Supporting Information
for

One-pot sequential synthesis of isocyanates and urea compounds via a microwave-assisted Staudinger–aza-Wittig reaction

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Detailed analytical data of the prepared compounds and a collection of NMR spectra

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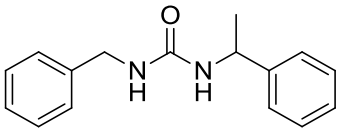
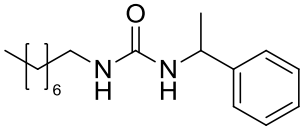
1. Analytical Data

***N*-benzyl-*N'*-(1-phenylethyl)urea 10 (Table 3, entry 1)** [1]**:**



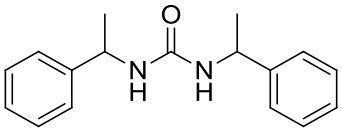
Yield 79 mg (98%); a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.13 (m, 10H), 4.76 (q, *J*=6.00 Hz, 1H), 4.33 (s, 2H), 1.46 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.13, 144.16, 139.07, 129.09, 128.91, 127.68, 127.60, 127.53, 126.16, 50.83, 44.69, 23.71 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{16}H_{18}N_2O$ $[M + H]^+$: 255.1419, found: 255.1416.

***N*-Octyl-*N'*-(1-phenylethyl)urea 11 (Table 3, entry 2):**



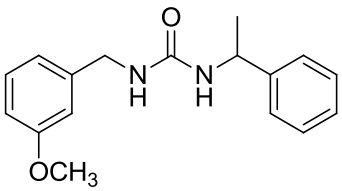
Yield 79 mg (90%); a yellowish oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.26 (m, 5H), 4.71 (q, *J*=6 Hz, 1H), 3.11 (t, *J*=6 Hz, 2H), 1.48 (m, 2H), 1.32 – 1.09 (o.s., 10H), 0.87 (o.s., 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.35, 137.62, 129.26, 128.25, 127.75, 45.25, 41.53, 32.09, 29.84, 29.49, 29.48, 27.00, 22.97, 14.44 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{17}H_{28}N_2O$ $[M + H]^+$: 277.2201, found: 277.2198.

***N,N'*-bis(1-phenylethyl)urea 12 (Table 3, entry 3)** [2]**:**



Yield 78 mg (92%); a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.09 (m, 10H), 4.73 (q, *J*=6 Hz, 2H), 1.34 (d, *J*=6 Hz, 6H) ppm. ^{13}C NMR (300 MHz, $CDCl_3$) δ 157.57, 144.13, 129.02, 127.57, 126.05, 50.74, 23.69 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{17}H_{20}N_2O$ $[M + H]^+$: 269.1576, found: 269.1569.

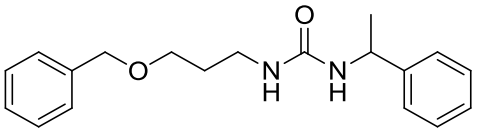
***N*-(3-methoxybenzyl)-*N'*-(1-phenylethyl)urea 13 (Table 3, entry 4):**



Yield 87 mg (97%); a withe solid. ^{1}H NMR (75 MHz, CD_3OD) δ 7.34 – 7.13 (m, 5H), 6.87 – 6.72 (m, 4H), 4.84 (q, *J*=6 Hz, 1H), 4.27 (s, 2H), 3.74 (s, 3H), 1.42 (d, *J*=9 Hz, 3H) ppm. ^{13}C NMR (75

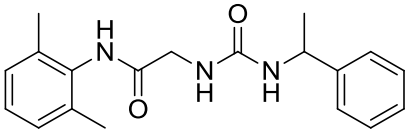
MHz, CD_3OD) δ 161.33, 160.30, 146.43, 142.89, 130.45, 129.47, 127.85, 126.81, 120.30, 113.52, 113.46, 64.30, 55.56, 44.56, 23.57 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{17}H_{20}N_2O_2$ $[M + H]^+$: 285.1524, found: 285.1527.

***N*-(3-(benzyloxy)propyl)-*N'*-(1-phenylethyl)urea 14 (Table 3, entry 5):**



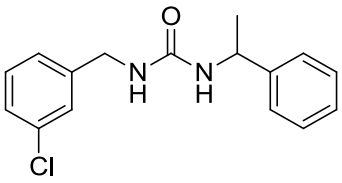
Yield 96 mg (97%); a yellowish oil. ^1H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.15 (m, 10H), 4.50 (q, 1H), 4.38 (s, 2H), 3.48 (t, 2H), 3.26 (t, 2H), 1.72 (m, 2H), 1.38 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.93, 143.28, 138.14, 128.98, 128.73, 128.09, 127.72, 126.02, 73.31, 63.97, 51.33, 39.04, 29.51, 23.46 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{19}H_{24}N_2O_2$ $[M + H]^+$: 313.1838, found: 313.1836.

***N*-(2,6-dimethylphenyl)-2-(3-(1-phenylethyl)ureido)acetamide 15 (Table 3, entry 6):**



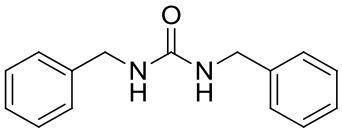
Yield 100 mg (98%); a white solid. ^1H NMR (300 MHz, CD_3OD) δ 7.47 – 7.07 (m, 8H), 4.45 (q, *J*=6 Hz, 1H), 4.24 (s, 2H), 2.16 (s, 6H), 1.63 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.60, 157.85, 137.95, 136.83, 129.98, 129.30, 128.08, 127.28, 126.21, 52.12, 47.09, 20.68, 18.17 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{19}H_{23}N_3O_2$ $[M + H]^+$: 326.1790, found: 326.1793.

***N*-(3-chlorobenzyl)-*N'*-(1-phenylethyl)urea 16 (Table 3, entry 7):**



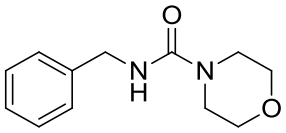
Yield 86 mg (94%); a withe solid. ^1H NMR (300 MHz, $CDCl_3$) δ 7.32 – 6.97 (m, 9H), 4.76 (q, *J*=6 Hz, 1H), 3.76 (s, 2H), 1.41 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.97, 140.16, 134.72, 134.70, 130.20, 129.13, 127.88, 127.26, 125.94, 125.38, 64.00, 43.98, 23.59 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{16}H_{17}ClN_2O$ $[M + H]^+$: 289.1029, found: 289.1033.

***N,N'*-bis-benzylurea 27 (Table 4, entry 1)** [1]:



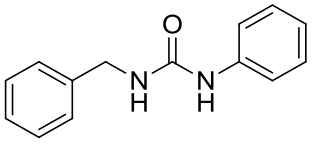
Yield 74 mg (98%); a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.23 (m, 10H), 4.38 (s, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.78, 139.06, 128.95, 127.67, 127.65, 44.82 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{15}H_{16}N_2O$ $[M + H]^+$: 241.1262, found: 241.1259.

***N*-benzylmorpholine-4 carboxamide 28 (Table 4, entry 2)** [3]**:**



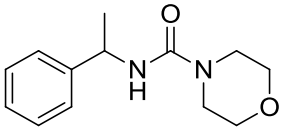
Yield 68 mg (98%); a yellowish solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 7.20 (m, 5H), 4.38 (s, 2H), 3.63 (t, *J*=6 Hz, 4H), 3.30 (t, *J*=6 Hz, 4H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.75, 139.27, 129.04, 120.16, 127.83, 66.68, 45.38, 44.60 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{12}H_{16}N_2O_2$ $[M + H]^+$: 221.1212, found: 221.1219.

N-benzyl-*N'*-phenylurea **29 (Table 4, entry 3)** [4]**:**



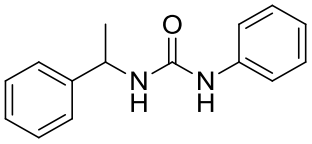
Yield 69 mg (98%); a yellowish solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.13 (m, 10H), 4.44 (s, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.72, 129.72, 129.20, 129.04, 127.81, 127.76, 127.62, 124.74, 121.97, 44.64 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{14}H_{14}N_2O$ $[M + H]^+$: 227.1106, found: 227.1100.

