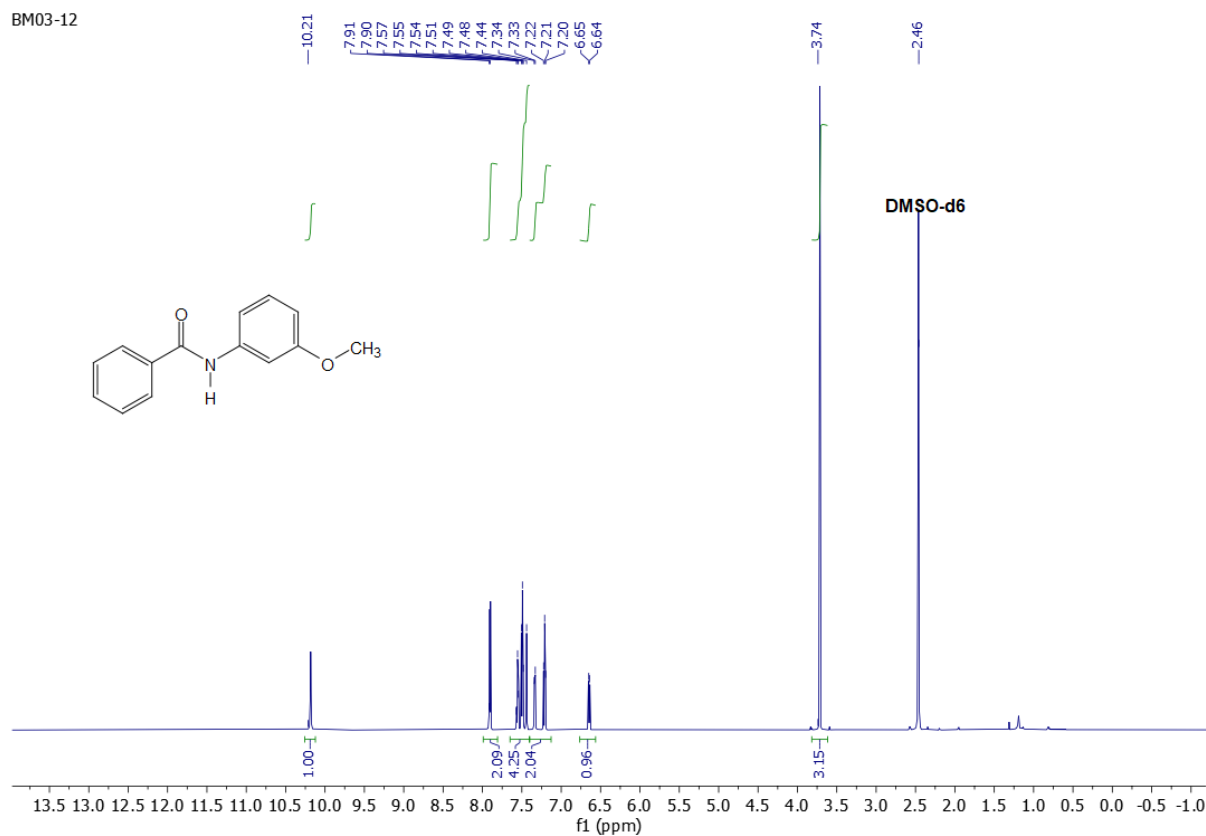
BM02-12



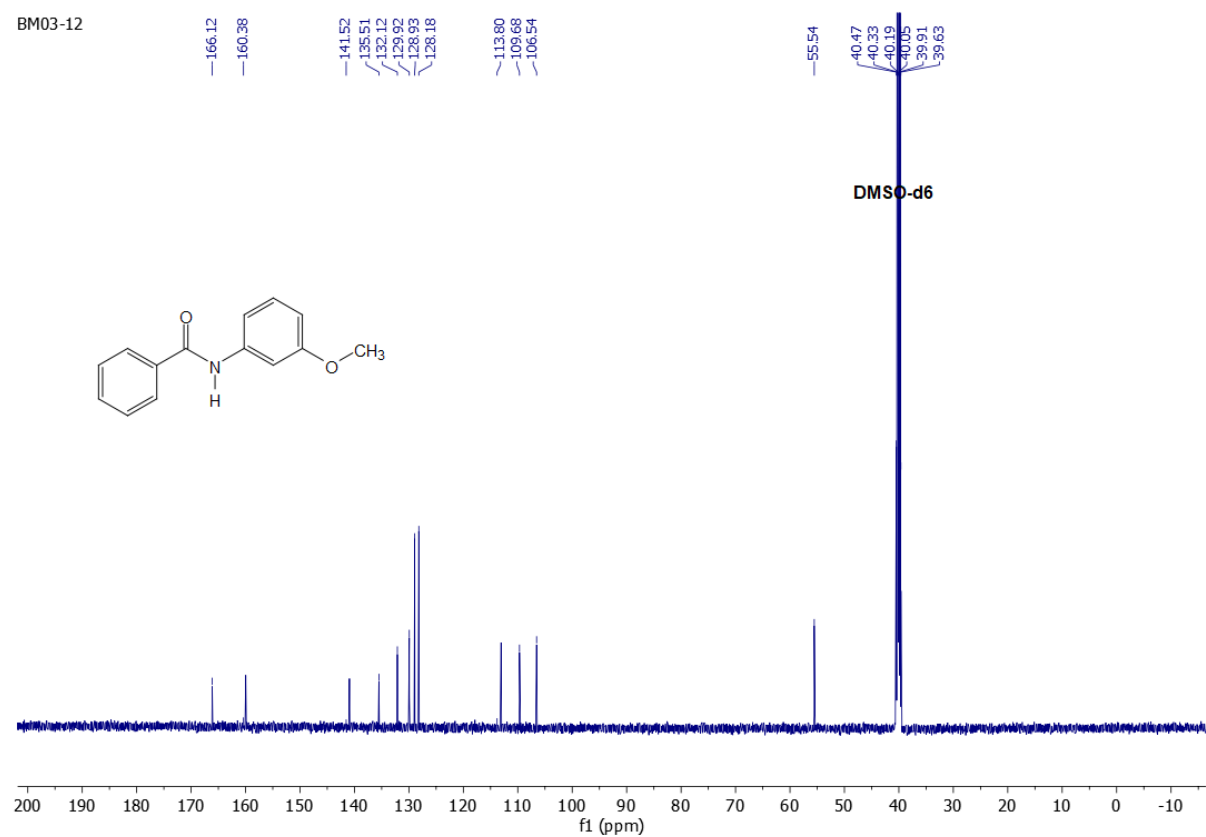
BM02-12



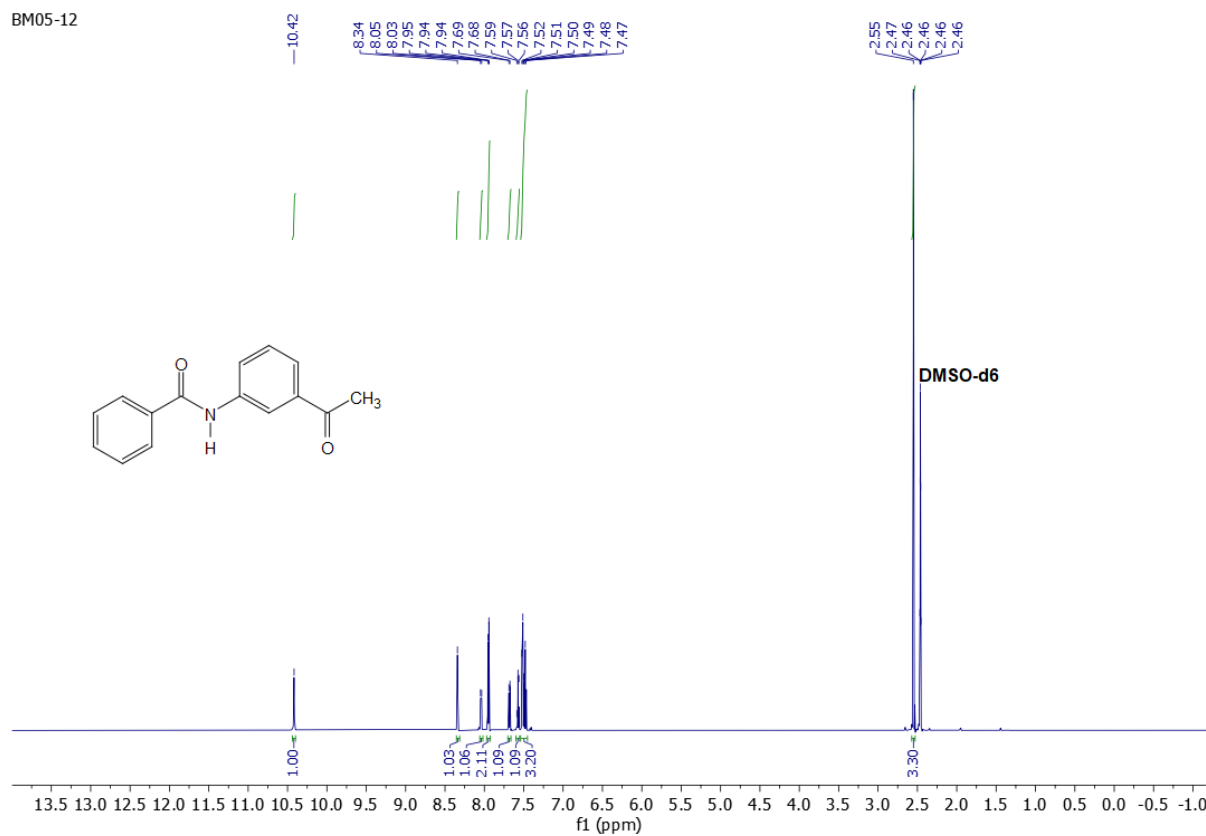
BM03-12



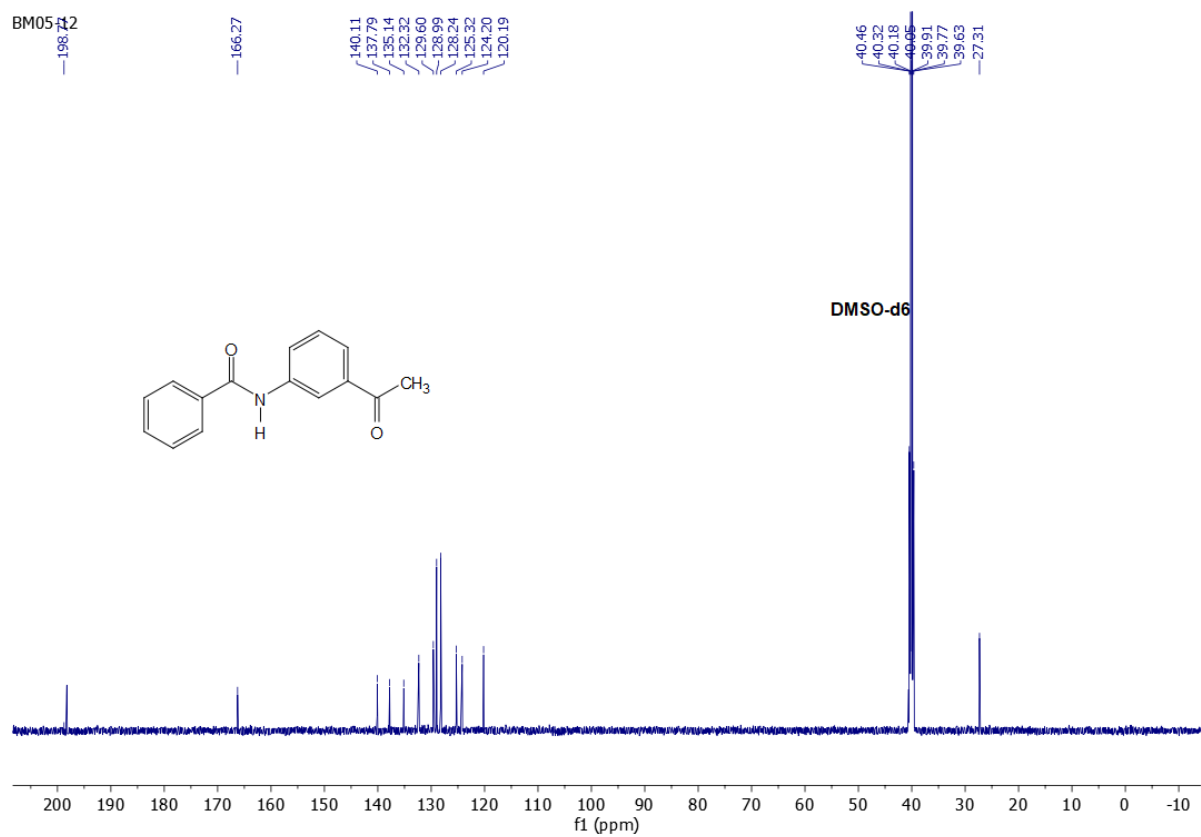
BM03-12



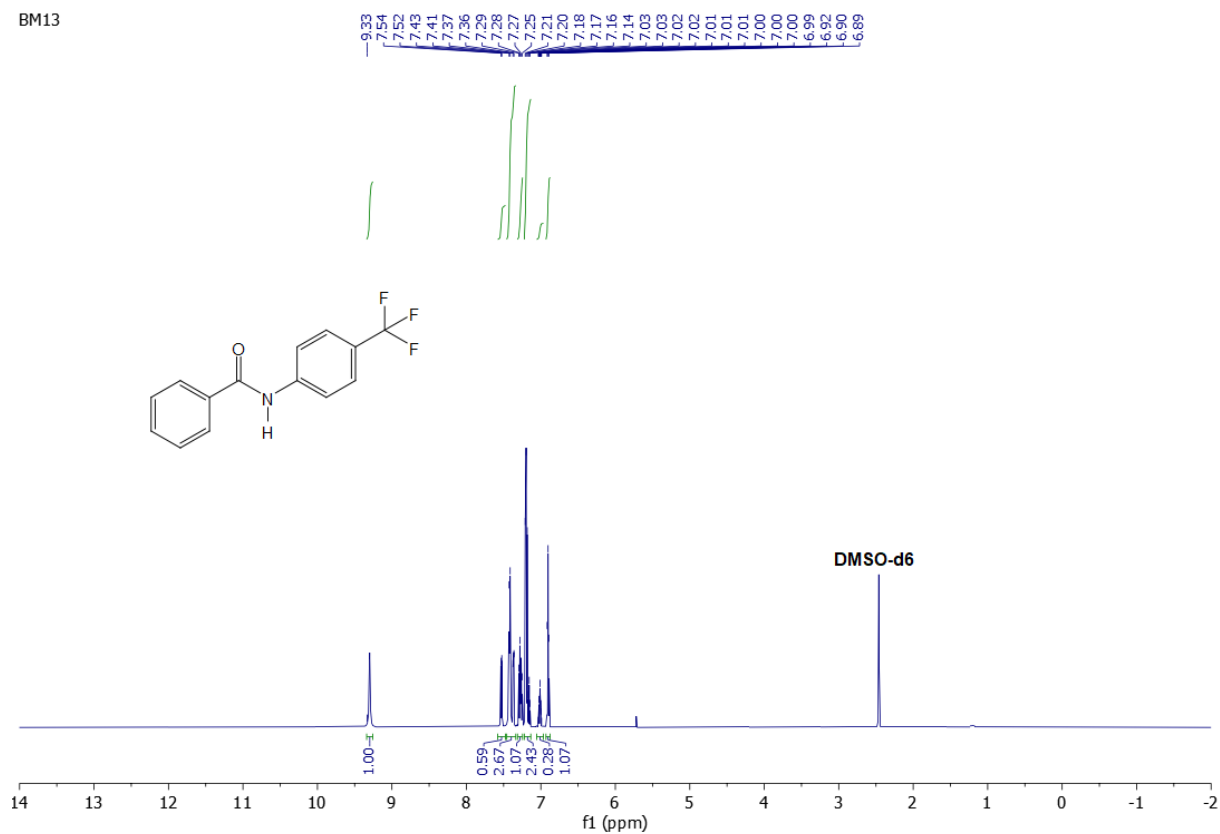
BM05-12



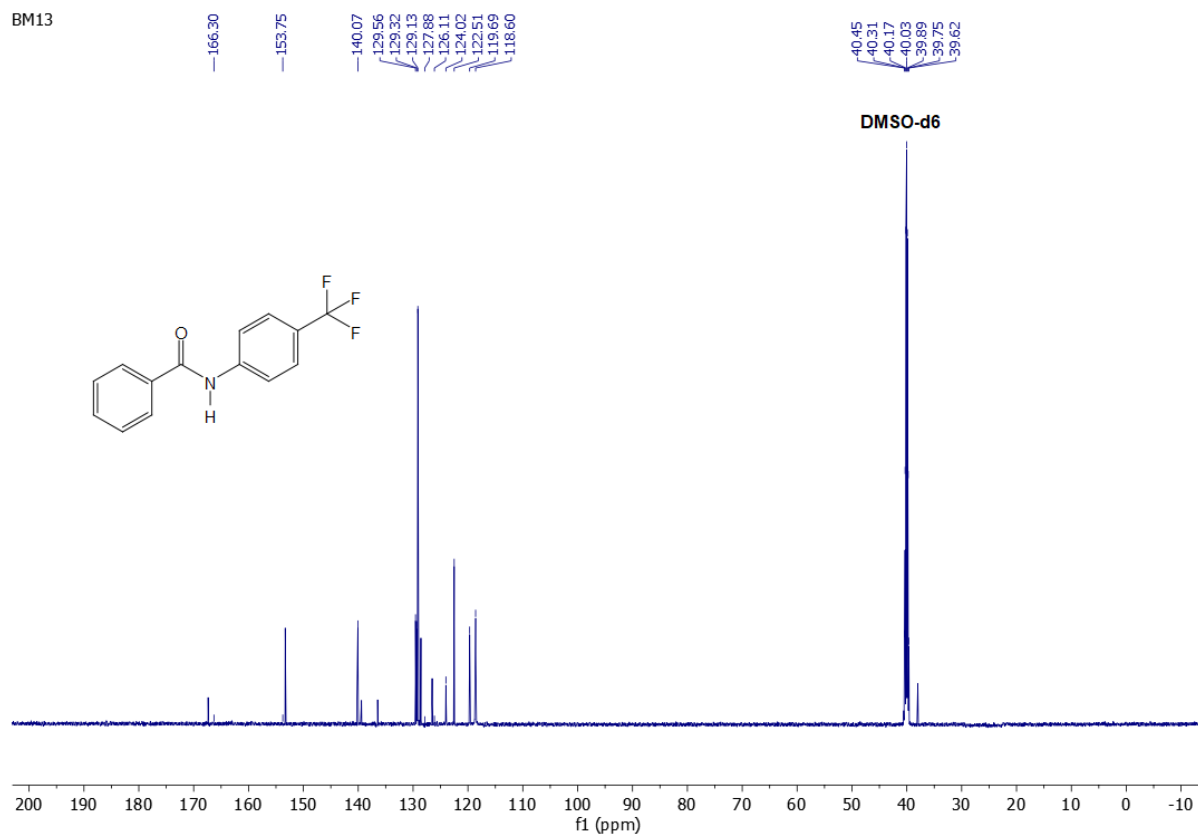