***N*-(1-phenylethyl)morpholine-4-carboxamide 30 (Table 4, entry 5)** [3]:



Yield 69 mg (94%); a yellowish solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 – 7.17 (m, 5H), 4.95 (q, *J*=6 Hz, 1H), 3.62 (t, *J*=6 Hz, 4H), 3.27 (t, *J*=6 Hz, 4H), 1.43 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.14, 144.36, 144.36, 128.99, 127.59, 126.45, 66.71, 50.51, 44.45, 22.84 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{13}H_{18}N_2O_2$ $[M + H]^+$: 235.1368, found: 235.1372.

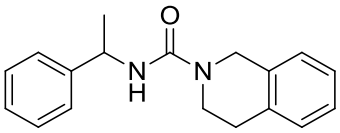
***N*-(1-phenylethyl)-*N'*-phenylurea 31 (Table 4, entry 6):**



Yield 75 mg (98%); a yellowish solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.06 (m, 10H), 4.95 (q, *J*=6 Hz, 1H), 1.48 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.86, 144.06, 138.61,

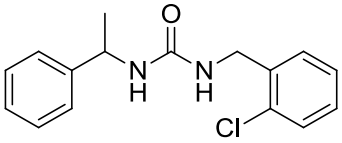
129.51, 129.06, 127.66, 126.23, 124.07, 121.12, 50.41, 23.28 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{15}H_{16}N_2O$ $[M + H]^+$: 241.1262, found: 241.1256.

***N*-(1-phenylethyl)-3,4-dihydroisoquinoline-2-carboxamide 32 (Table 4, entry 7):**



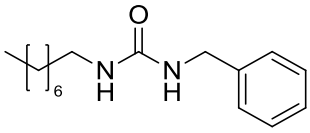
Yield 87 mg (98%); a withe solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 6.99 (m, 9H), 4.99 (q, *J*=6 Hz, 1H), 4.44 (s, 2H), 3.52 (t, *J*=6 Hz, 2H), 2.76 (t, *J*=6 Hz, 2H), 1.43 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.03, 144.71, 135.35, 133.59, 128.86, 128.64, 127.38, 126.92, 126.64, 126.60, 126.44, 50.38, 45.77, 41.46, 29.31, 22.92 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{18}H_{20}N_2O$ $[M + H]^+$: 281.1575, found: 281.1578.

***N*-(1-phenylethyl)-*N'*-(2-chlorobenzyl)urea 33 (Table 4, entry 8):**



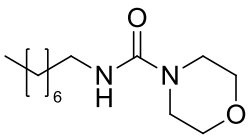
Yield 89 mg (98%); a yellowish solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 – 7.11 (m, 9H), 4.73 (q, *J*=6 Hz, 1H), 4.30 (s, 2H), 1.37 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.20, 129.59, 129.48, 129.02, 128.76, 127.58, 127.26, 126.12, 50.76, 42.36, 23.70 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{16}H_{17}ClN_2O$ $[M + H]^+$: 289.1029, found: 289.1031.

***N*-Octyl-*N'*-benzylurea 34 (Table 4, entry 9):**



Yield 74 mg (89%); a yellowish oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 7.26 (m, 5H), 4.38 (s, 2H), 3.15 (t, *J*=6 Hz, 2H), 1.47 (m, 2H), 1.25 (o.s., 10H), 0.82 (t, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.92, 139.46, 128.92, 127.66, 127.59, 44.77, 40.99, 32.13, 30.44, 29.56, 27.19, 22.98, 14.43 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{16}H_{26}N_2O$ $[M + H]^+$: 263.2045, found: 263.2041.

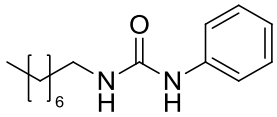
***N*-Octyl-morpholine-4-carboxamide 35 (Table 4, entry 10):**



Yield 68 mg (89%); a yellowish oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.70 (t, J=6 Hz, 4H), 3.33 (t, *J*=6 Hz, 4H), 3.22 (t, *J*=6 Hz, 2H), 1.50 (m, 2H), 1.35 – 1.17 (o.s., 10H), 0.87 (t, J=6 Hz, 3H) ppm.

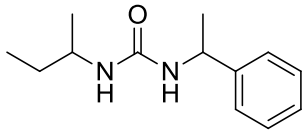
^{13}C NMR (75 MHz, $CDCl_3$) δ 158.20, 66.78, 44.27, 41.28, 32.09, 30.49, 29.52, 27.25, 22.93, 14.40 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{13}H_{26}N_2O_2$ $[M + H]^+$: 243.1994, found: 243.1996.

***N*-Octyl-*N'*-phenylurea 36 (Table 4, entry 11)** [5]**:**



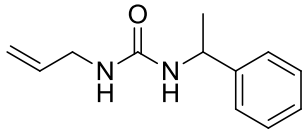
Yield 69 mg (88%); a yellowish oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 7.09 (m, 5H), 3.23 (t, *J*=6 Hz, 2H), 1.49 (m, 2H), 1.26 (o.s., 10H), 0.87 (t, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.84, 138.91, 129.50, 123.97, 121.28, 40.77, 32.14, 30.41, 29.57, 27.24, 22.98, 14.44 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{15}H_{24}N_2O$ $[M + H]^+$: 249.1888, found: 249.1893.

***N*-(*sec*-butyl)-*N'*-(1-phenylethyl)urea 37 (Table 4, entry 12):**



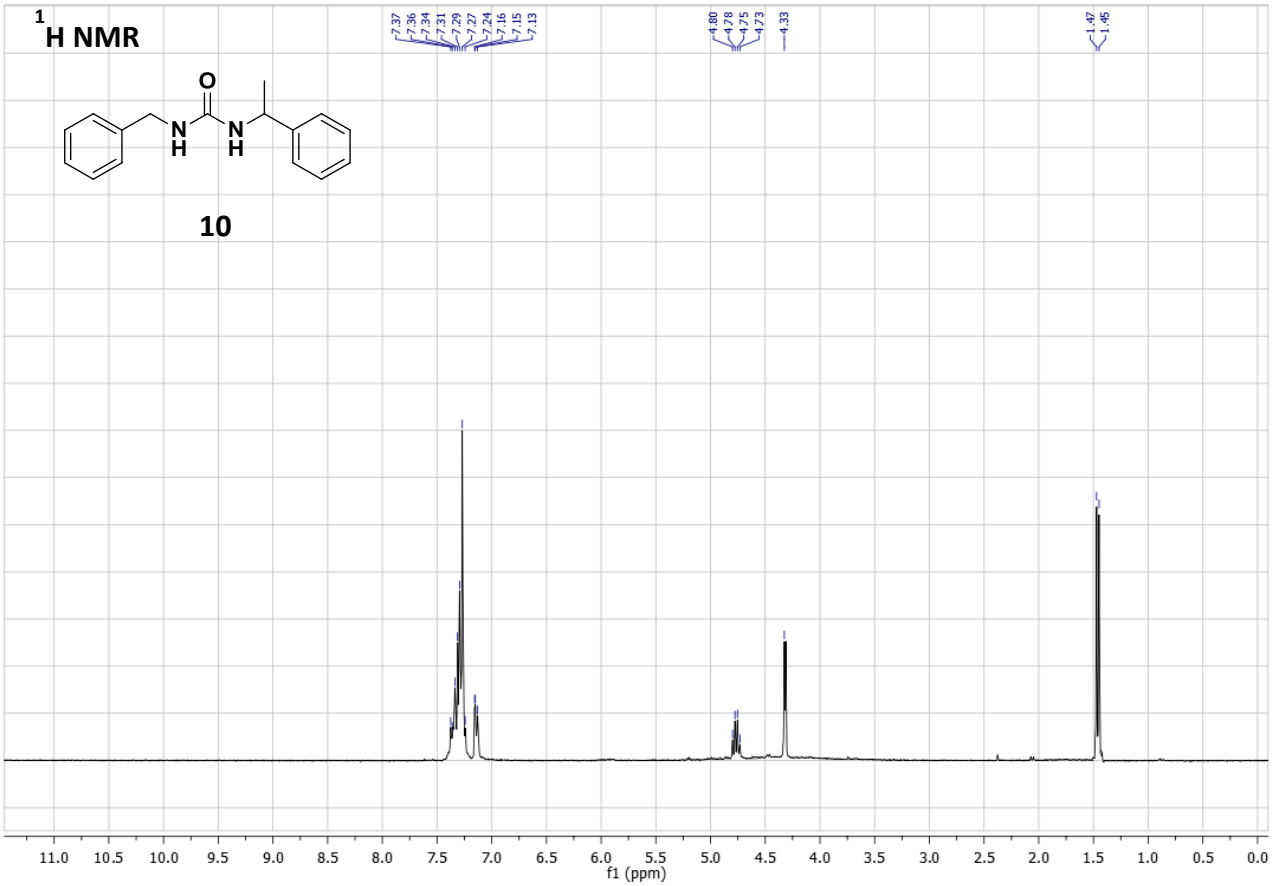
Yield 59 mg (85%); a yellowish solid. ^{1}H NMR (300 MHz, CD_3OD) δ 7.32 – 7.21 (m, 5H), 4.84 (q, *J*=6 Hz, 1H), 3.61 (m, *J*=6 Hz, 1H), 1.43 (m, 5H), 1.09 (d, *J*=6 Hz, 3H), 0.87 (t, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.72, 143.87, 128.98, 127.56, 126.02, 51.31, 48.90, 29.97, 20.92, 20.84, 10.58 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{13}H_{20}N_2O$ $[M + H]^+$: 221.1575, found: 221.1577.