BM05-12



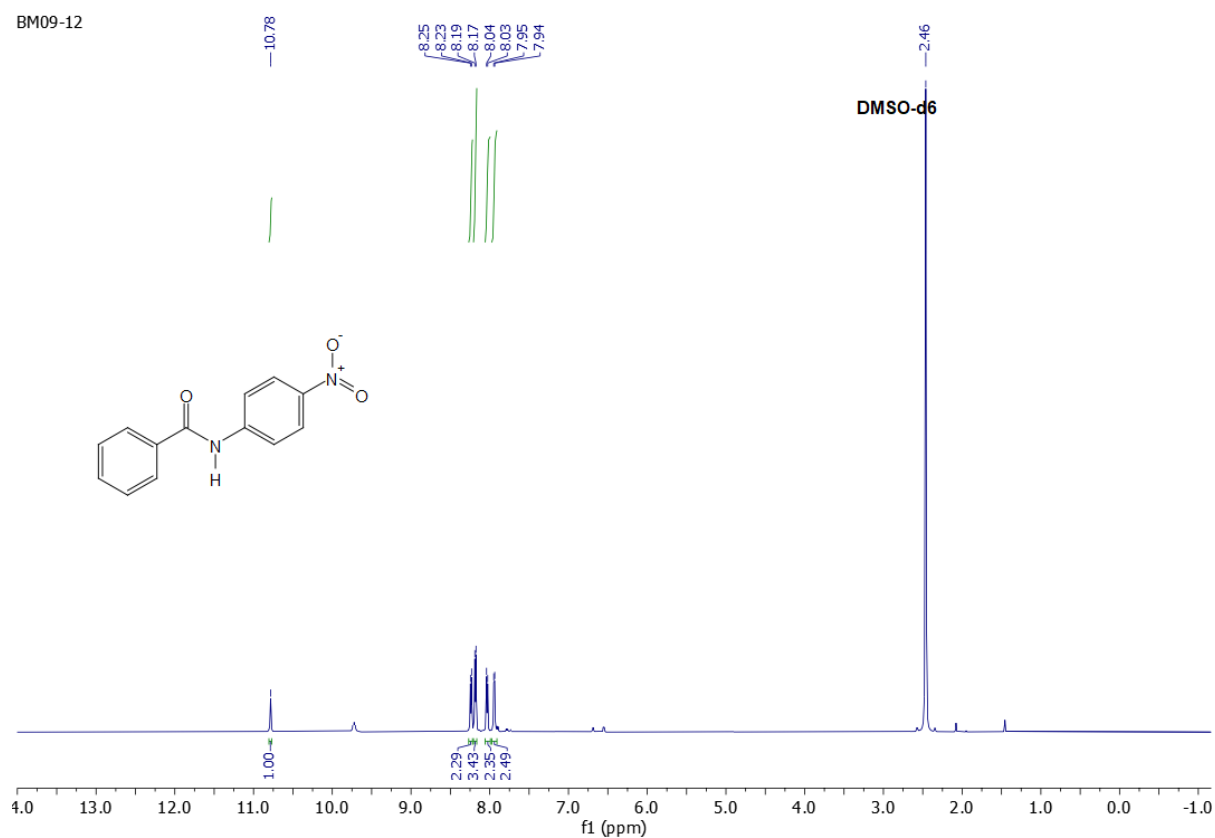
BM13



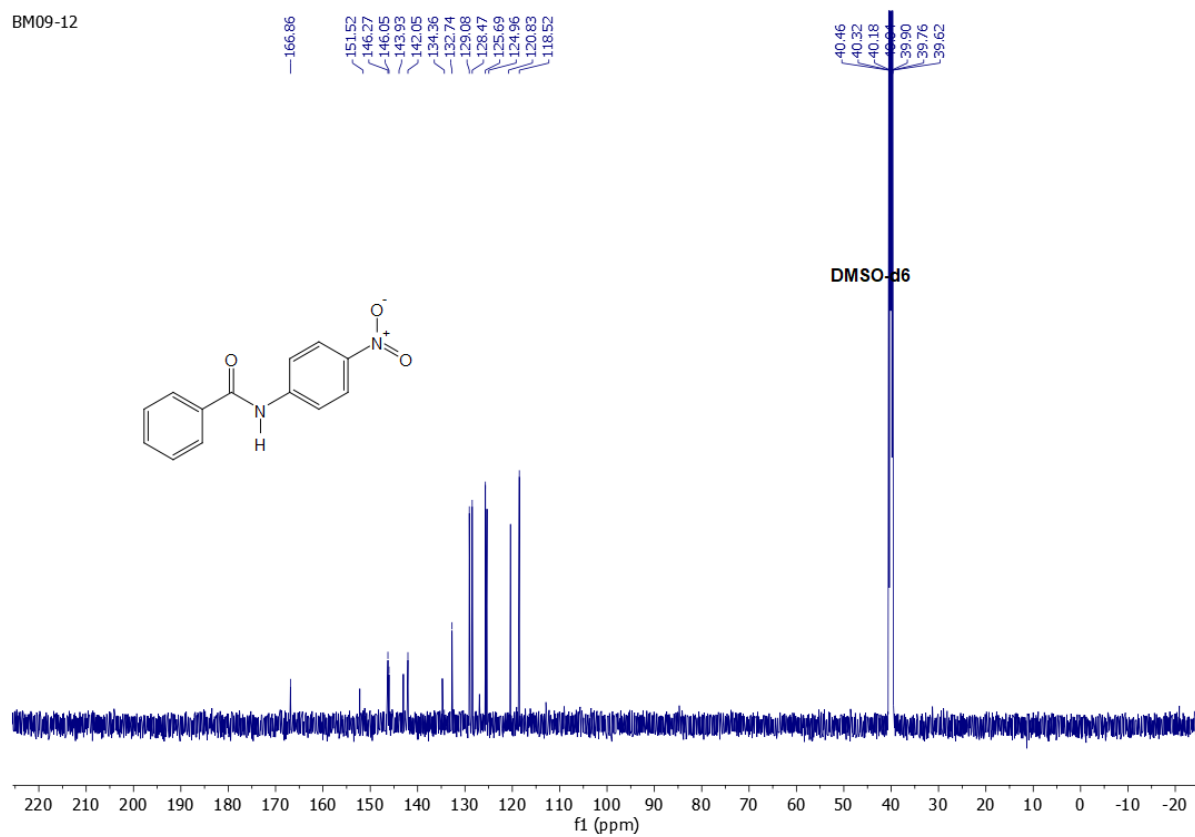
BM13



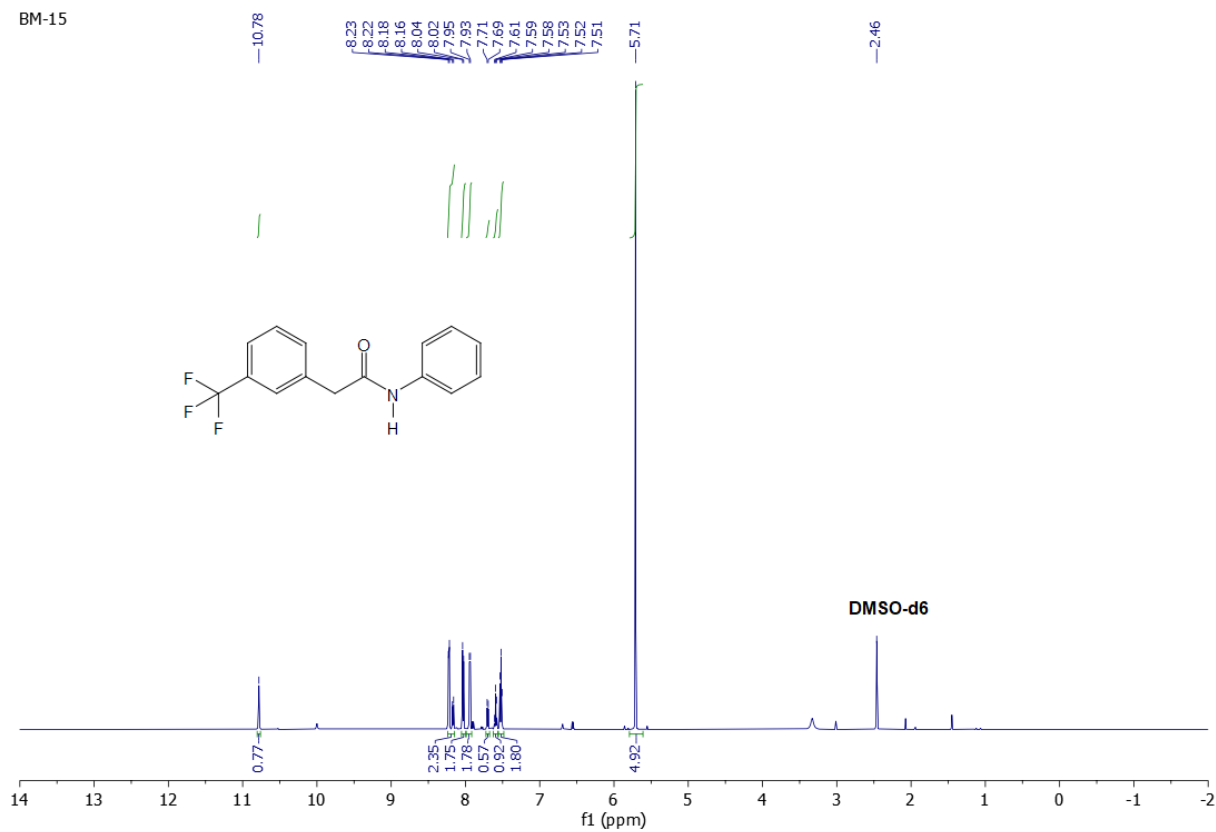
BM09-12



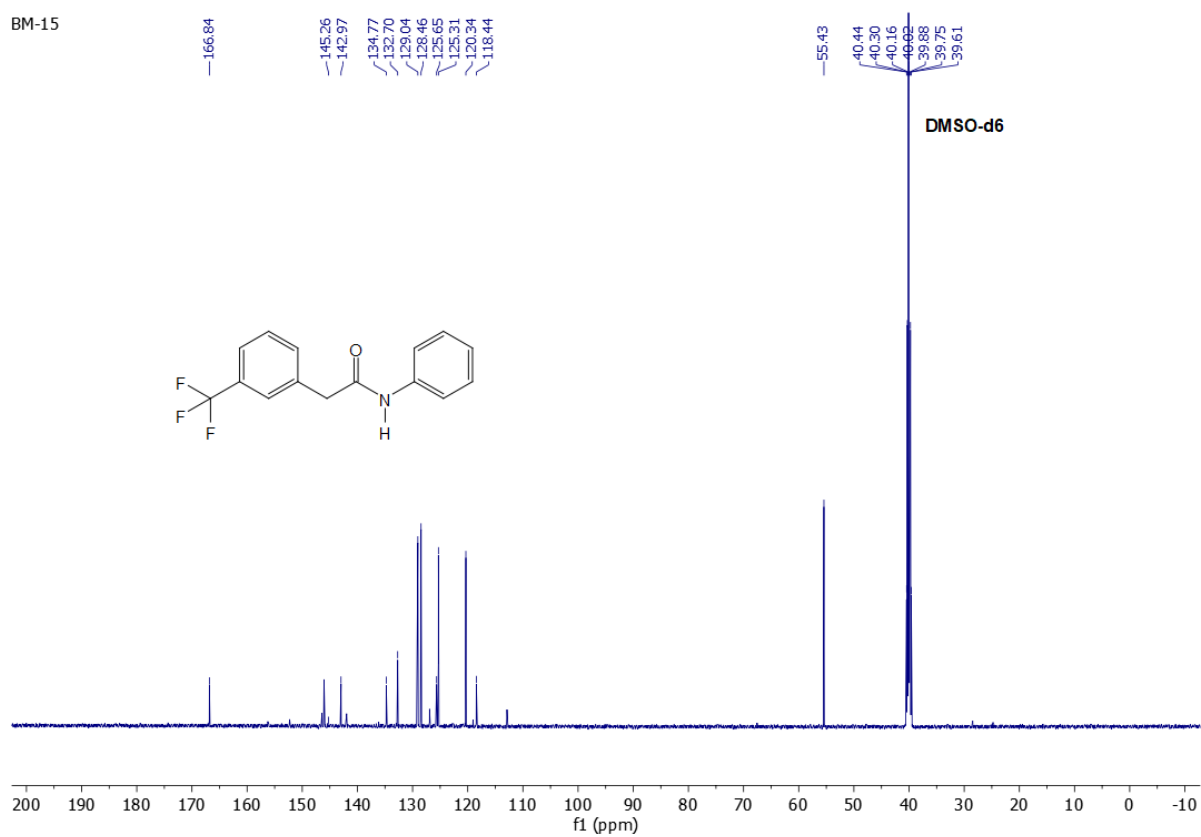
BM09-12

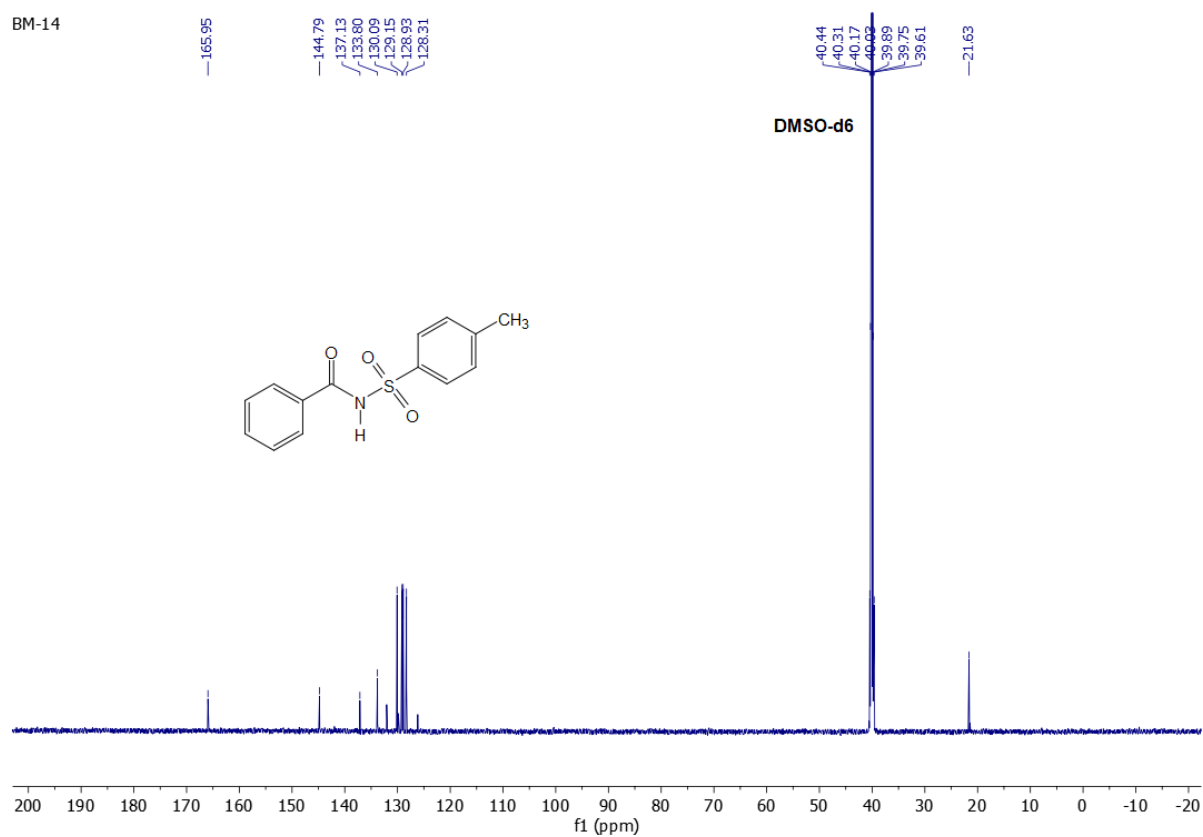
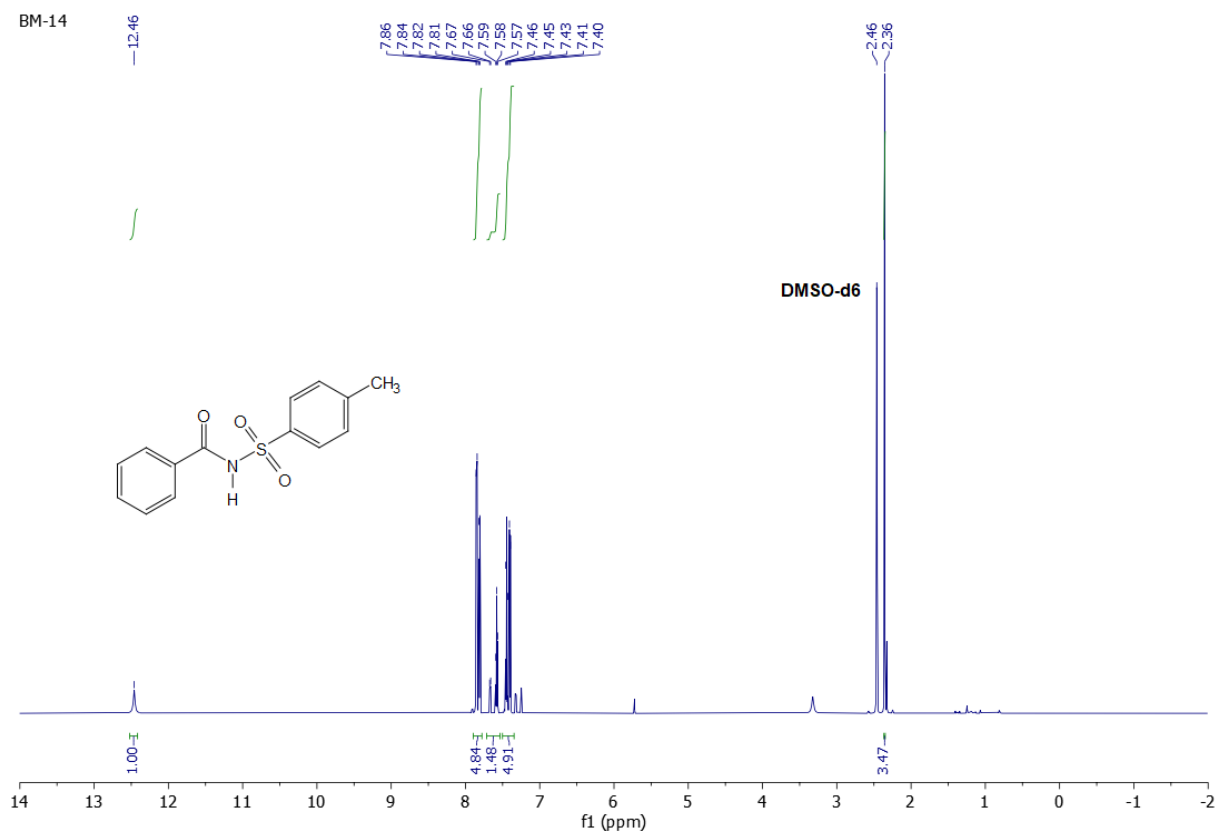


BM-15

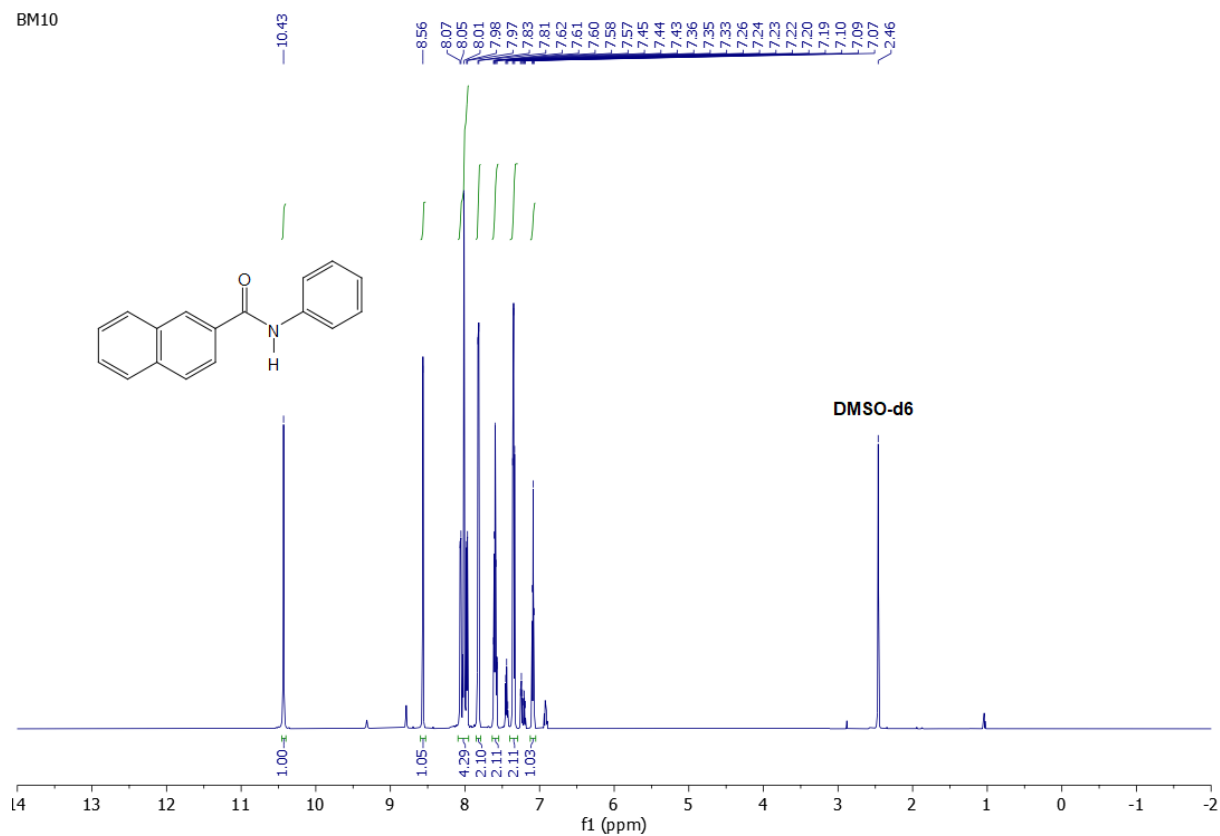


BM-15

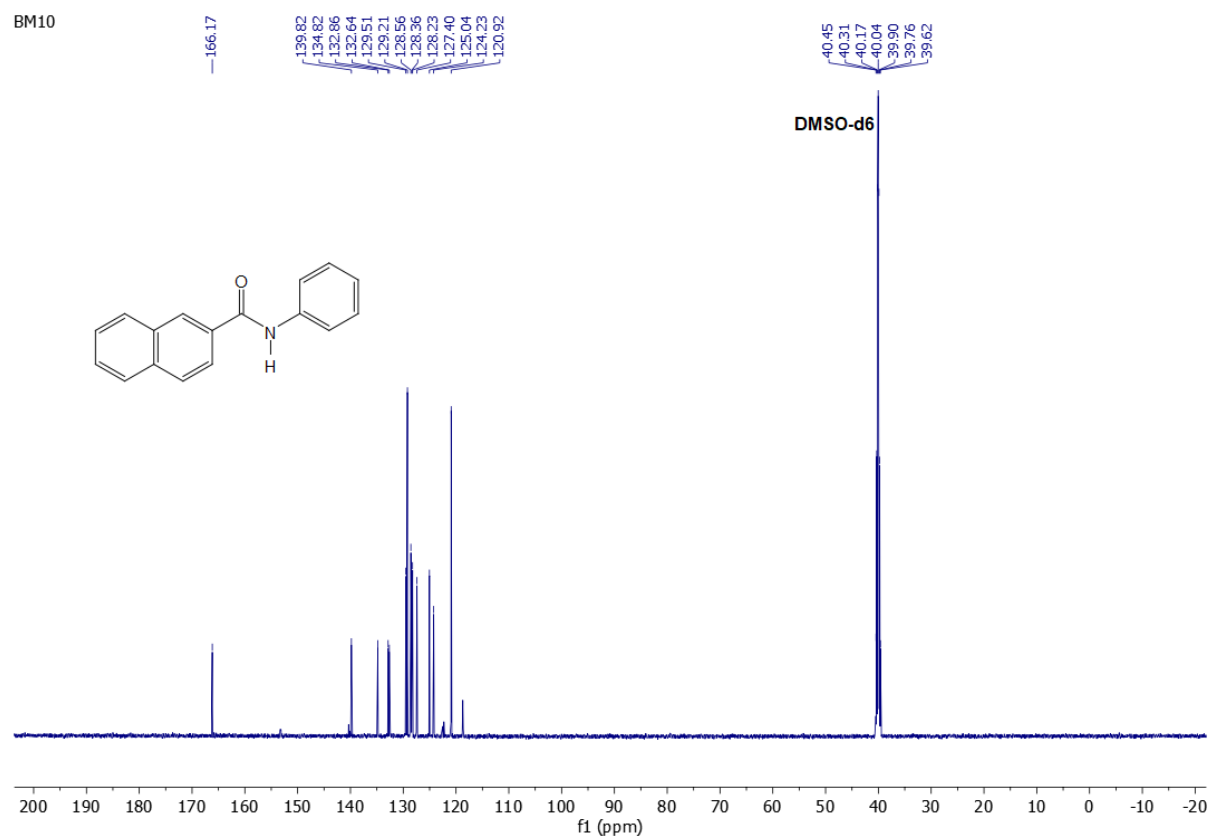




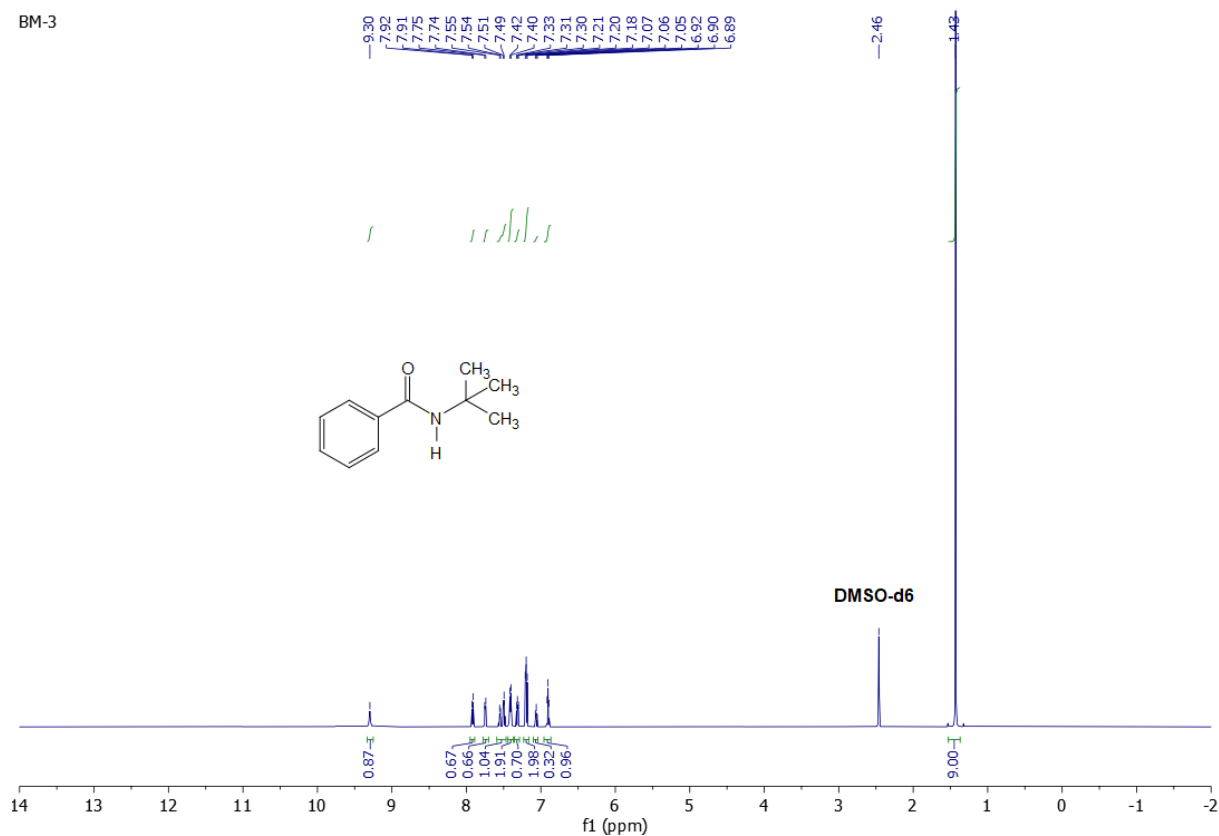
BM10



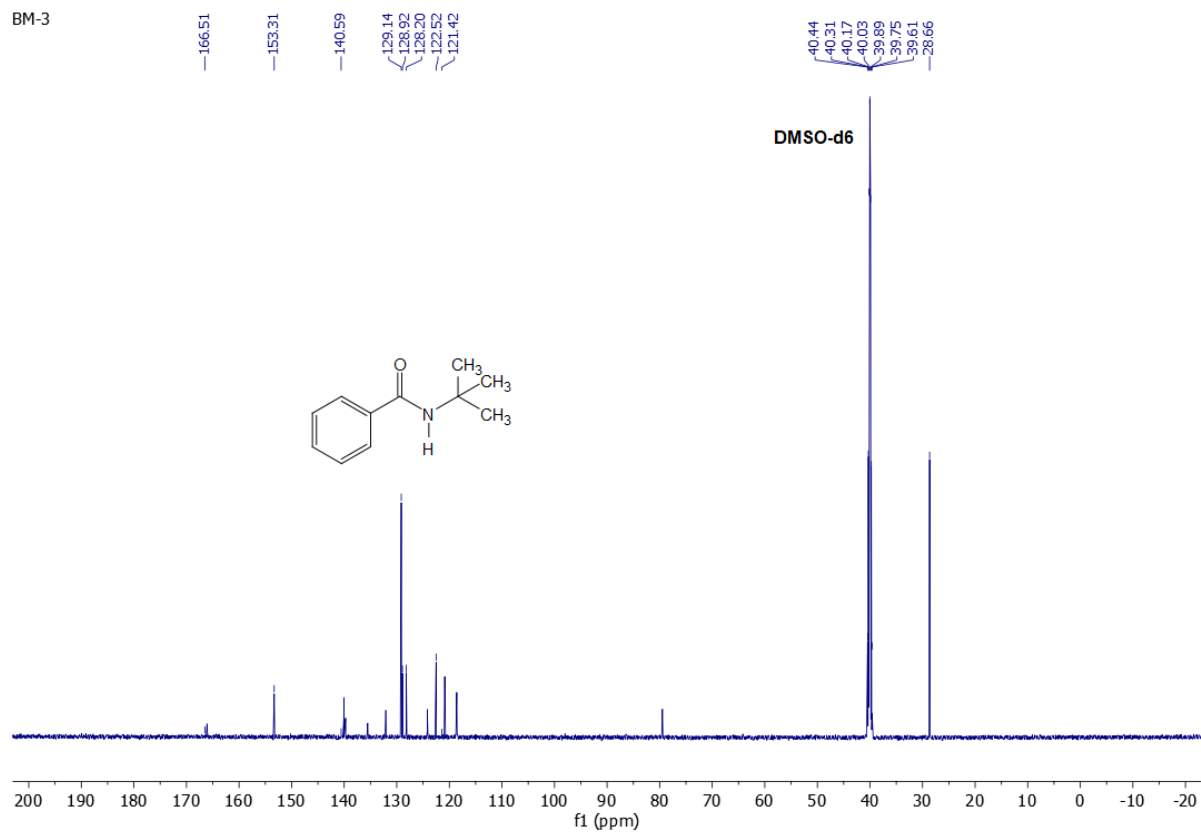
BM10



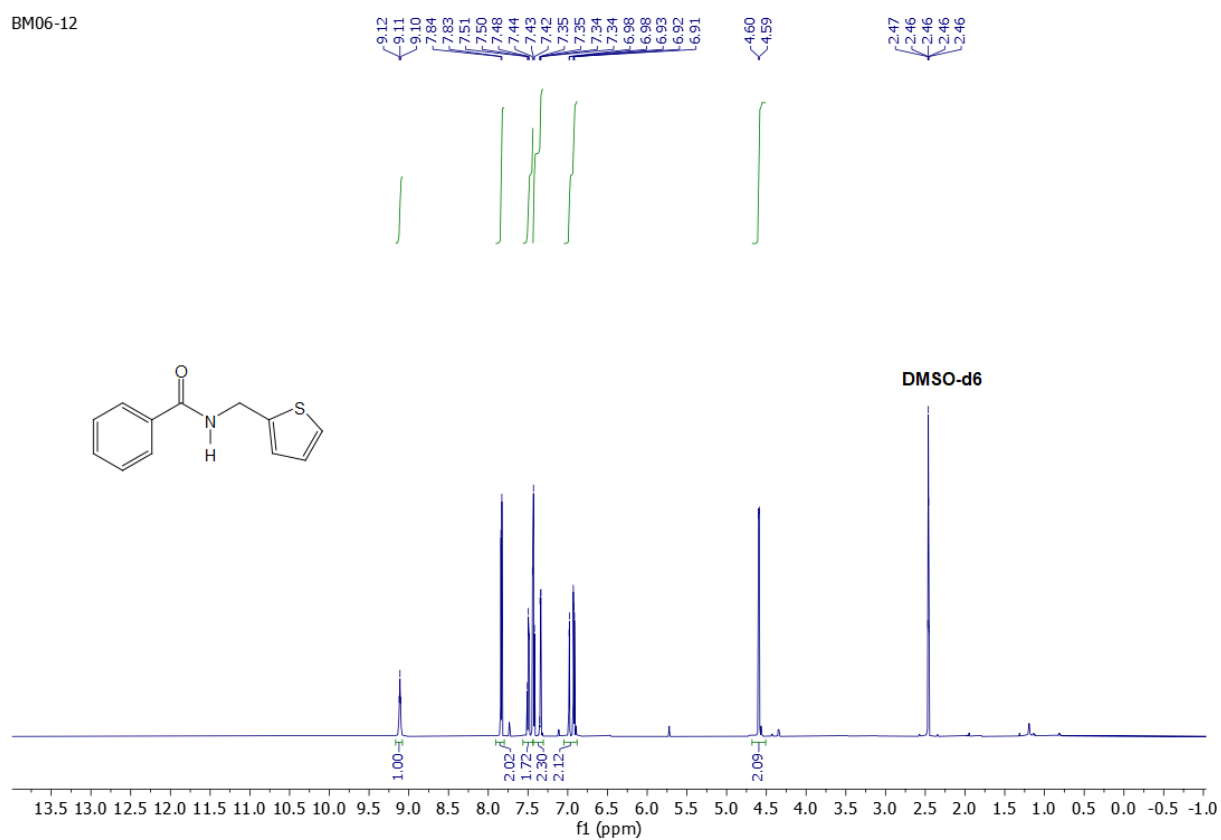
BM-3



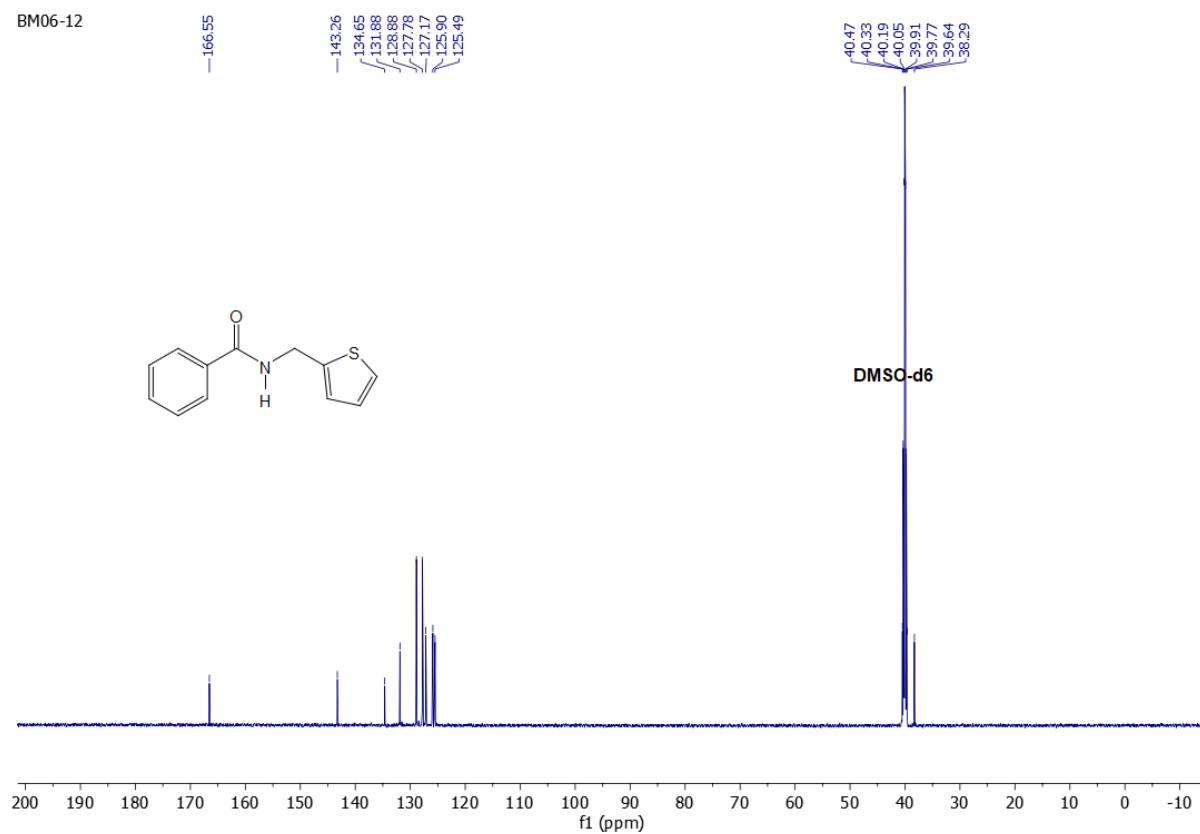