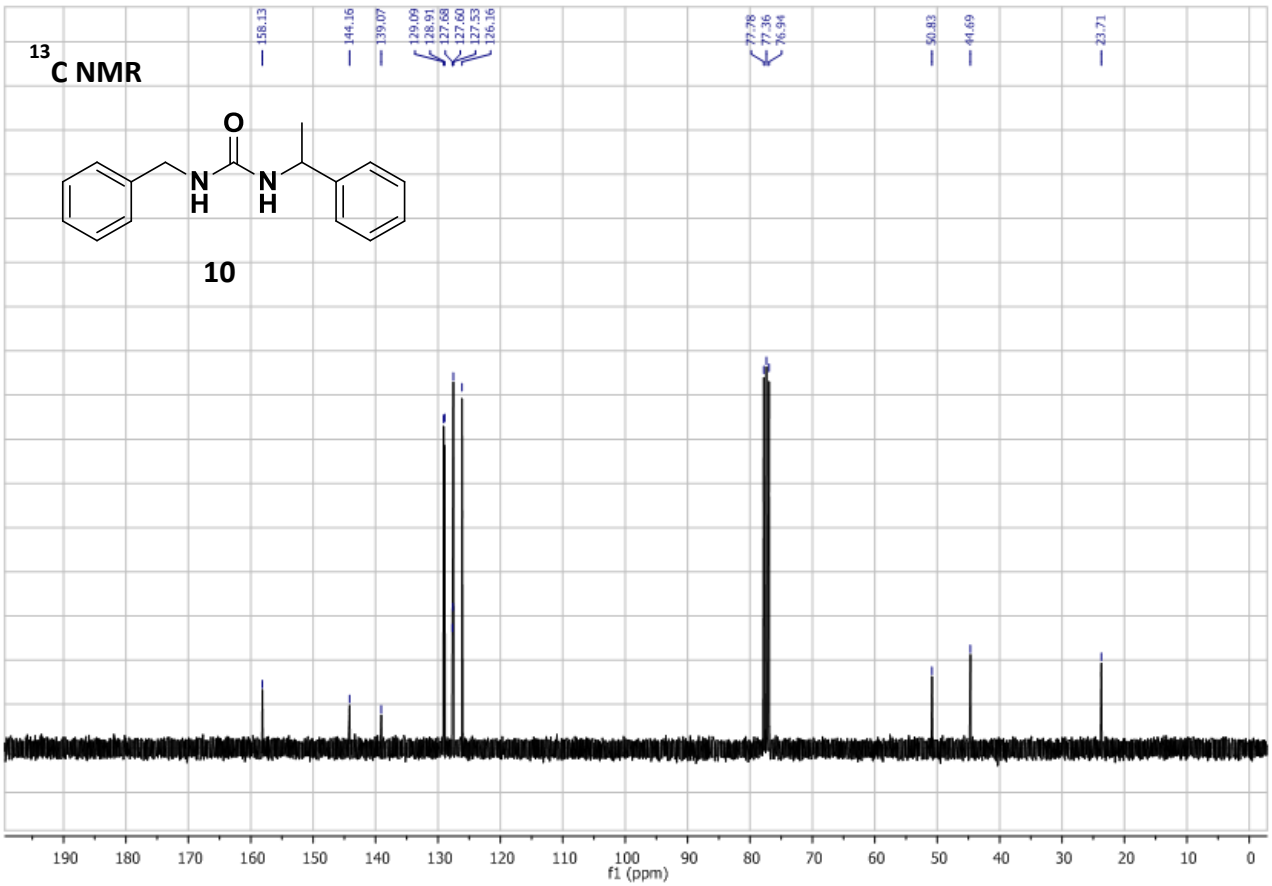
***N*-allyl-*N'*-(1-phenylethyl)urea 38 (Table 4, entry 13):**