BM-3



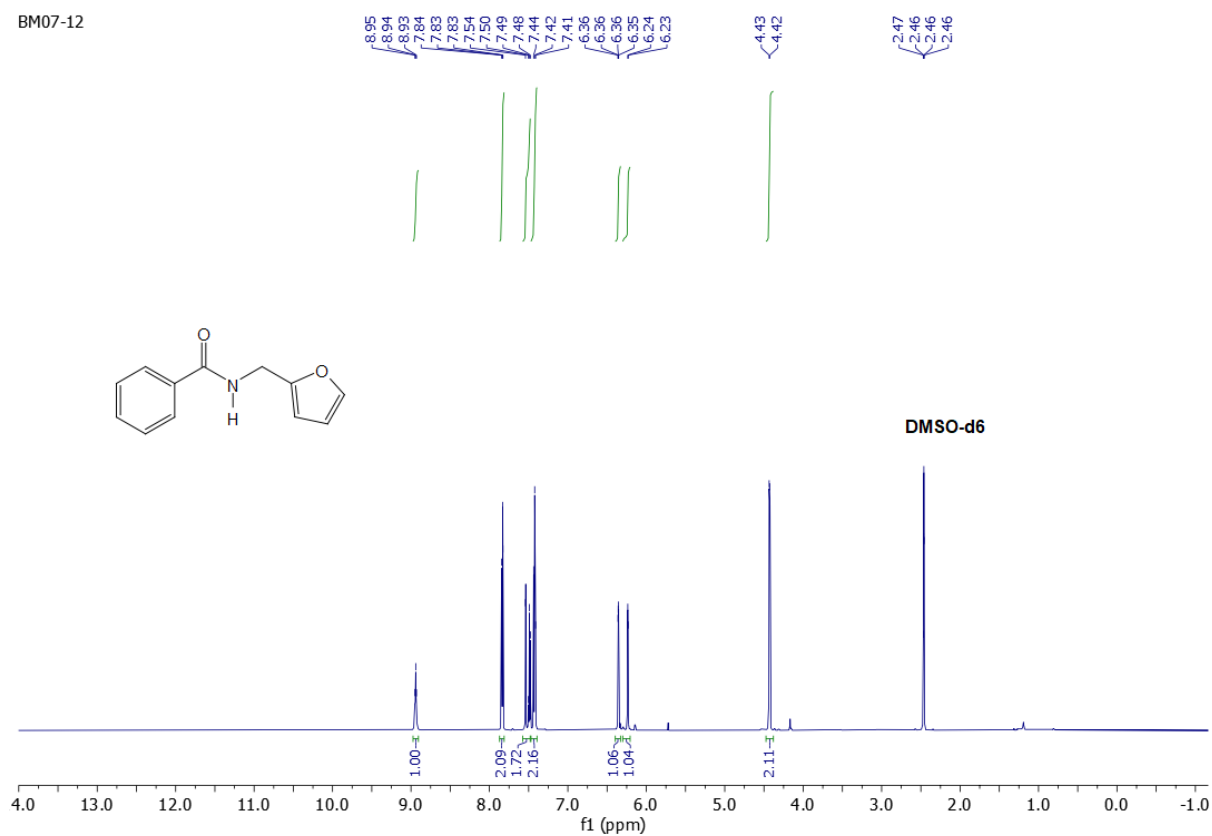
BM06-12



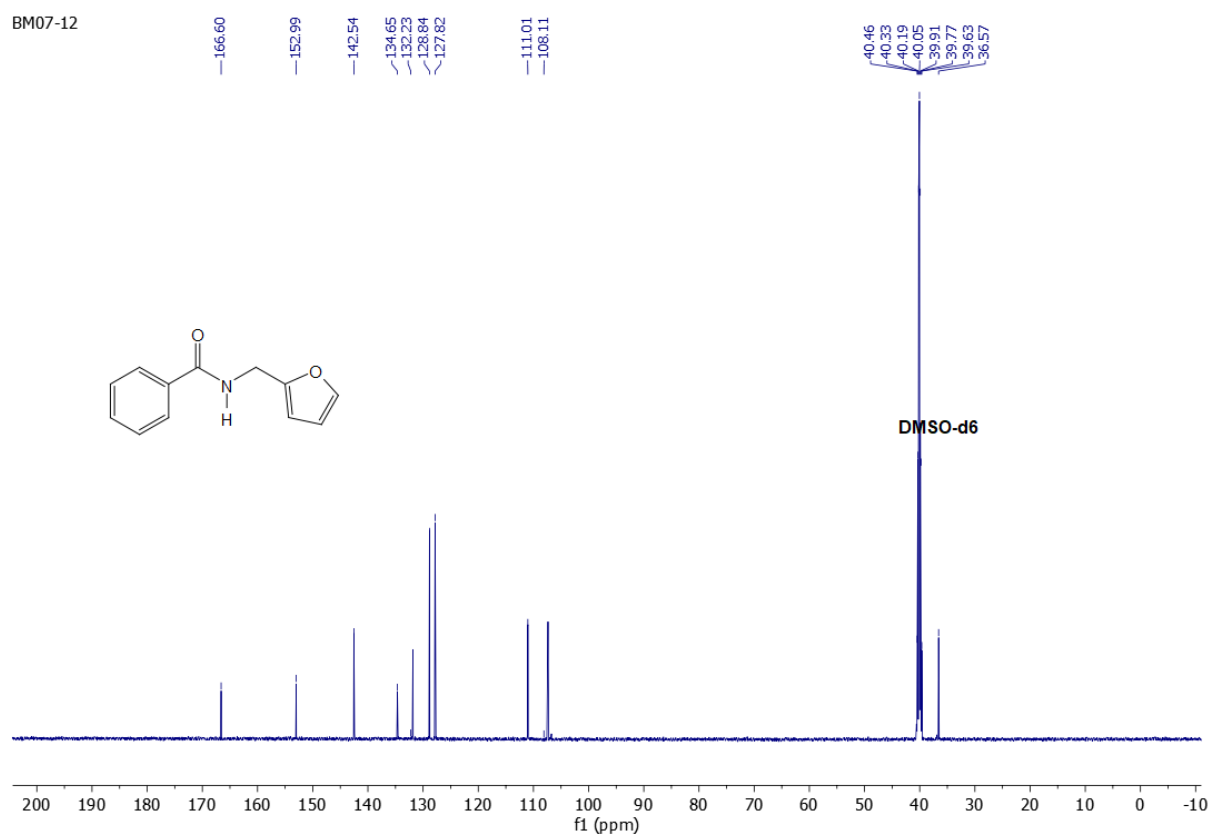
BM06-12



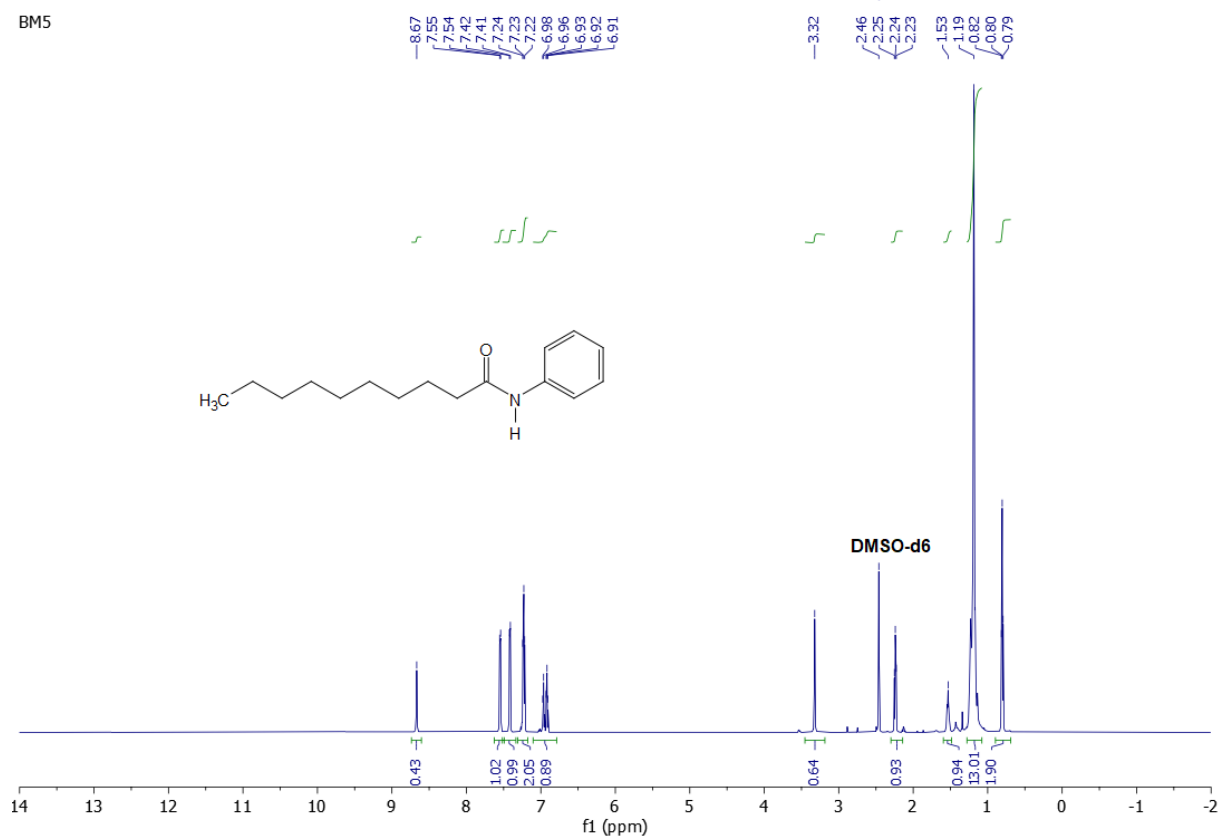
BM07-12



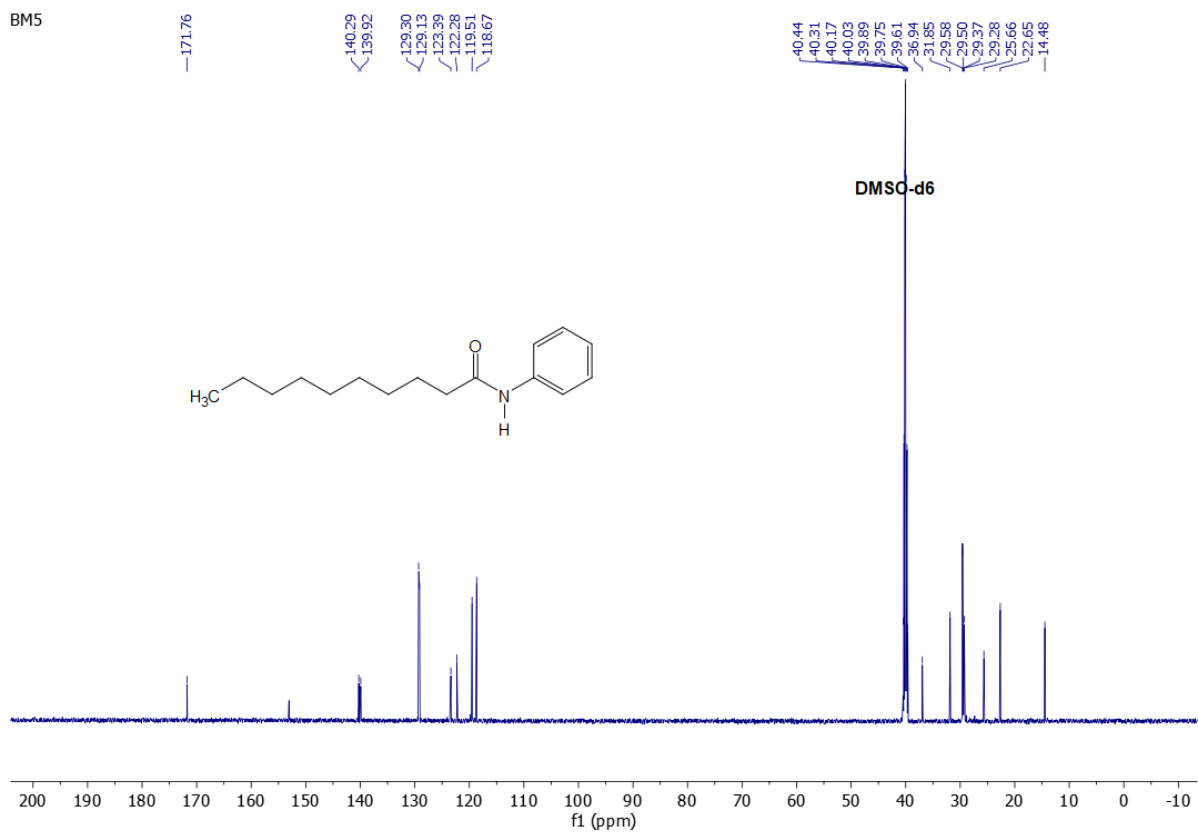
BM07-12



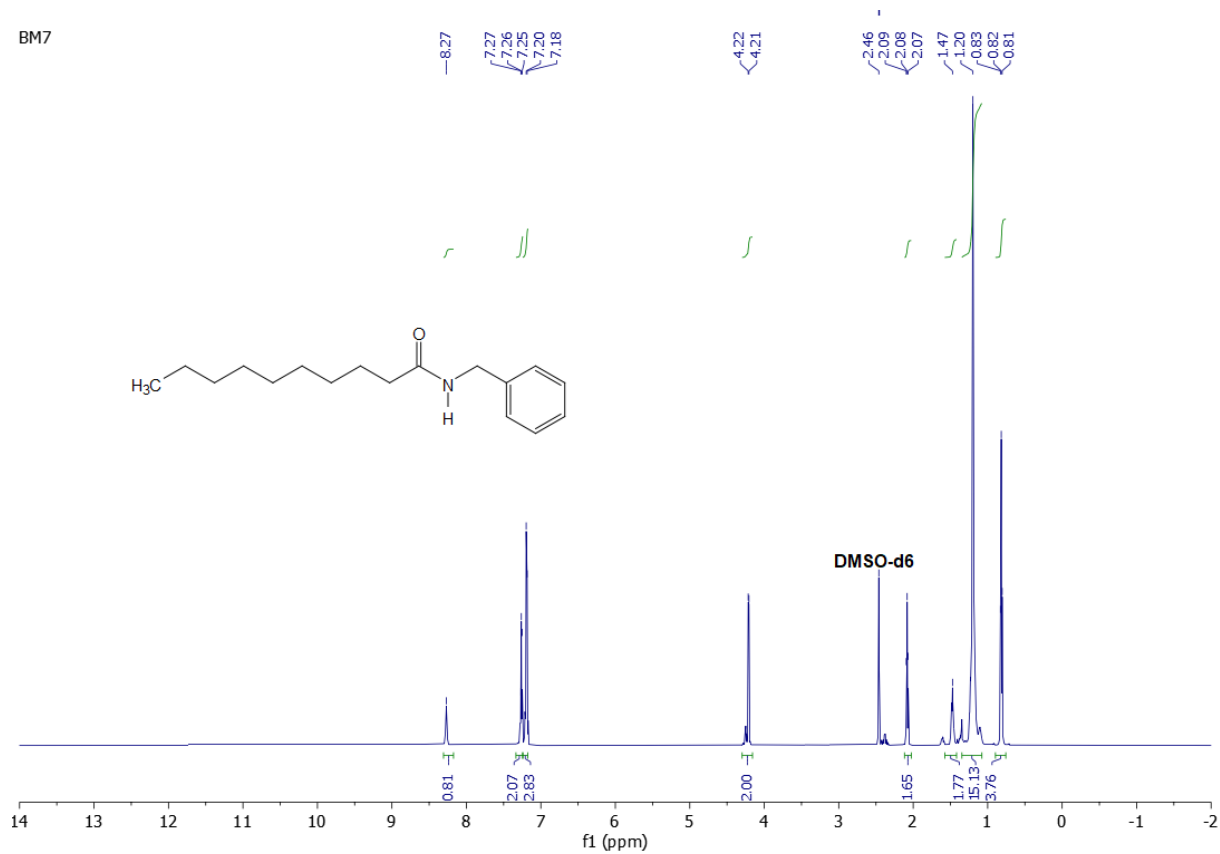
BM5



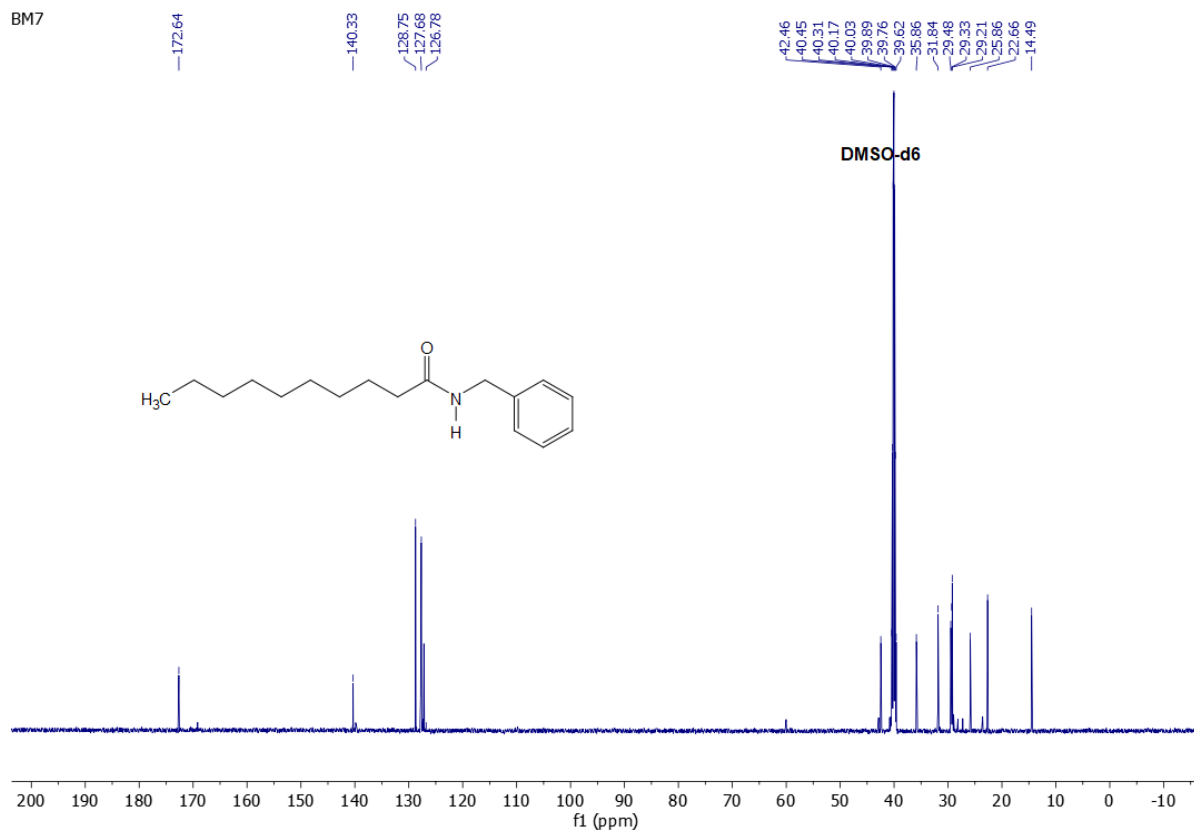