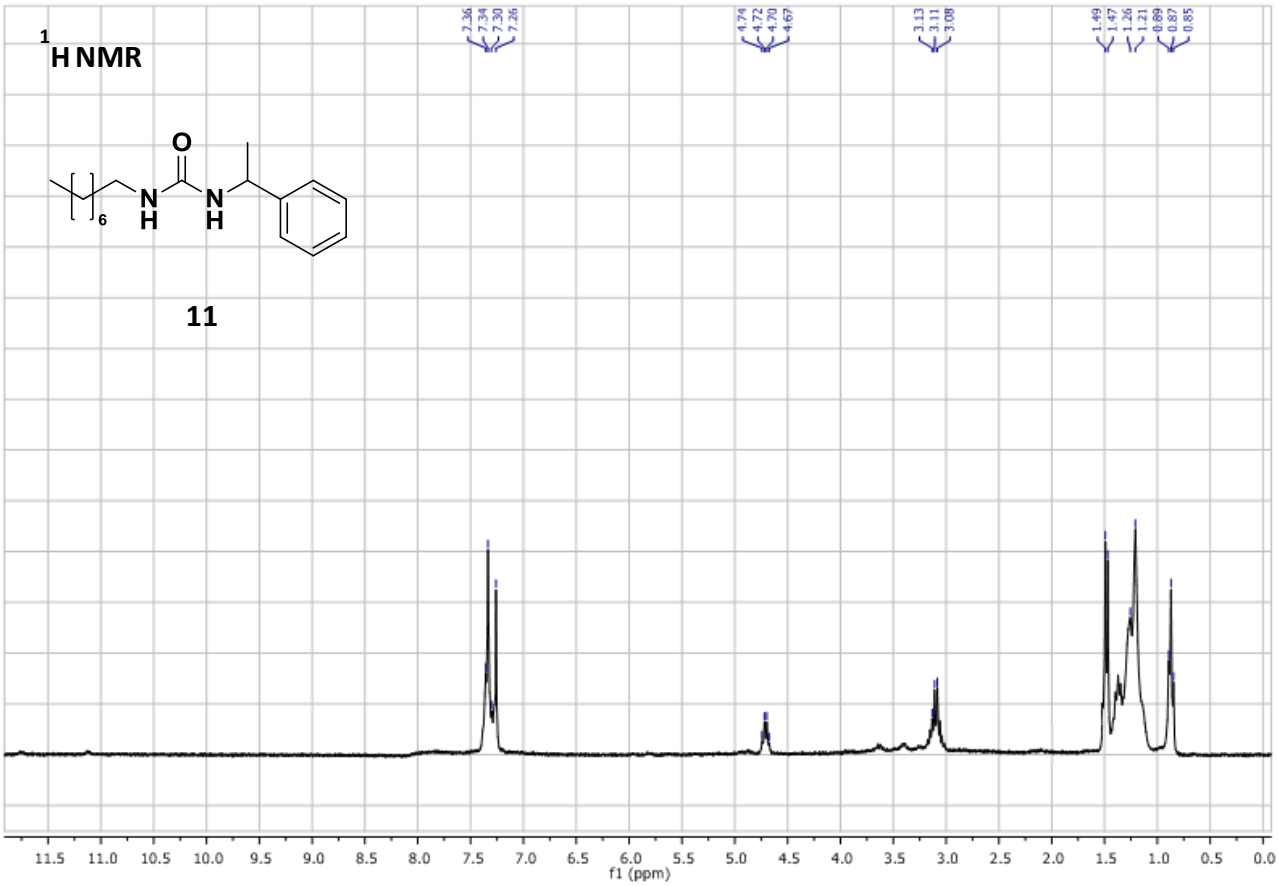


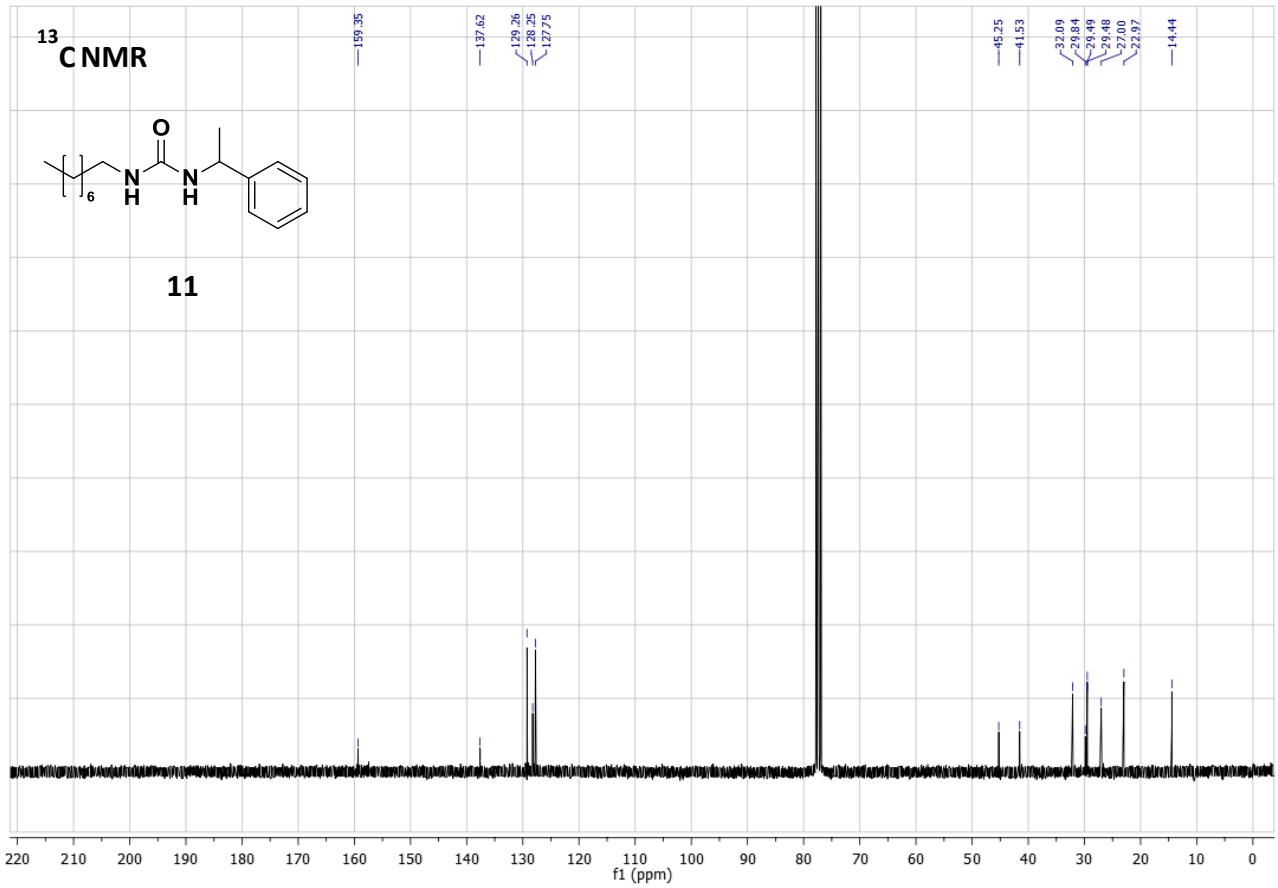
Yield 54 mg (83%); a yellowish solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.20 (m, 5H), 5.70 (m, 1H), 5.00 (m, 2H), 4.73 (q, *J*=6 Hz, 1H), 4.08 (d, *J*=9 Hz, 2H), 1.38 (d, *J*=6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.30, 144.44, 135.47, 120.02, 127.57, 126.17, 115.75, 50.58, 43.10, 23.70 ppm. *m/z* (MALDI-TOF MS): calcd for $C_{12}H_{16}N_2O$ $[M + H]^+$: 205.1262, found: 205.1266.

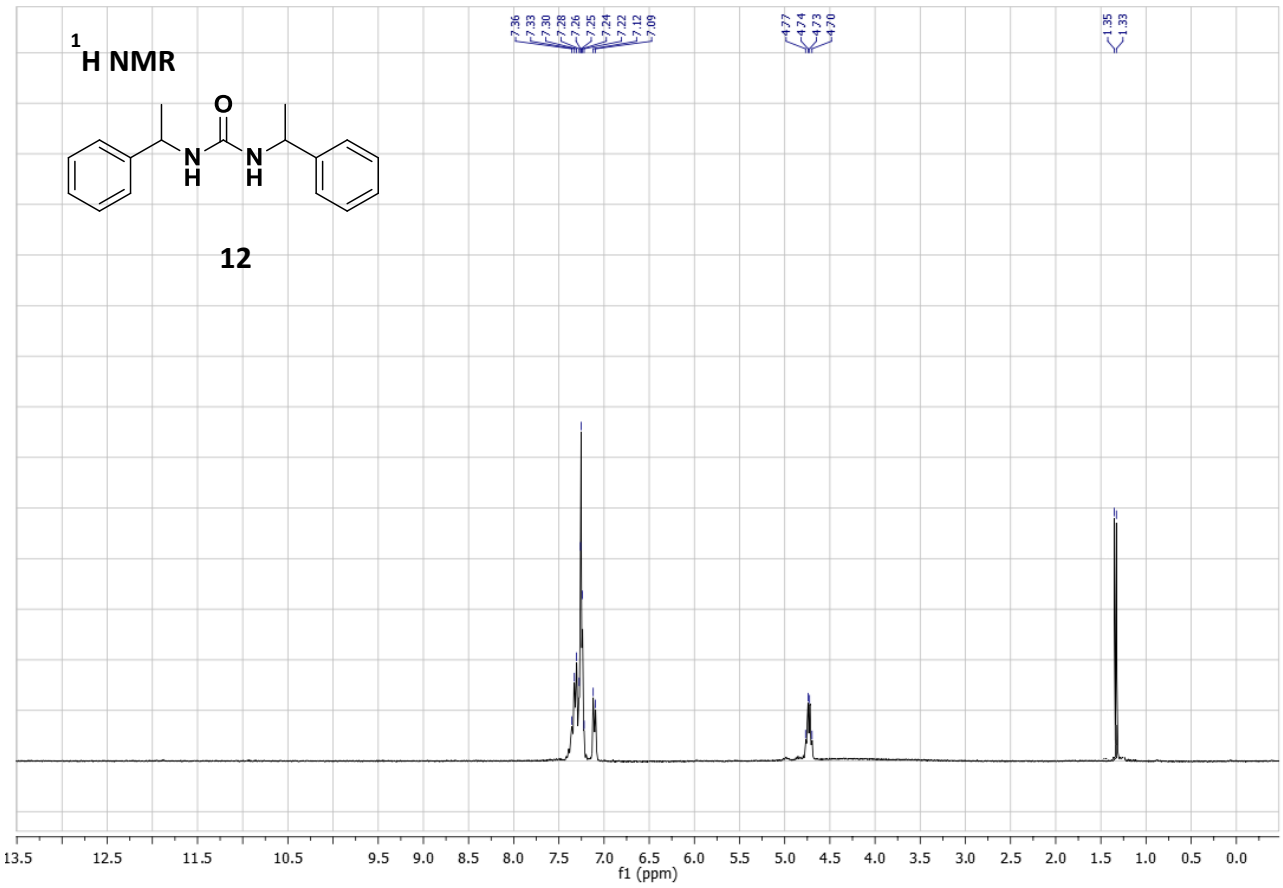
2. ^{1}H and ^{13}C NMR spectra

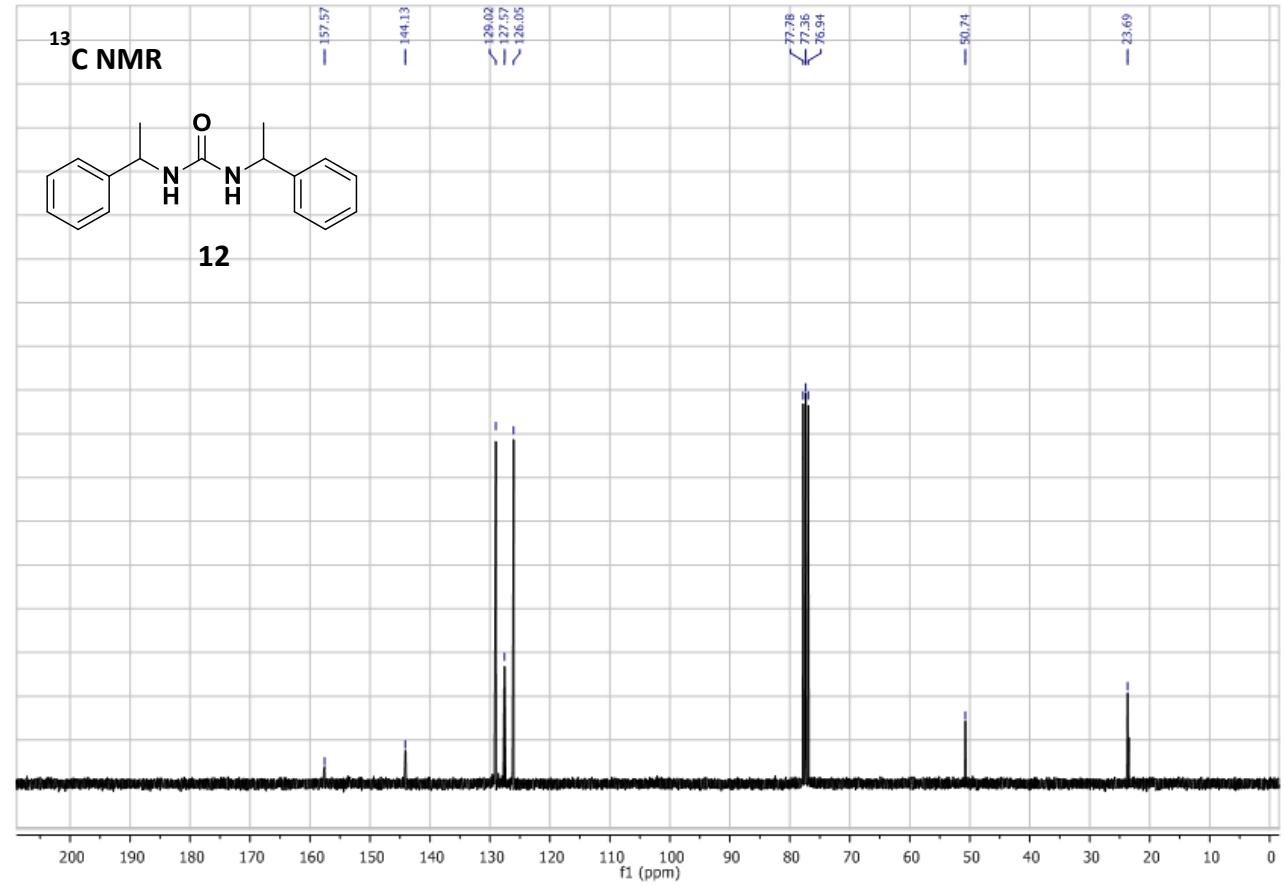





1H NMR
11
f1 (ppm)


13C NMR
11
f1 (ppm)


1H NMR
12
7.36
7.33
7.30
7.28
7.26
7.25
7.24
7.22
7.12
7.09
4.77
4.74
4.73
4.70
1.35
1.33
f1 (ppm)