BM5



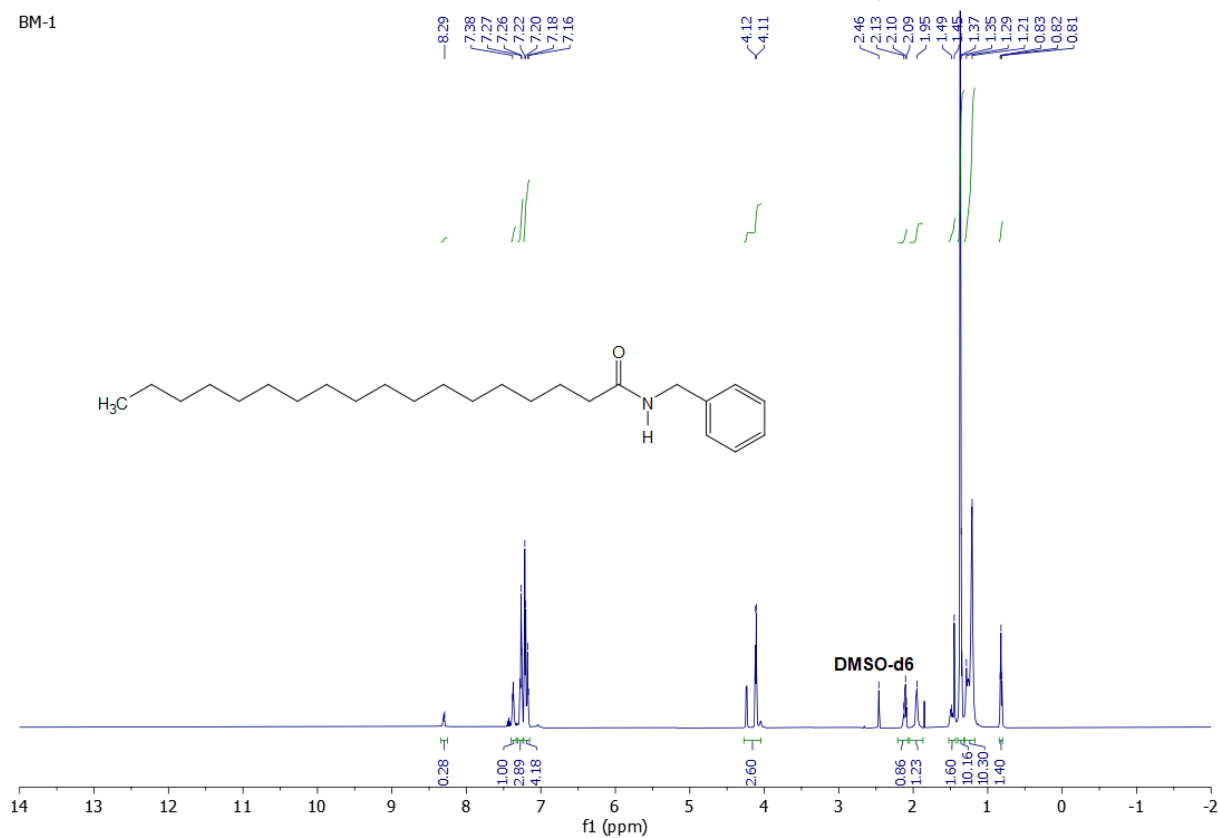
BM7



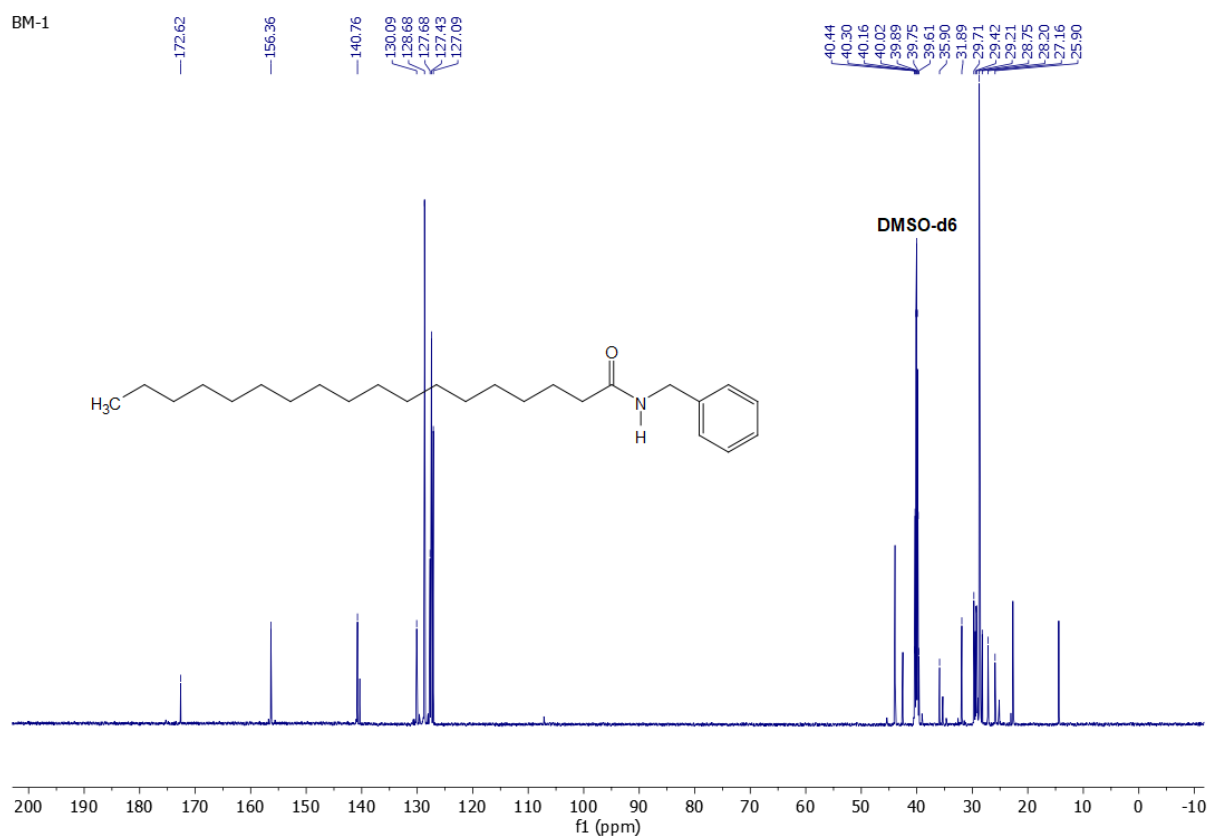
BM7



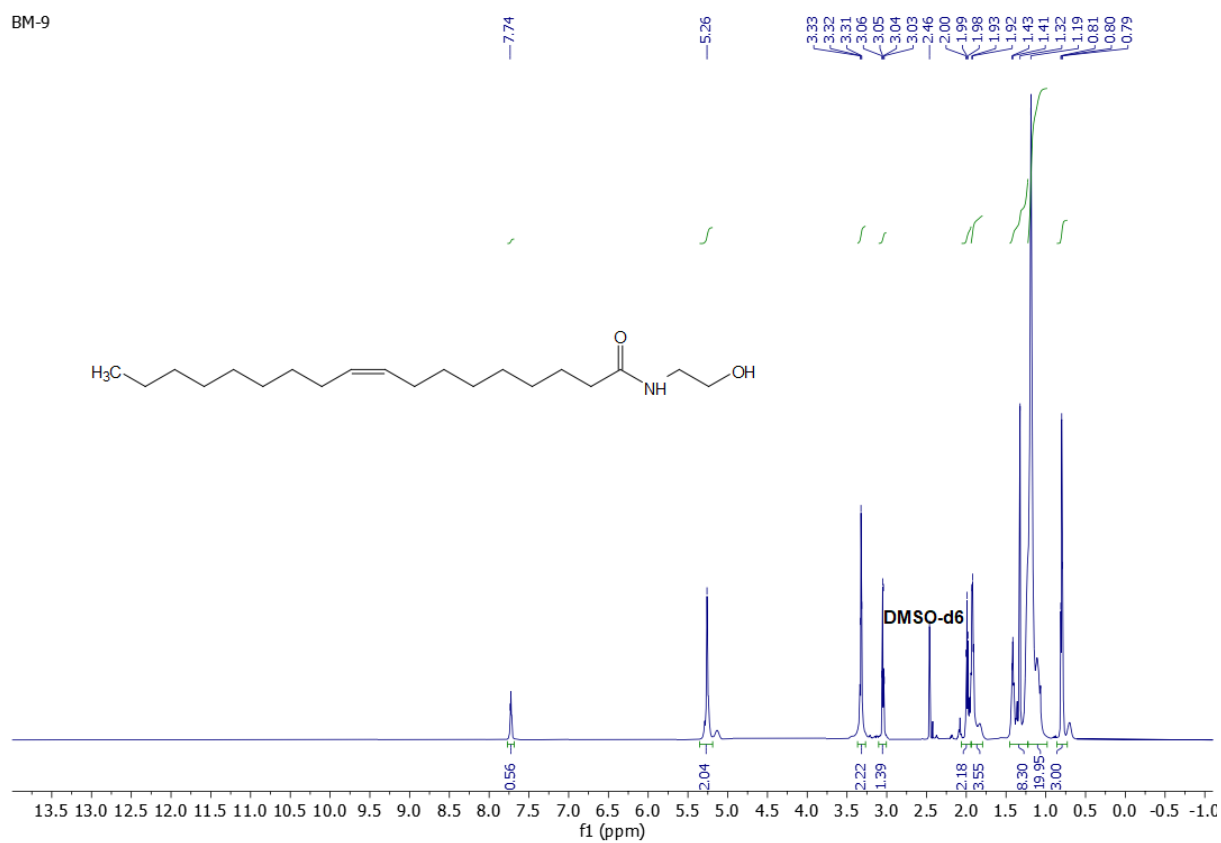
BM-1



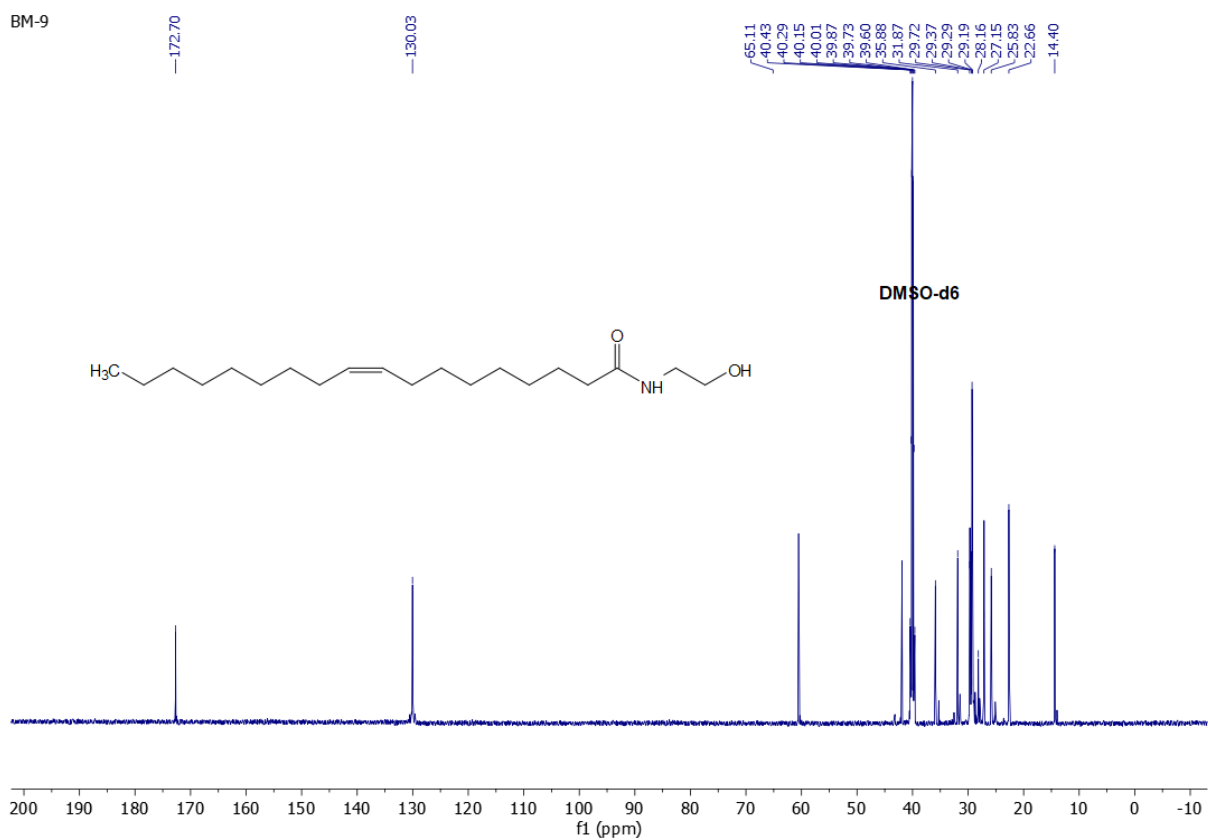
BM-1

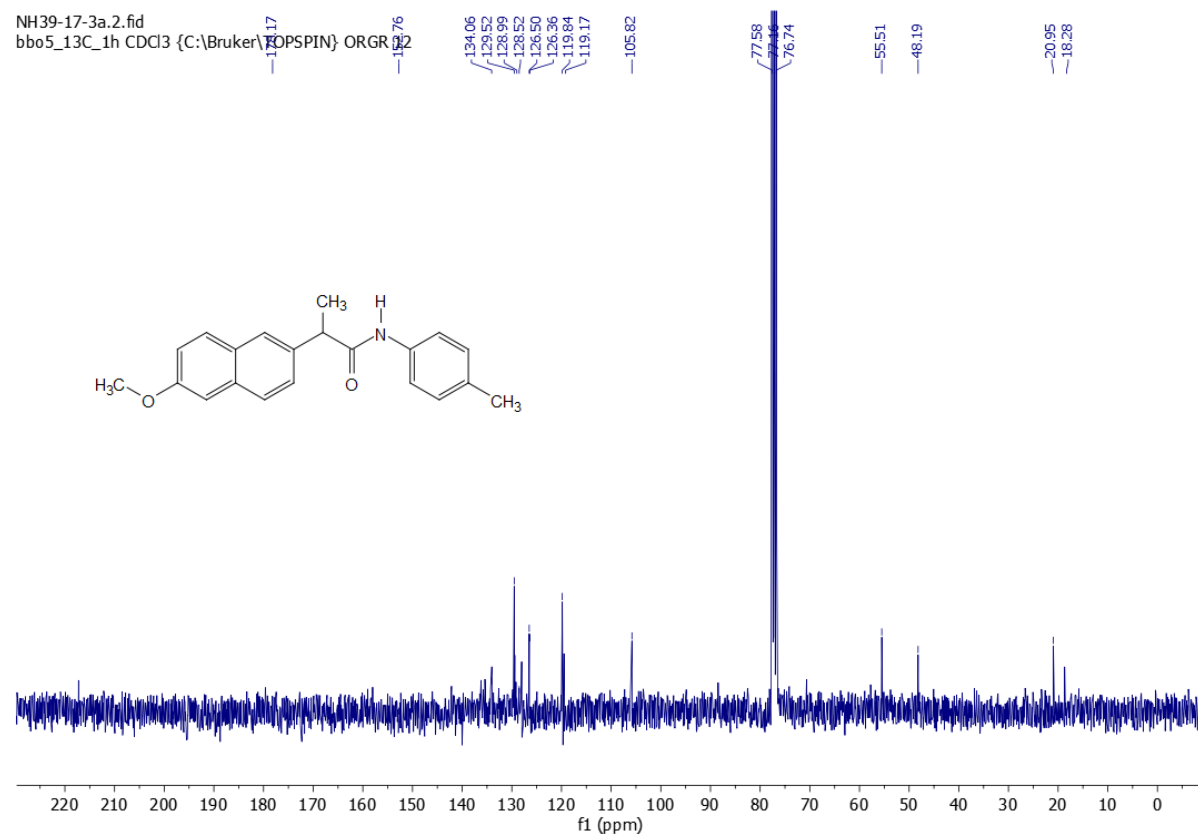
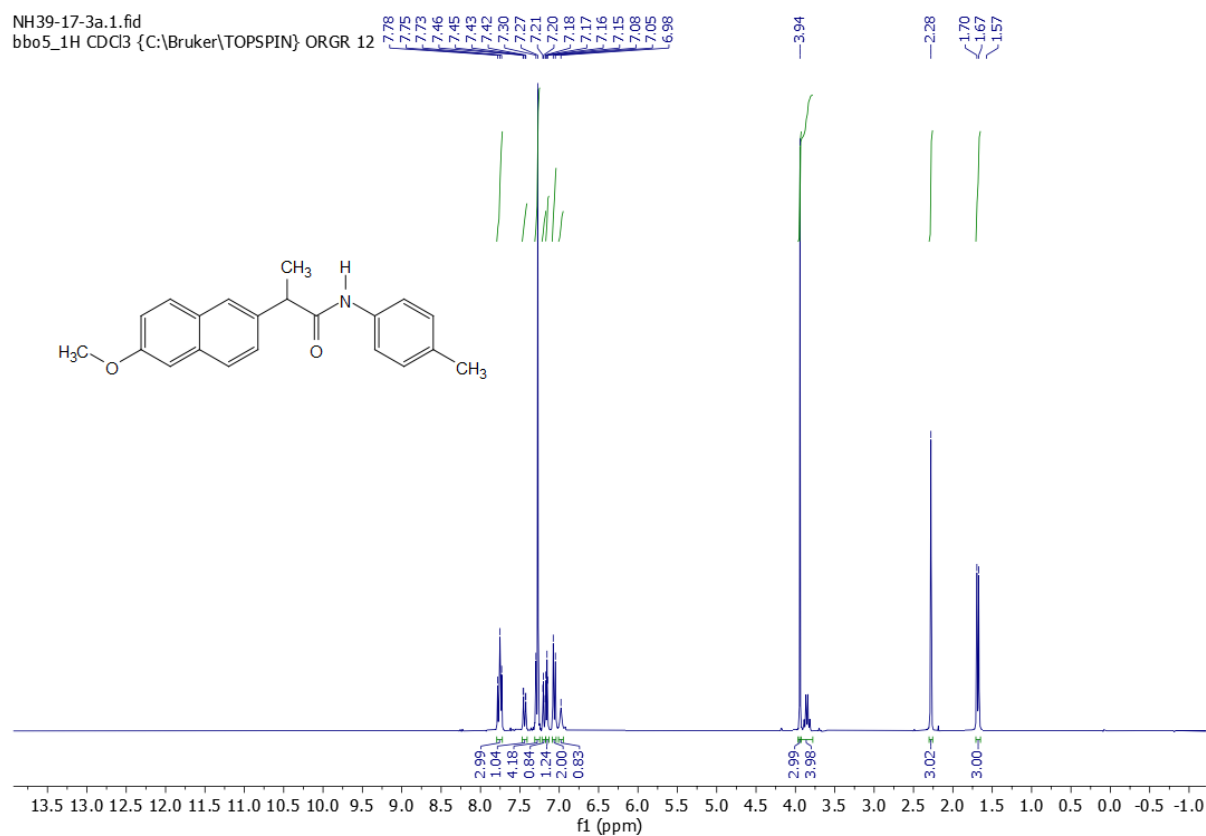