13C NMR
12
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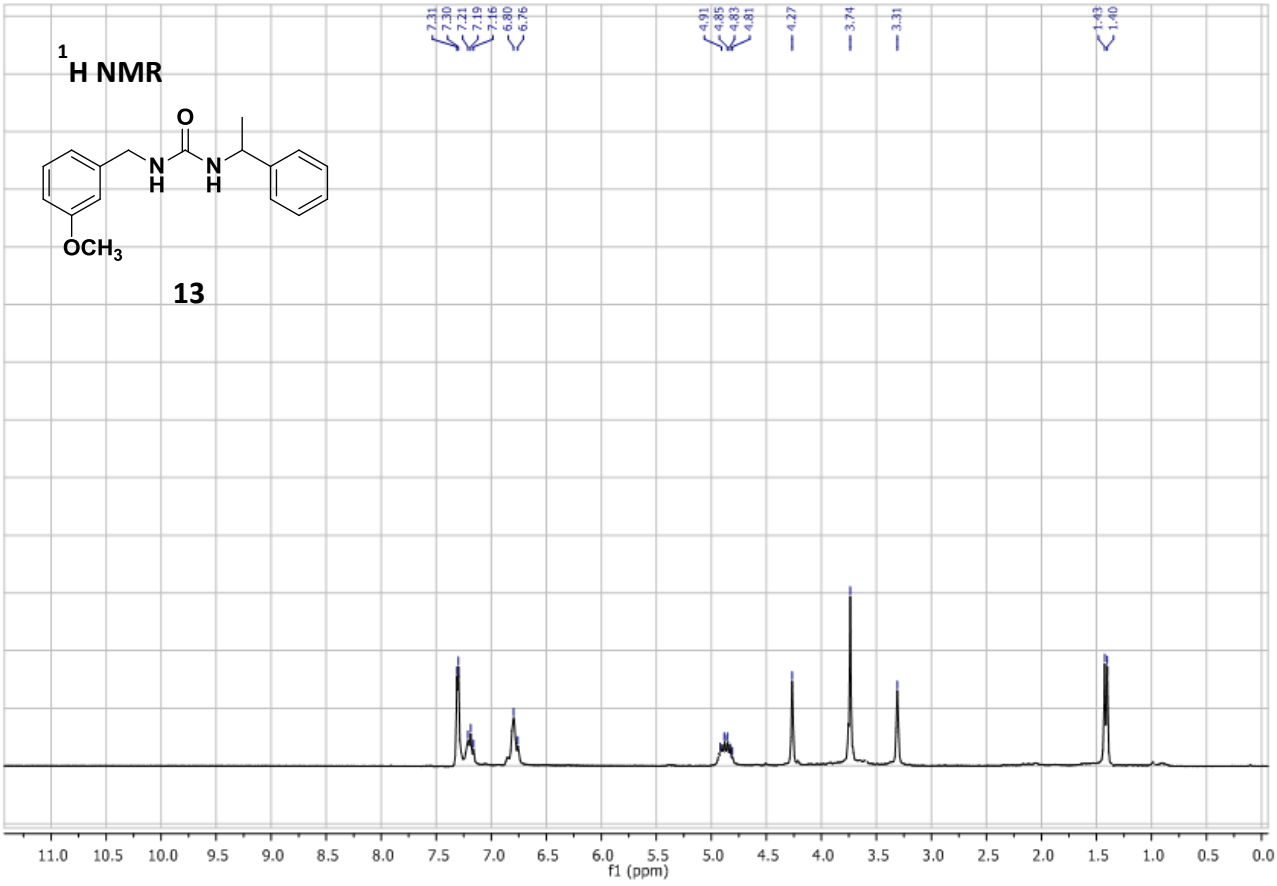
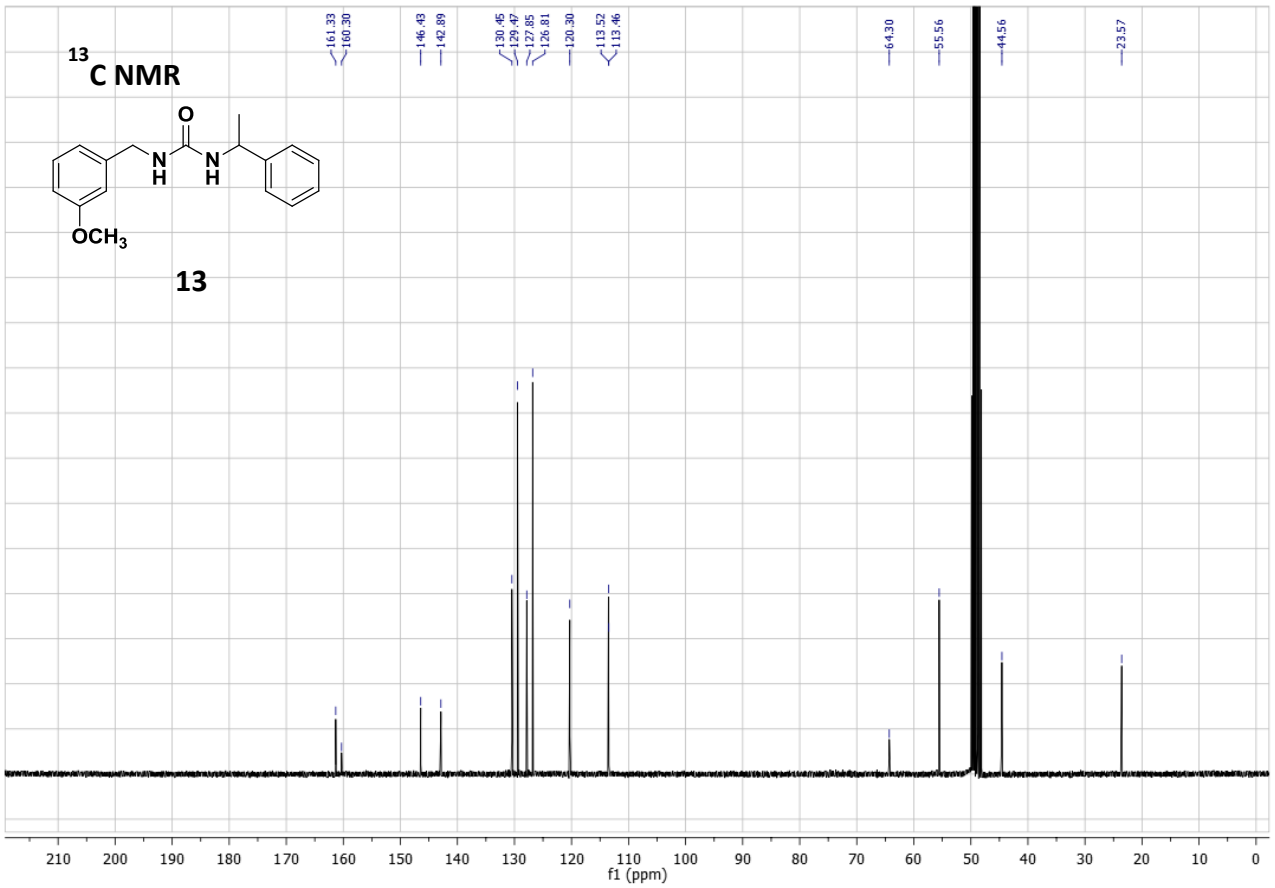
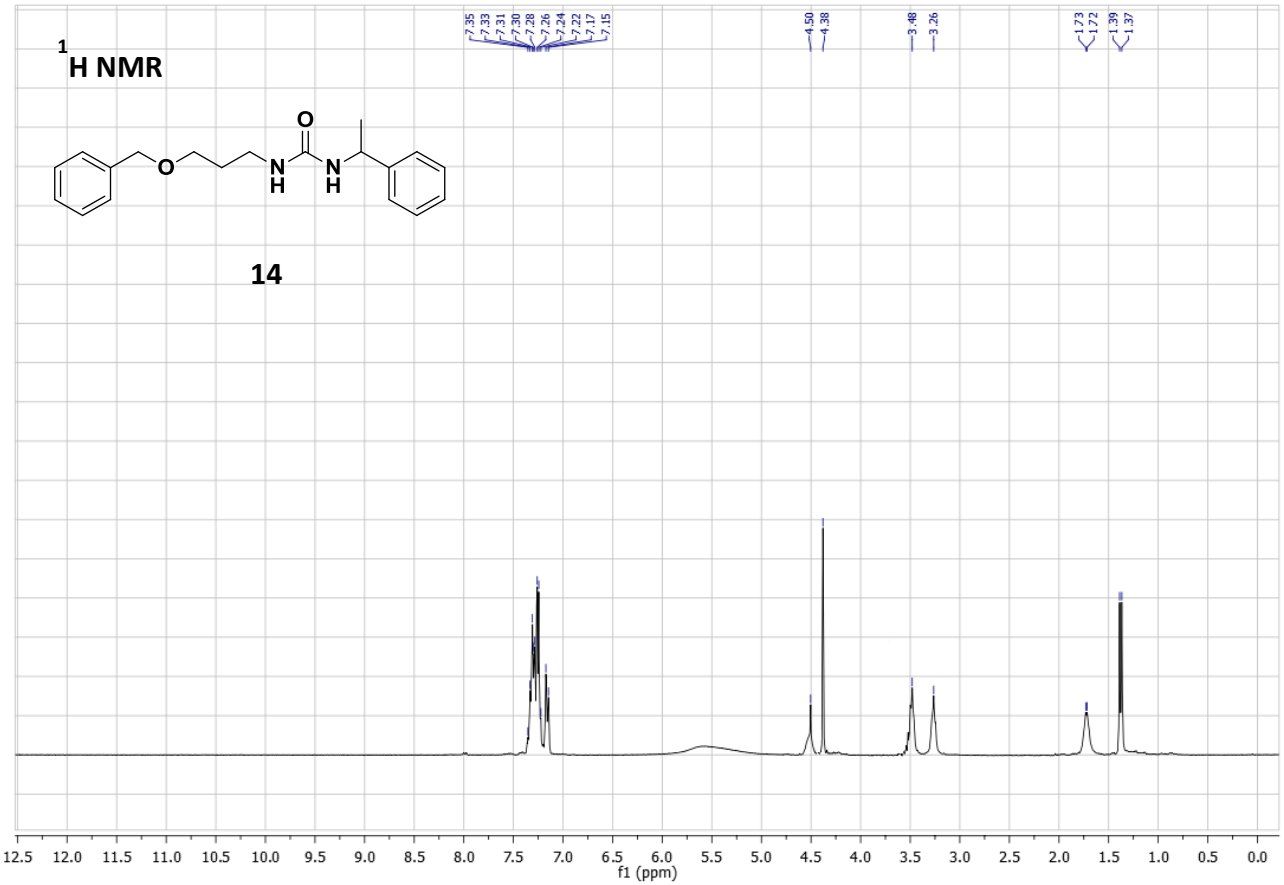
129.02
127.57
126.05
77.38
77.36
76.94
50.74
23.69
f1 (ppm)


1H NMR
13
f1 (ppm)


13C NMR
13
f1 (ppm)


1H NMR
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7.35
7.33
7.31
7.30
7.28
7.26
7.24
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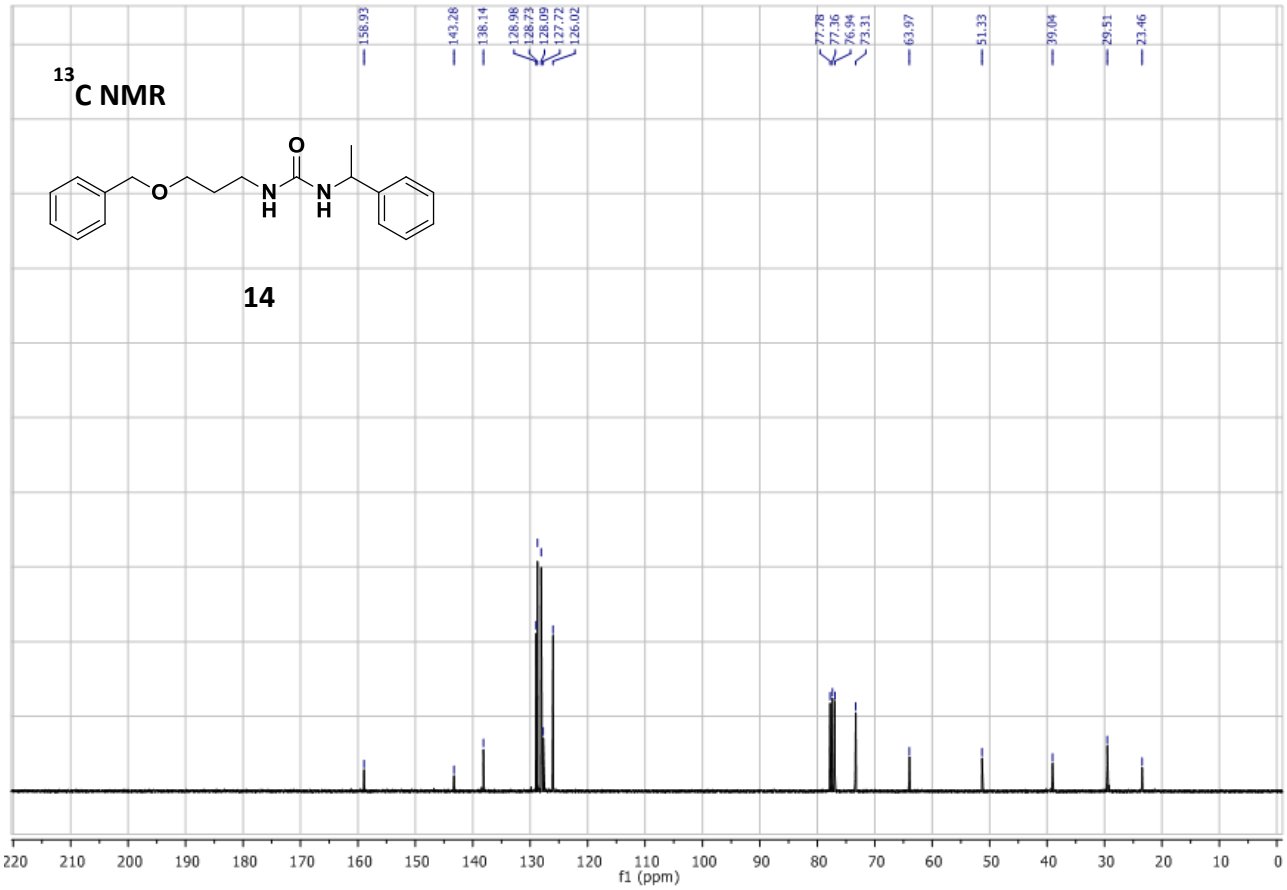
4.50
4.38
3.48
3.26
1.73
1.72
1.39
1.37
f1 (ppm)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14
158.93
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128.73
128.09
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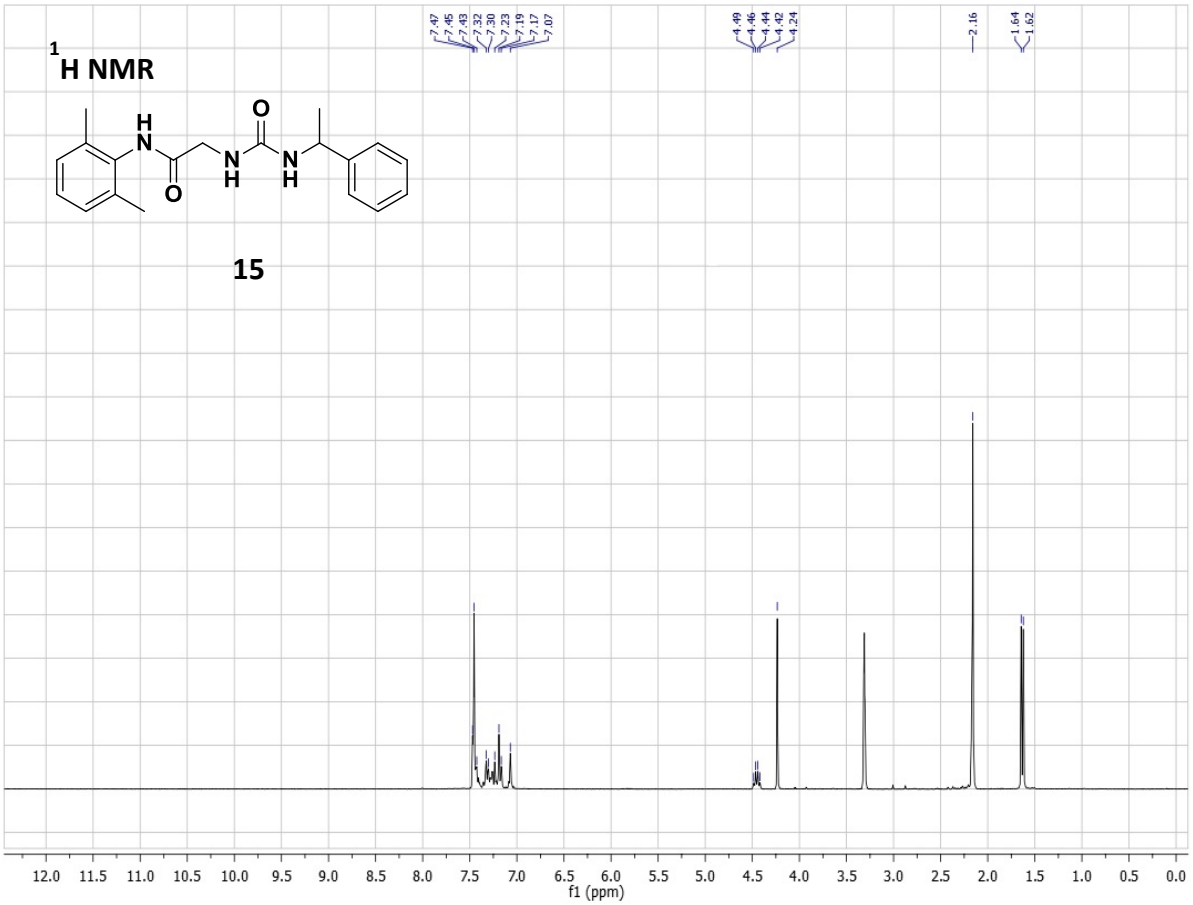
51.33
39.04
29.51
23.46
f1 (ppm)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7.47