BM-9

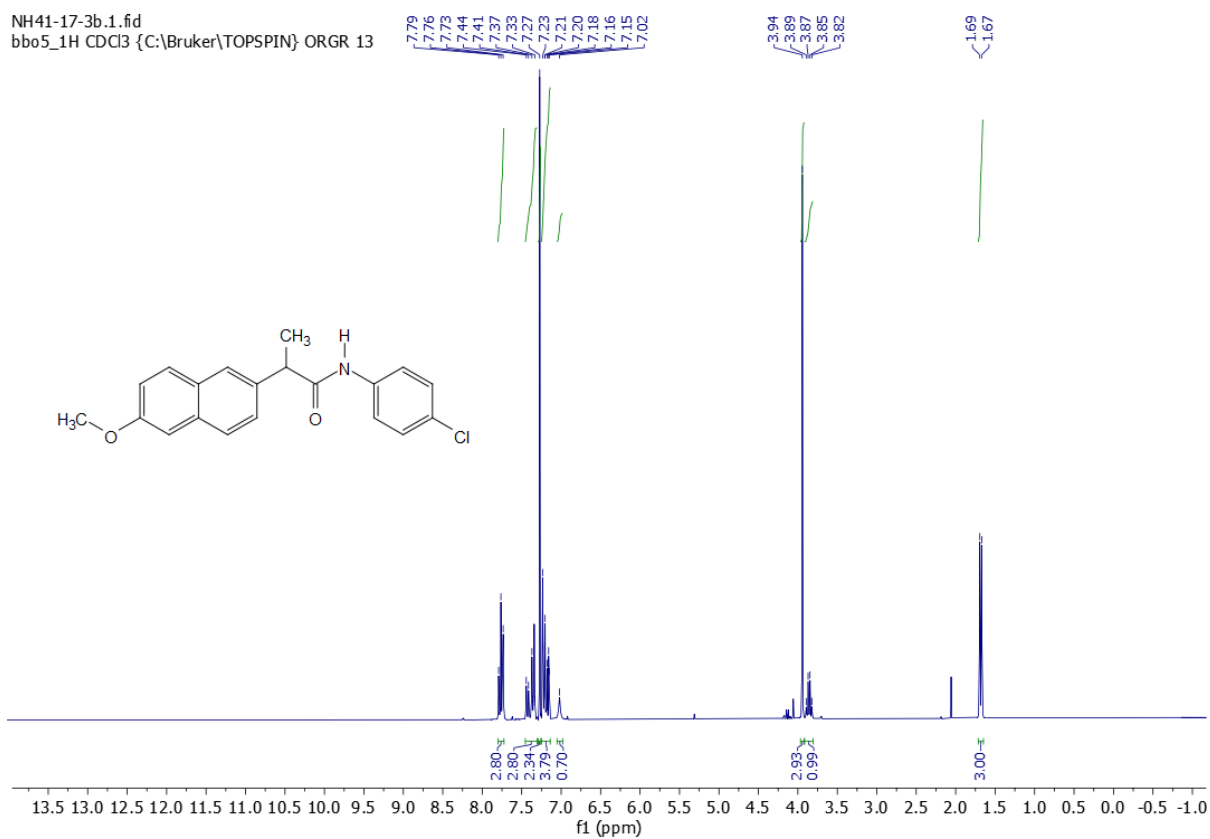


BM-9

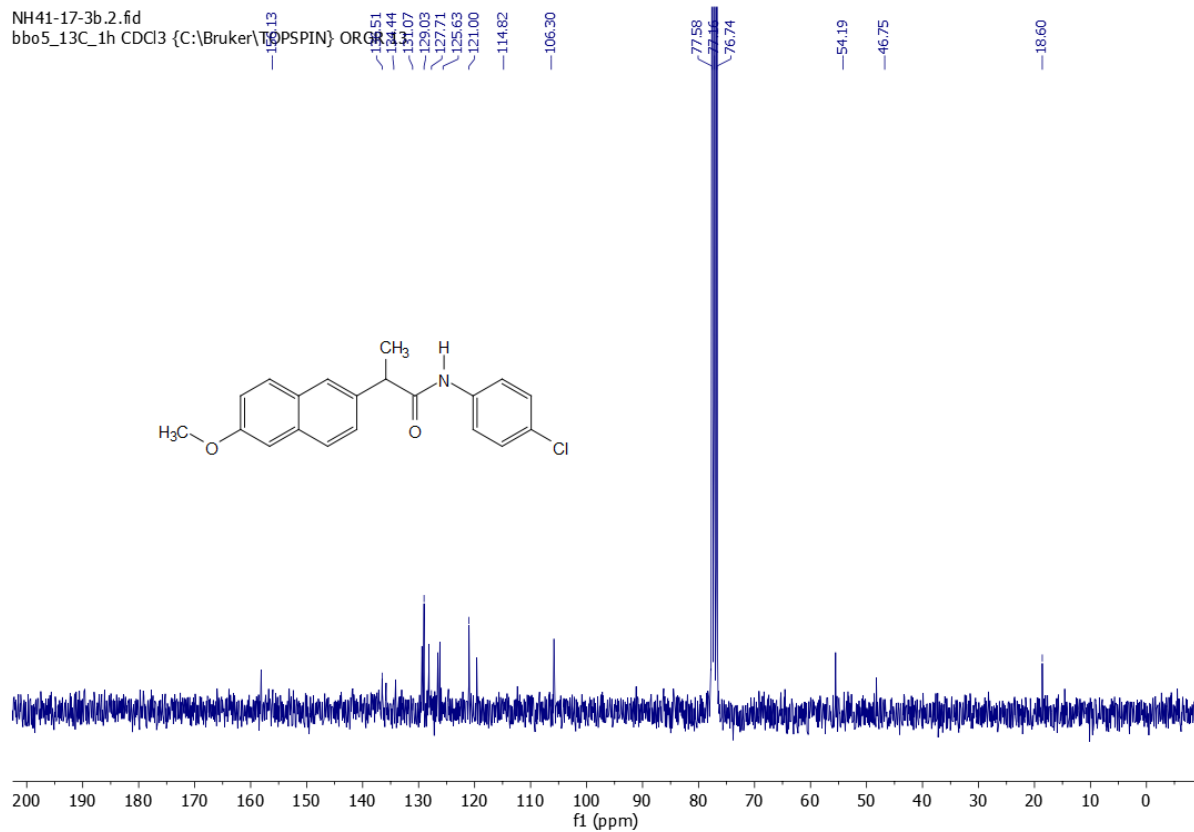




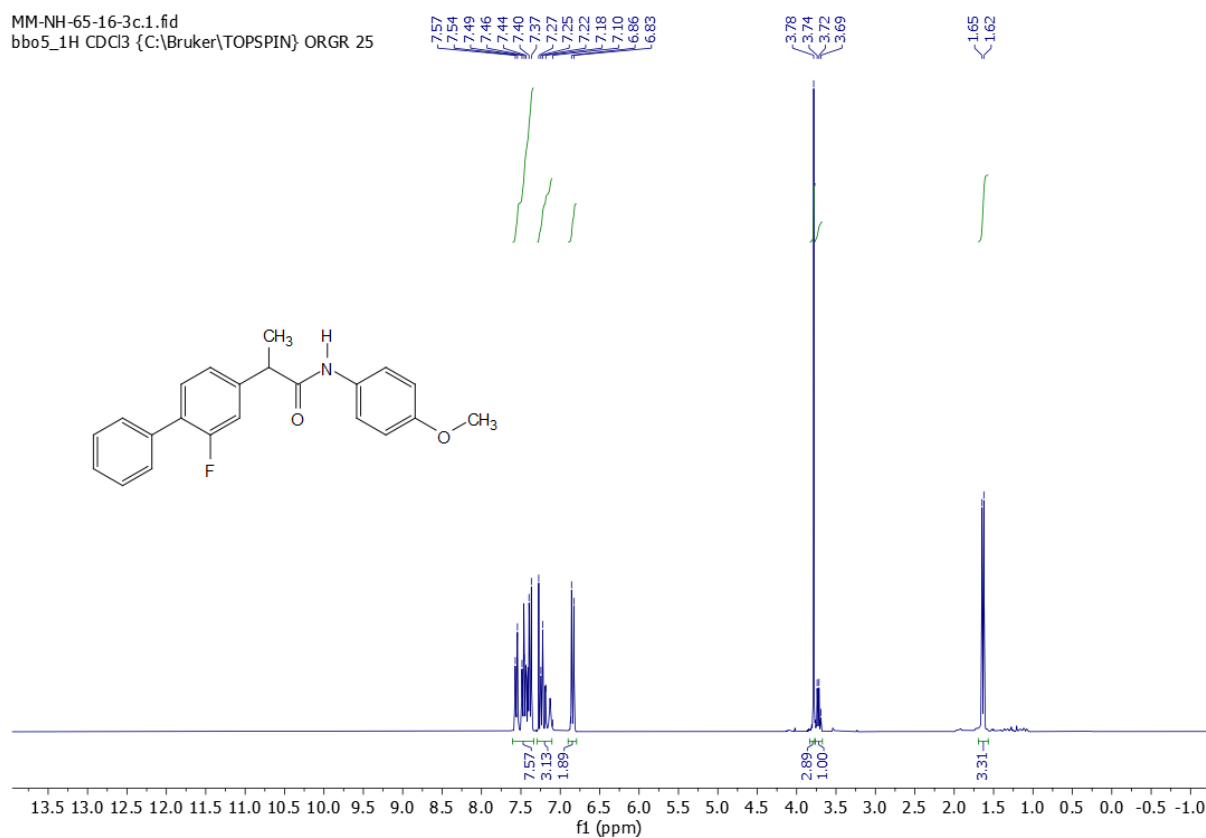
NH41-17-3b.1.fid
bbo5_1H CDCl3 {C:\Bruker\TOPSPIN} ORGR 13



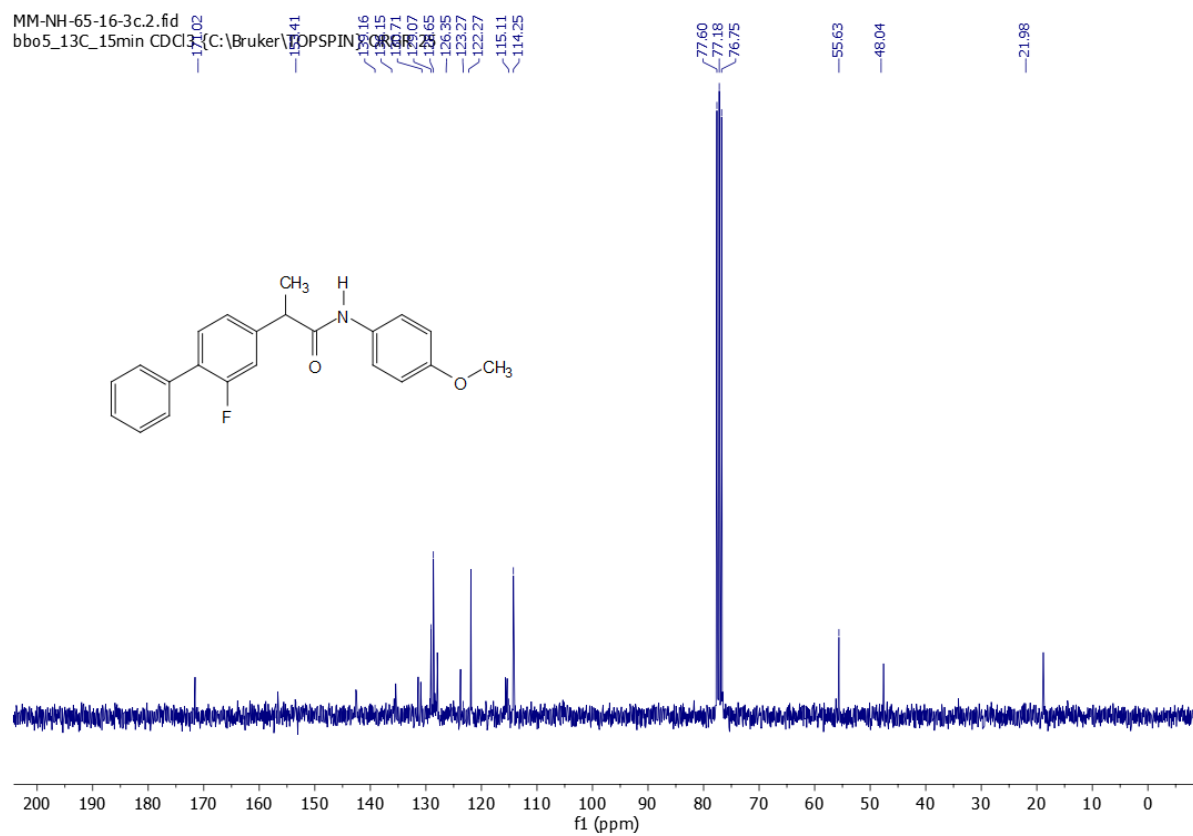
NH41-17-3b.2.fid
bbo5_13C_1h CDCl3 {C:\Bruker\TOPSPIN} ORGR 13

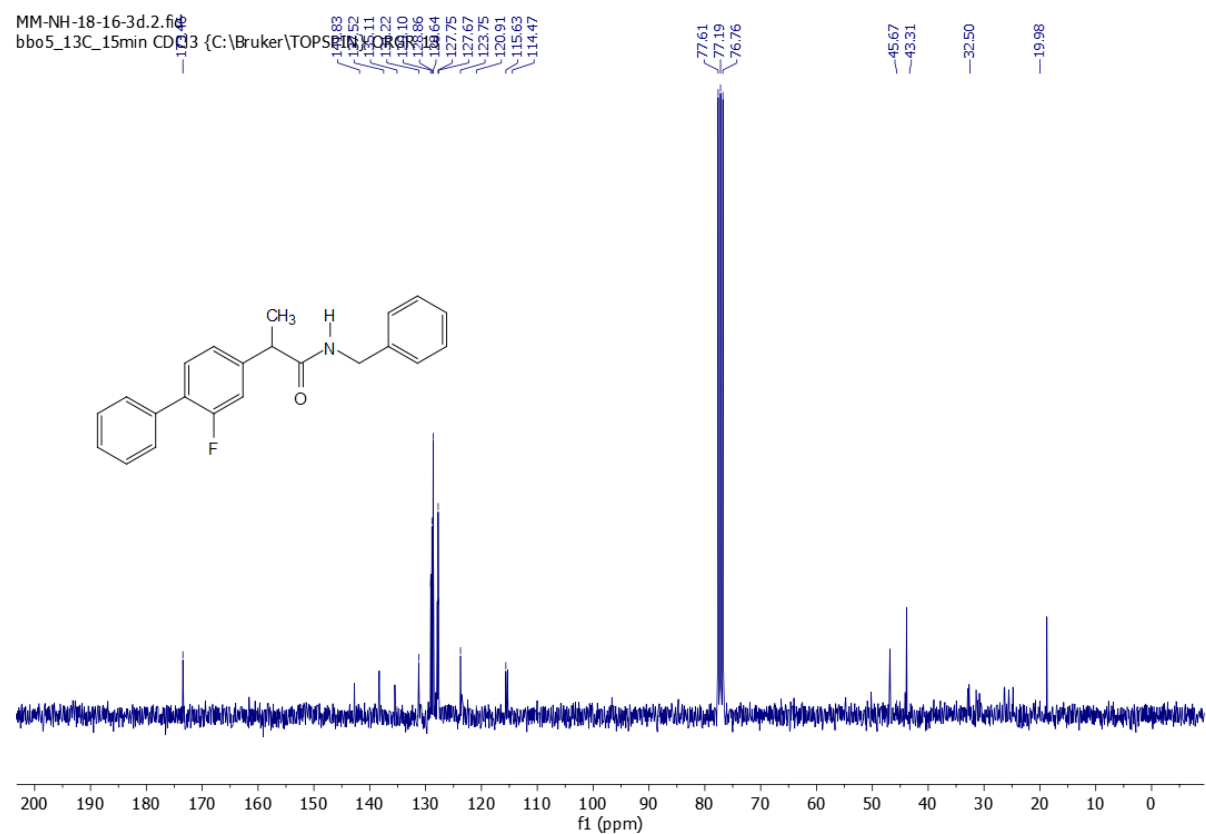
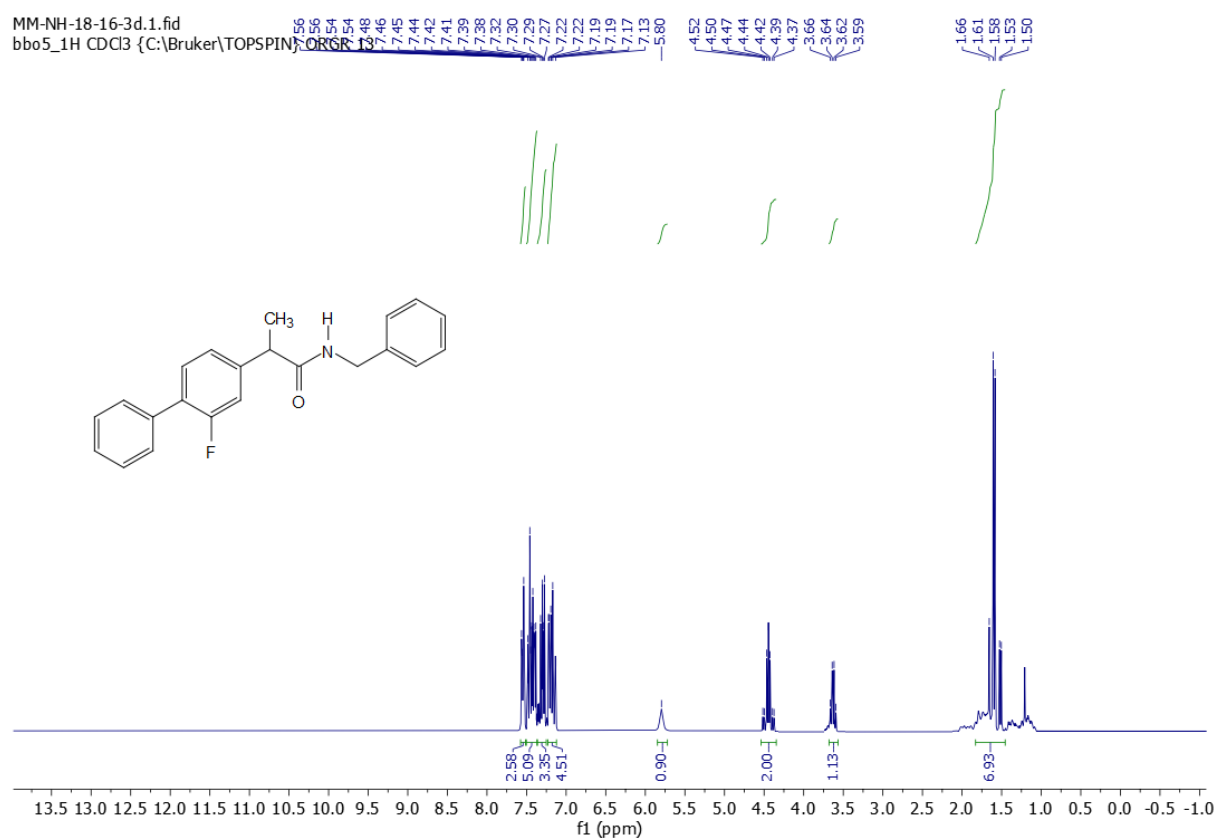


MM-NH-65-16-3c.1.fid
bbo5_1H CDCl3 {C:\Bruker\TOPSPIN} ORGR 25



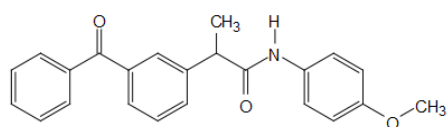
MM-NH-65-16-3c.2.fid
bbo5_13C_15min CDCl3 {C:\Bruker\TOPSPIN} ORGR 25





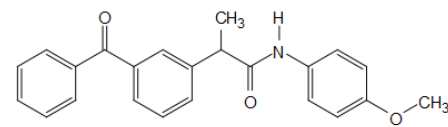
bbo5_1H CDCl3 {C:\Bruker\TOPSPIN}

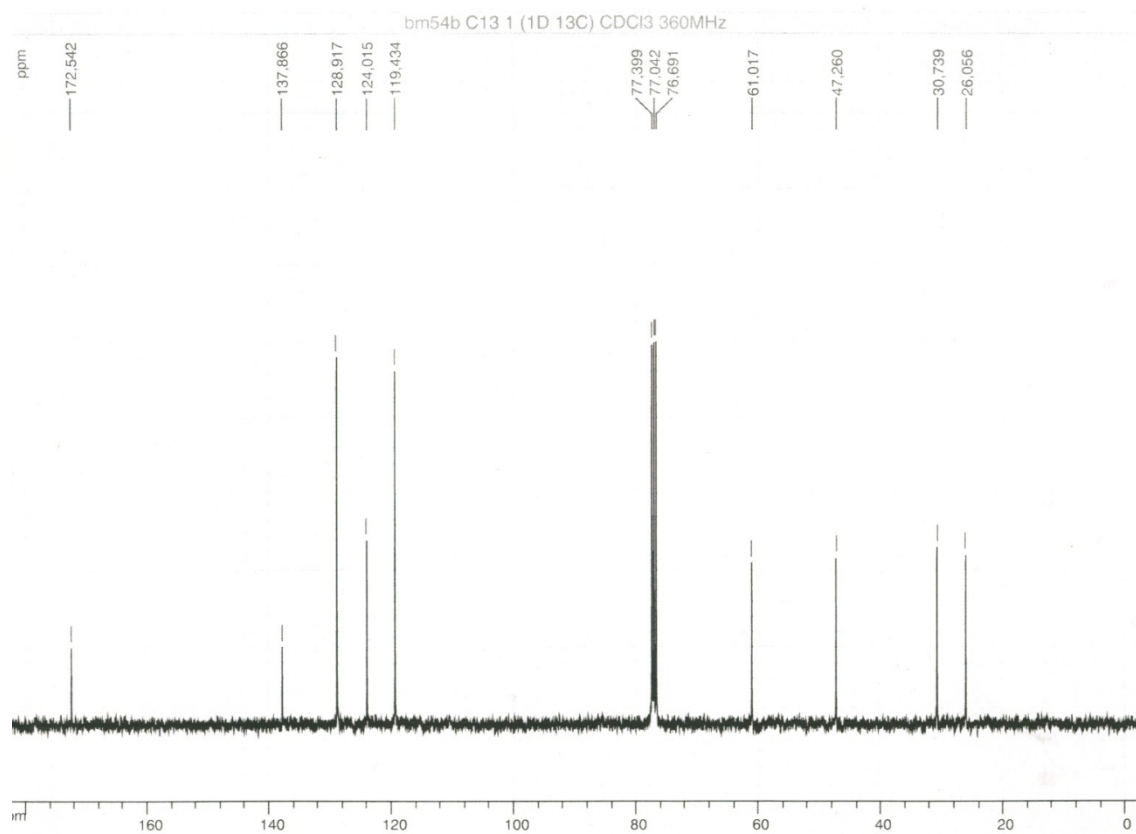
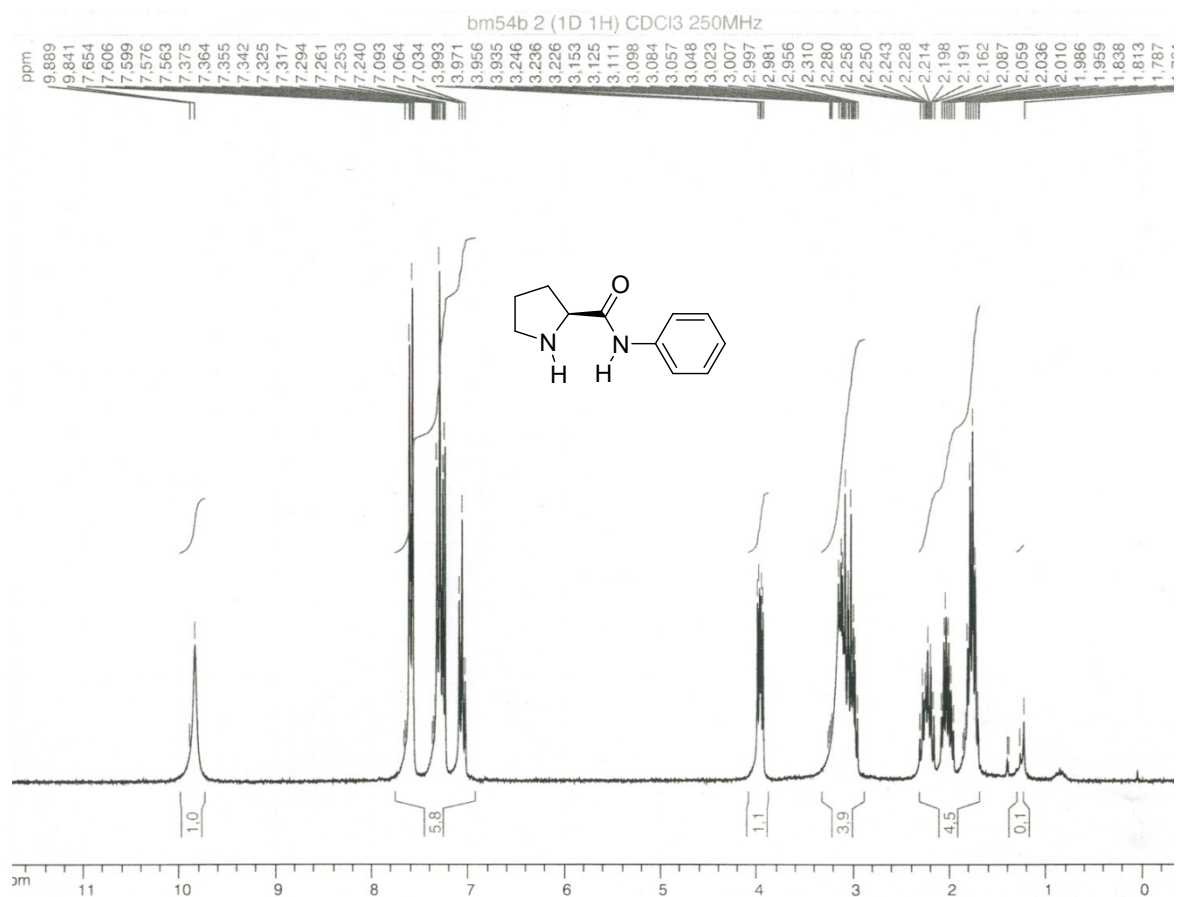
bbo5_1H CDCl3 {C:\Bruker\TOPSPIN} ORGR 26

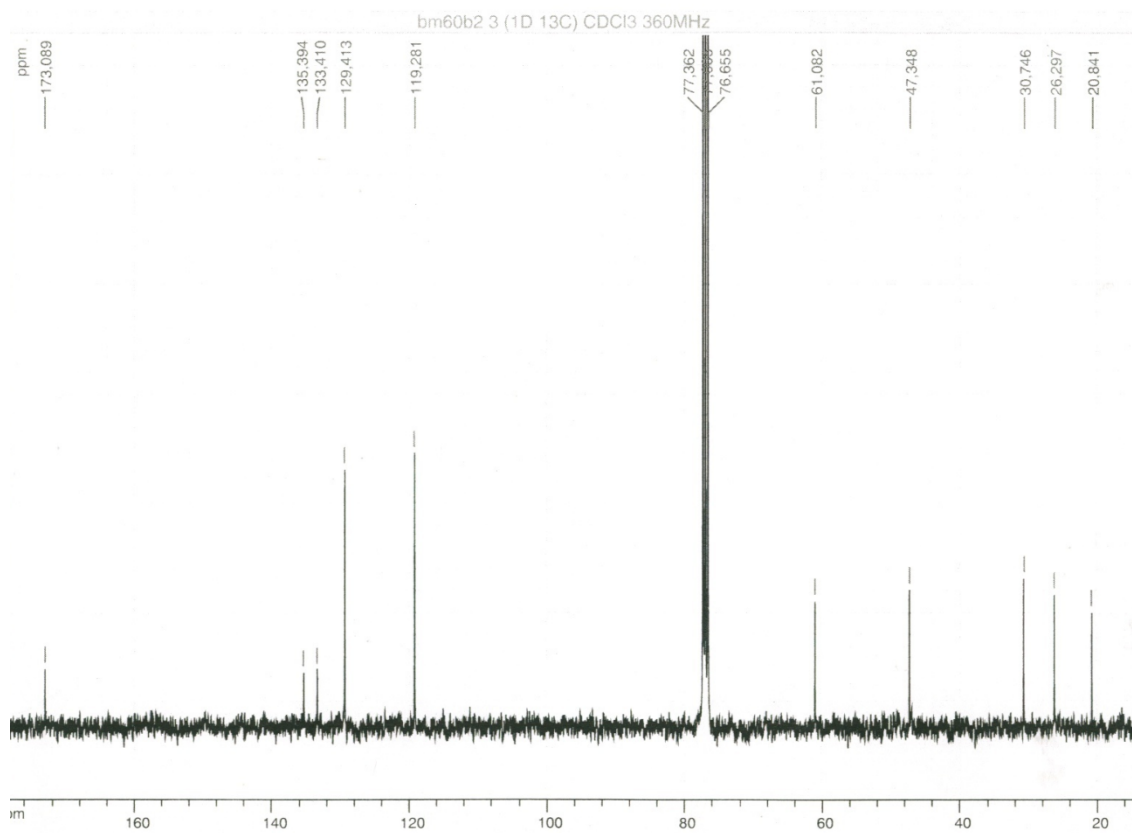
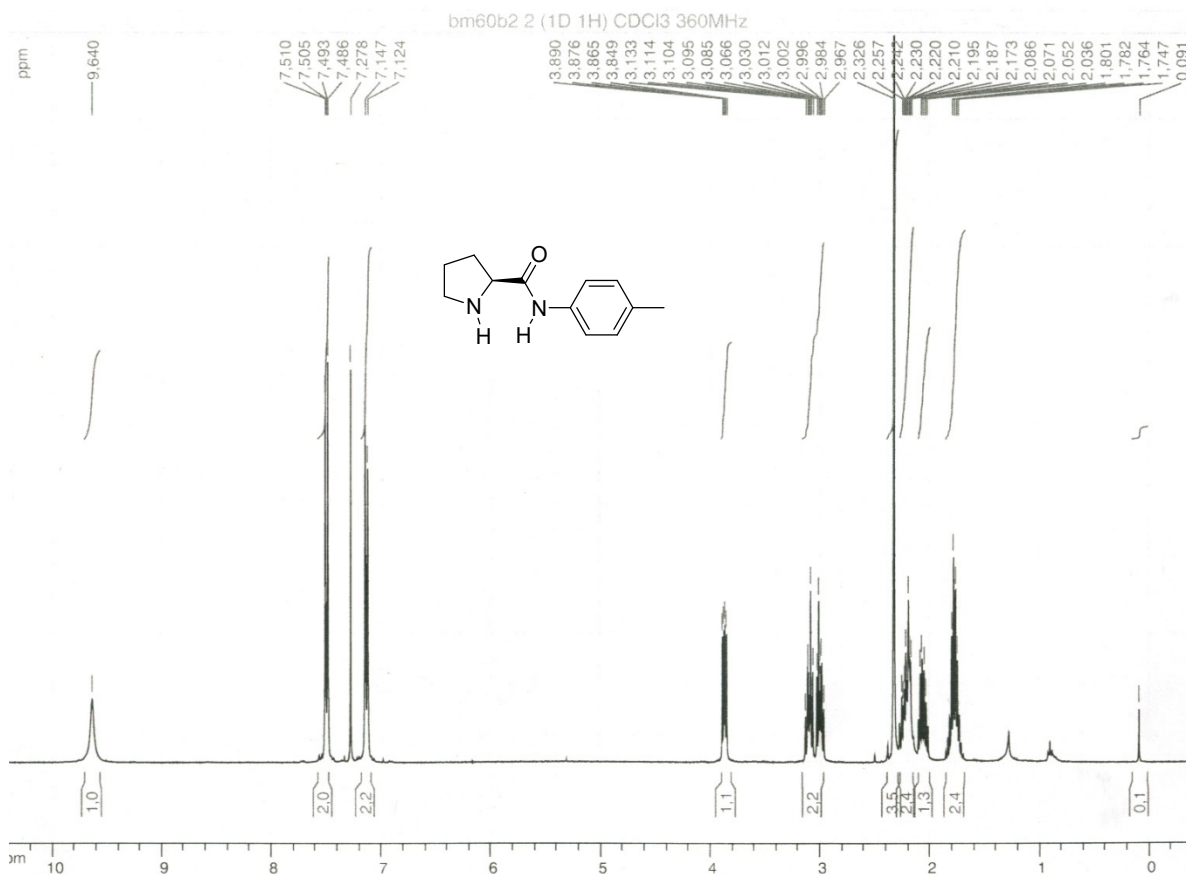


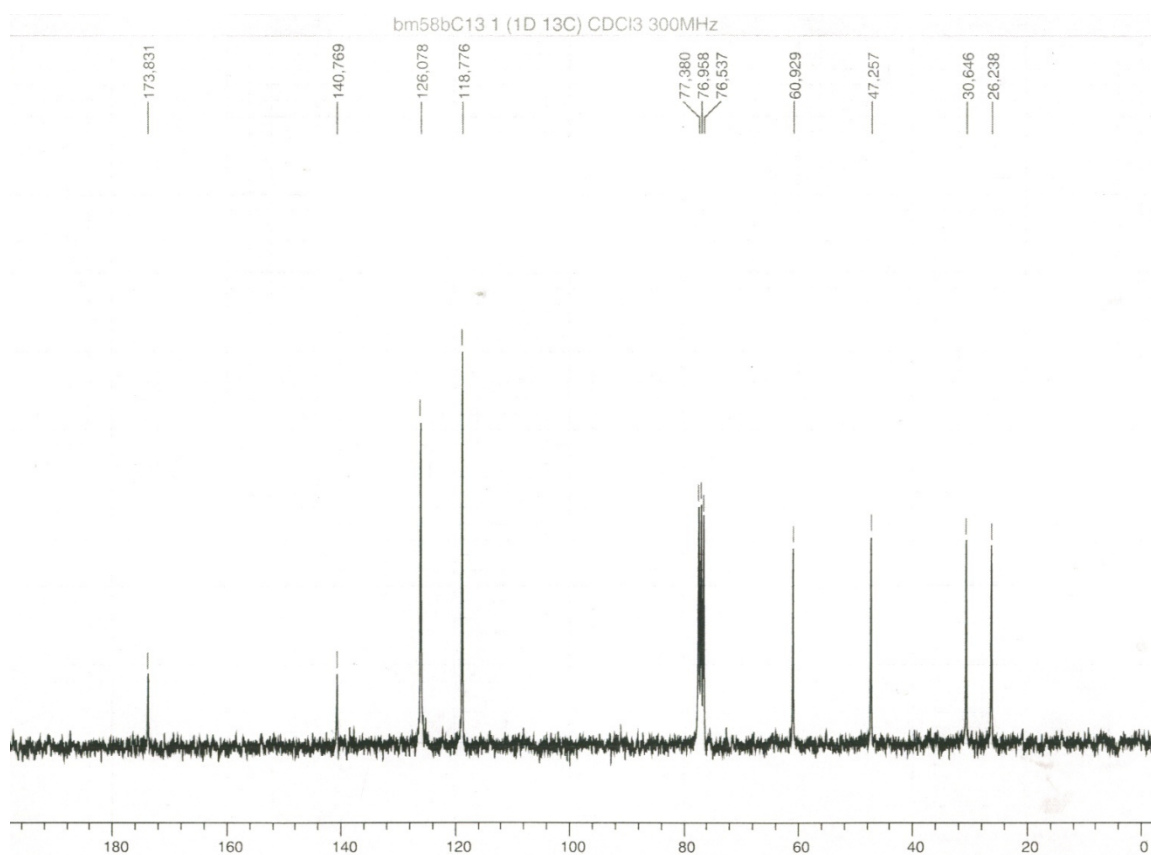
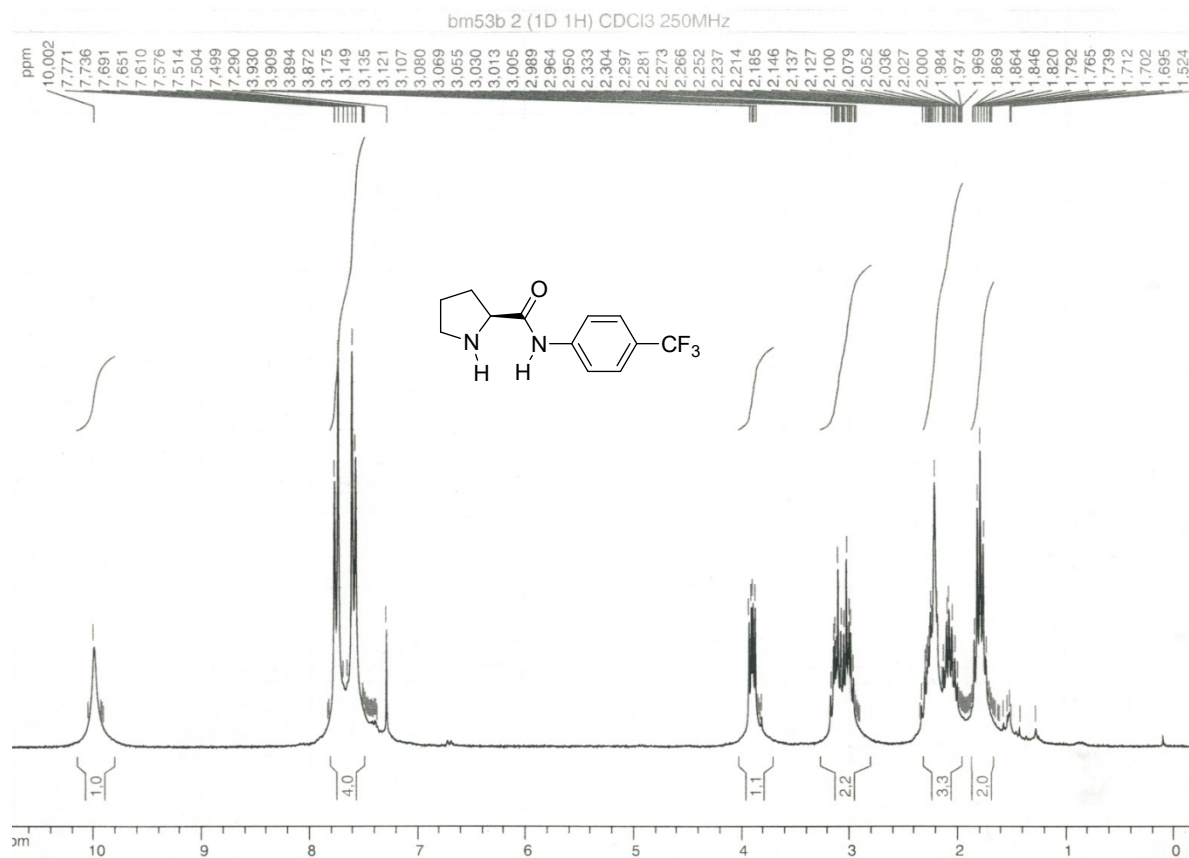
bbo5_13C_15min CDCl₃ {C:\Bruker\}

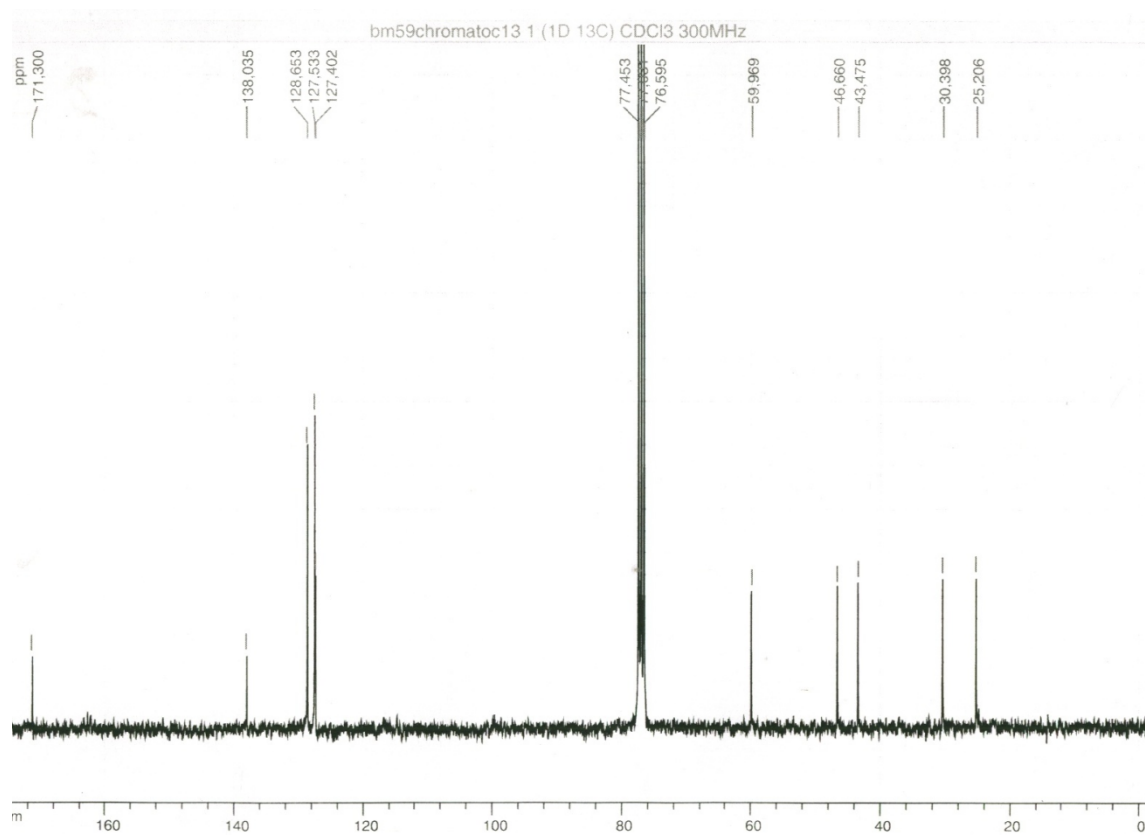
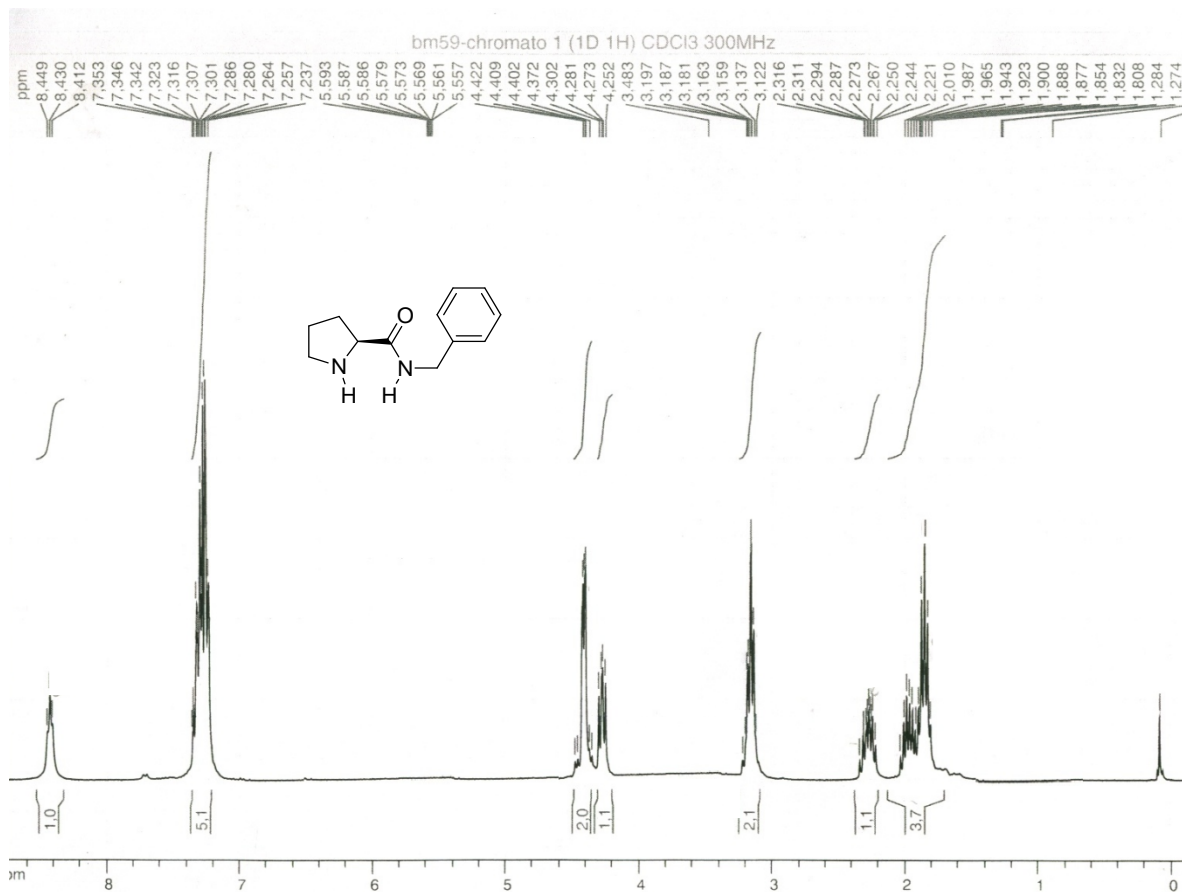
bbo-5-13C_15min CDC (3:1) {C:\Bruker\TOPSPIN\ORIG\26528521.8

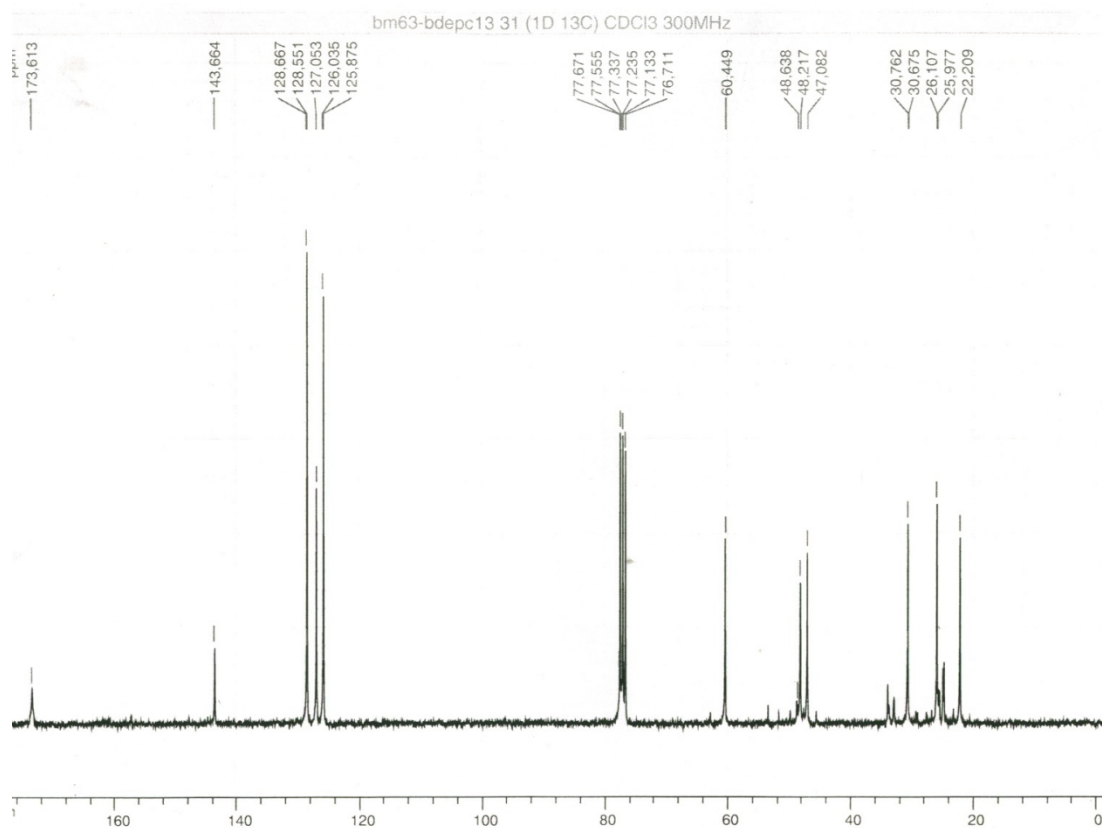
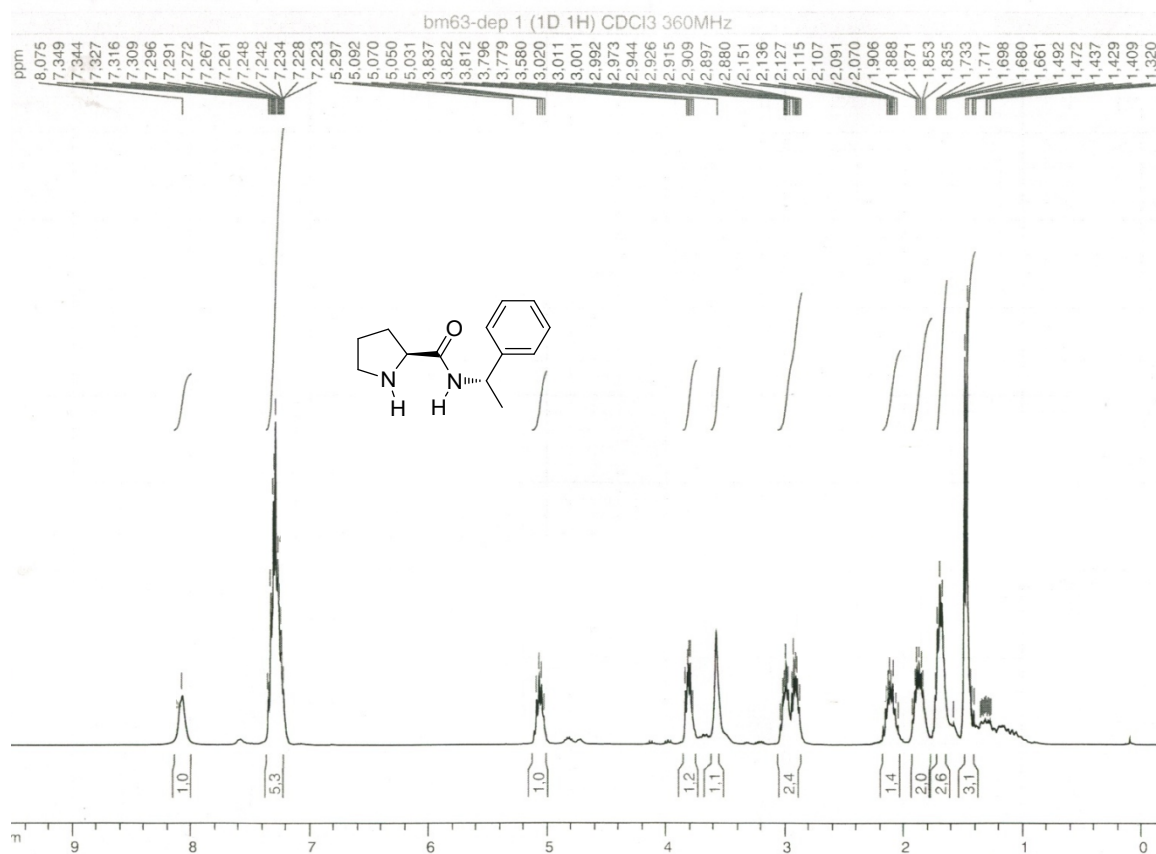


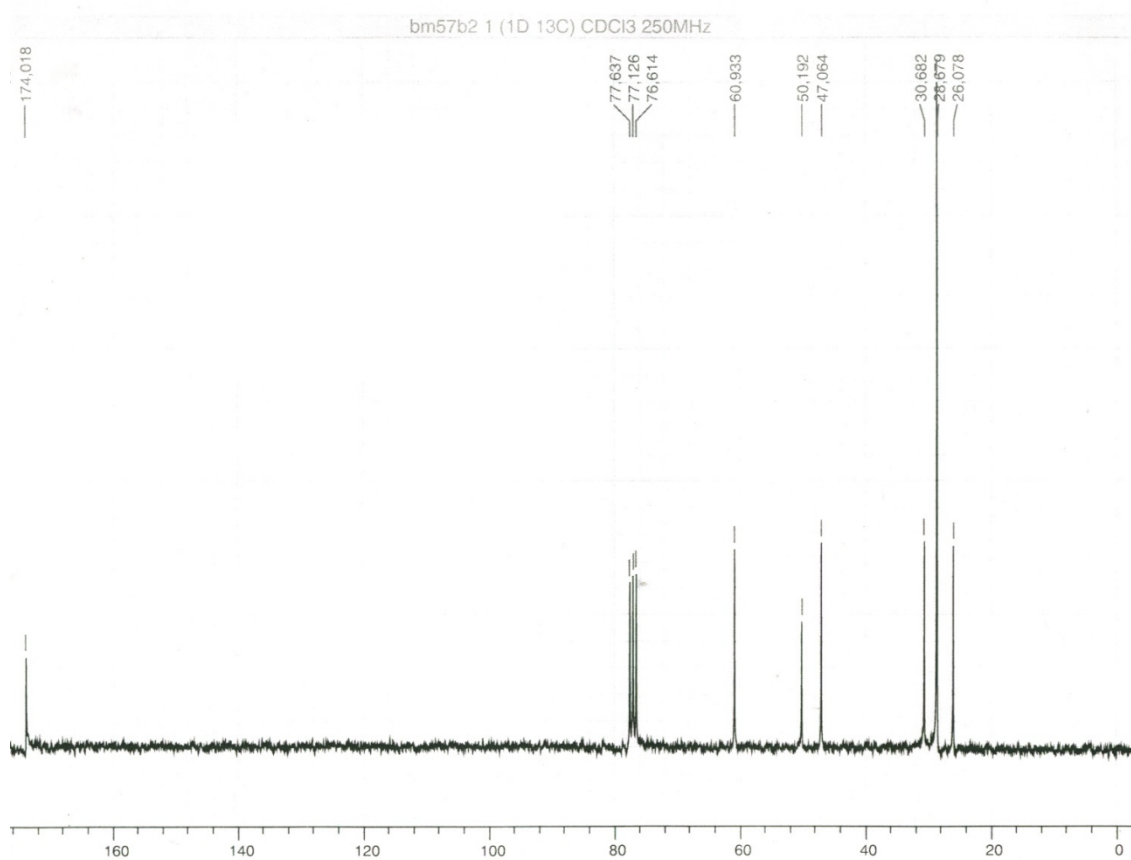
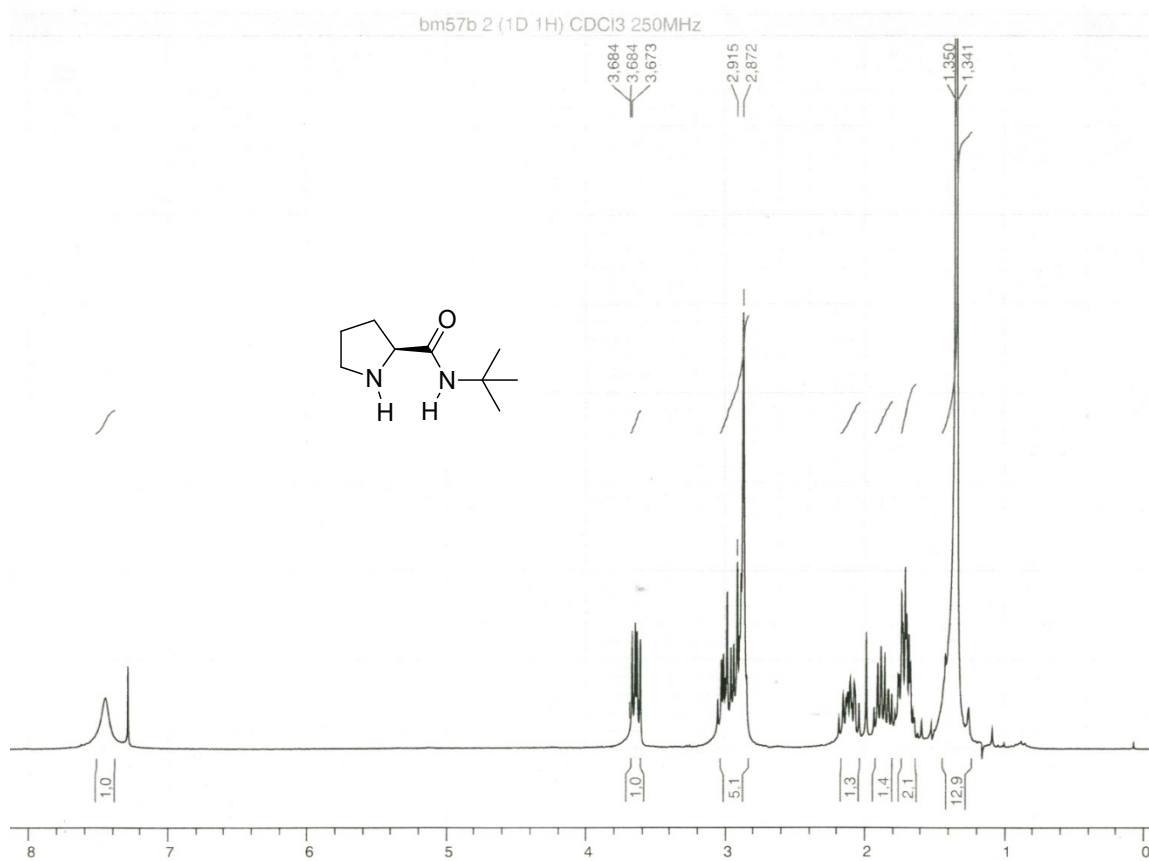












3. References:

1. G. Hong, S. Wu, X. Zhu, D. Mao, L. Wang. *Tetrahedron.*, 2016, **72**, 436–441.
2. R. K. Chang, B. P. Clairmont, S. Lin, A. H. R. MacArthur. *Organometallics.*, 2019, **38**, 4448–4454
3. A. Alalla, M. Merabet-Khelassi, L. Aribi-Zouiouèche, and O. Riant. *Synth. Commun.*, 2014, **44**, 2364–2376,
4. H. Liu, G. Laurenczy, N. Yan, P. J. Dyson. *Chem. Commun.*, 2014, **50**, 341–343.
5. S. Kumar, A. M. Nair, C. M. R. Volla. *Chem. Commun.*, 2021, **57**, 6280–6283.
6. N. Benamara, M. Merabet-Khelassi, L. Aribi-Zouiouèche, and O. Riant. *Chem. Pap.*, 2021, **75**, 4045–4053.
7. B. S. Gore, G. C. Senadi, A. M. Garkhedkar, J. J. Wang. *Adv. Synth. Catal.*, 2017, **359**, 3014–3021.
8. R. Ni, S. Bhandari, P. R. Mitchell, Jr., G. Suarez, N. B. Patel, K. Lamb, K. S. Bisht, D. J. Merkler. *Molecules.*, 2021, **26**, 2543.
9. D. Charles, J. Irving, T. Floreancig, S. Laulhé. *ACS Omega.*, 2020, **25**, 15734-15745
10. E. Brenna, V. De Fabritiis, F. Parmeggiani, F. Tentori, D. Tessaro. *ACS Sustainable Chem. Eng.*, 2023, **11**, 2764–2772.
11. M. H. Helal, S. Y. Abbas, M. A. Salem, A. A. Farag, Y. A. Ammar. *Medicinal Chemistry Research.*, 2013, **22**, 5598-5609.
12. <https://patents.google.com/patent/CN113651768A/en>
13. <https://patents.google.com/patent/WO2007095383A2>
14. G. D. Yadav, S. Singh. *Tetrahedron. Asymmetry.*, 2015, **26**, 1156–1166.
15. S. Zeror, J. Collin, J.-C. Fiaud, L. Aribi-Zouiouèche, *Adv. Synth. Catal.*, 2008, **350**, 197-204.
16. M. Boukachabia, S. Zeror, J. Collin, J.-C. Fiaud, L.-A. Zouiouèche, *Tetrahedron Letters.*, 2011, **52**, 1485–1489.