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7.30
7.23
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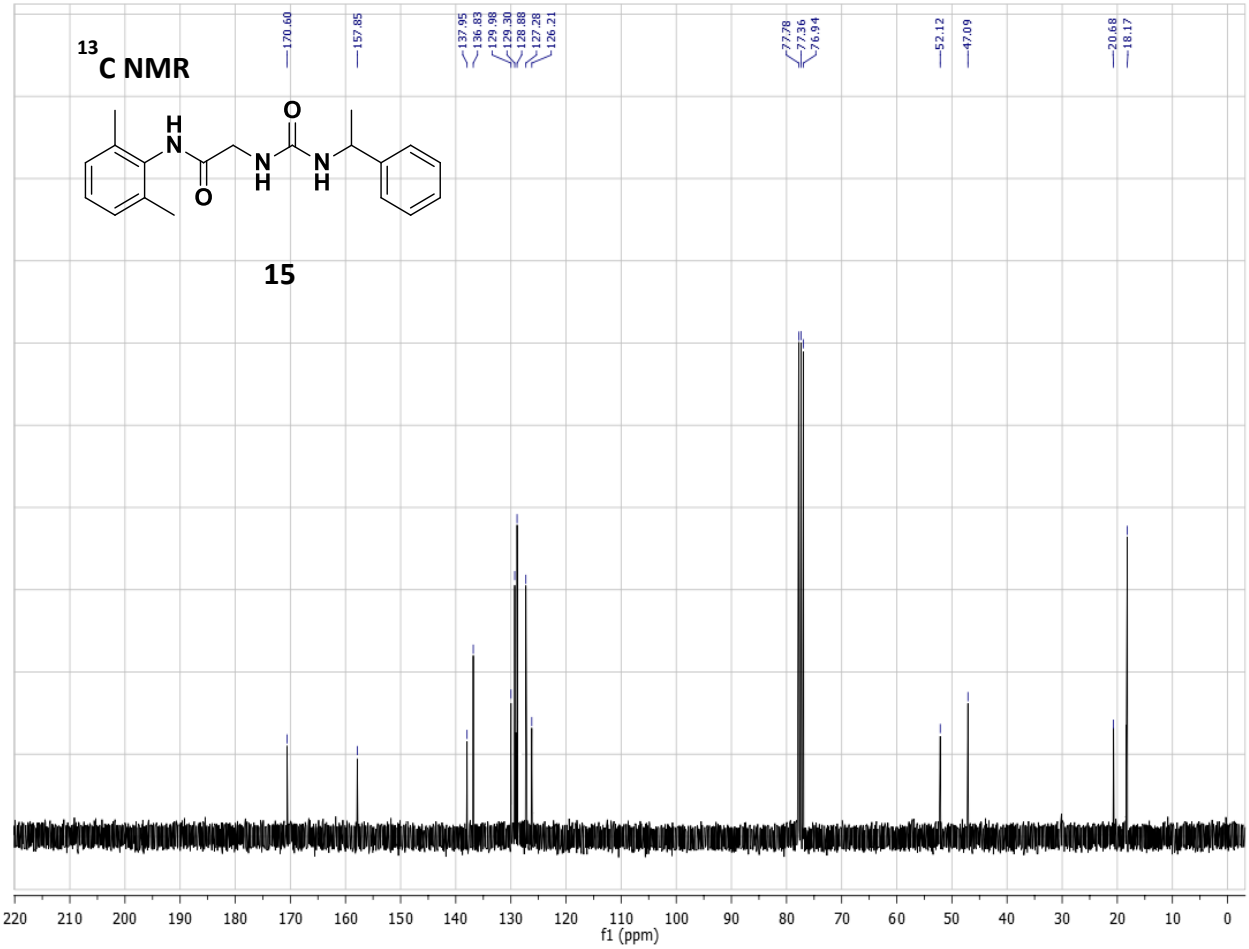
7.17
7.07
4.49
4.46
4.44
4.42
4.24
2.16
1.64
1.62
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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170.60
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137.95
136.83
129.98
129.30
128.88
127.28
126.21
77.78
77.36
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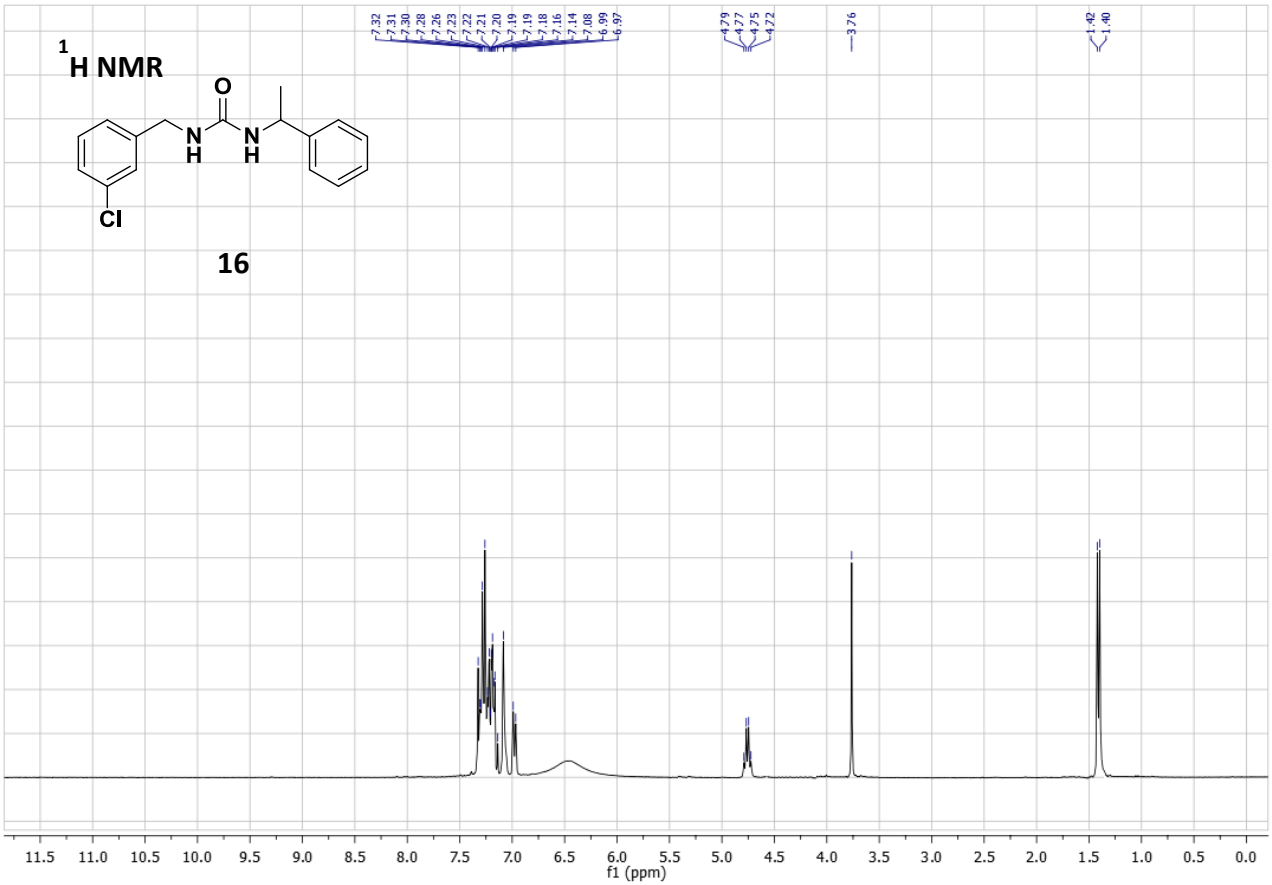
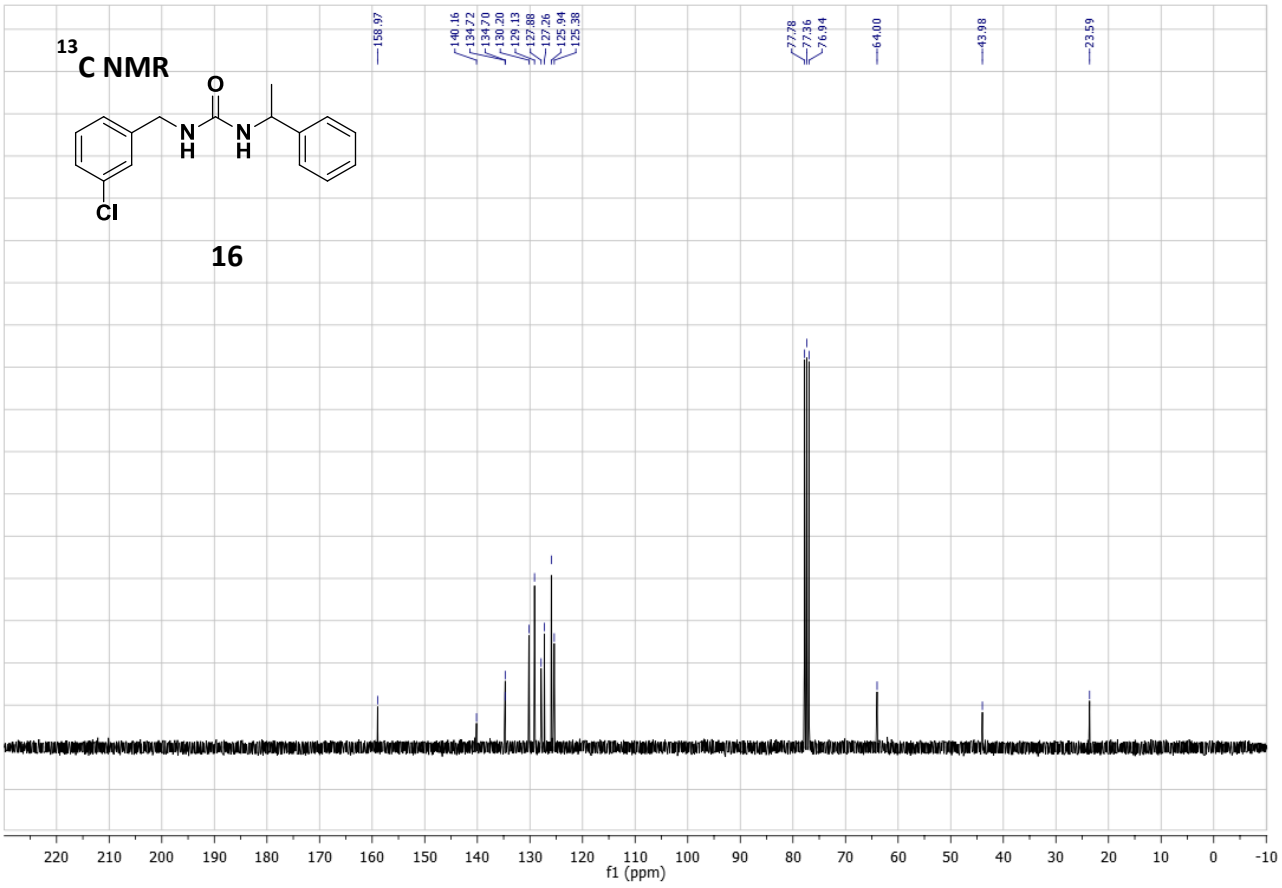
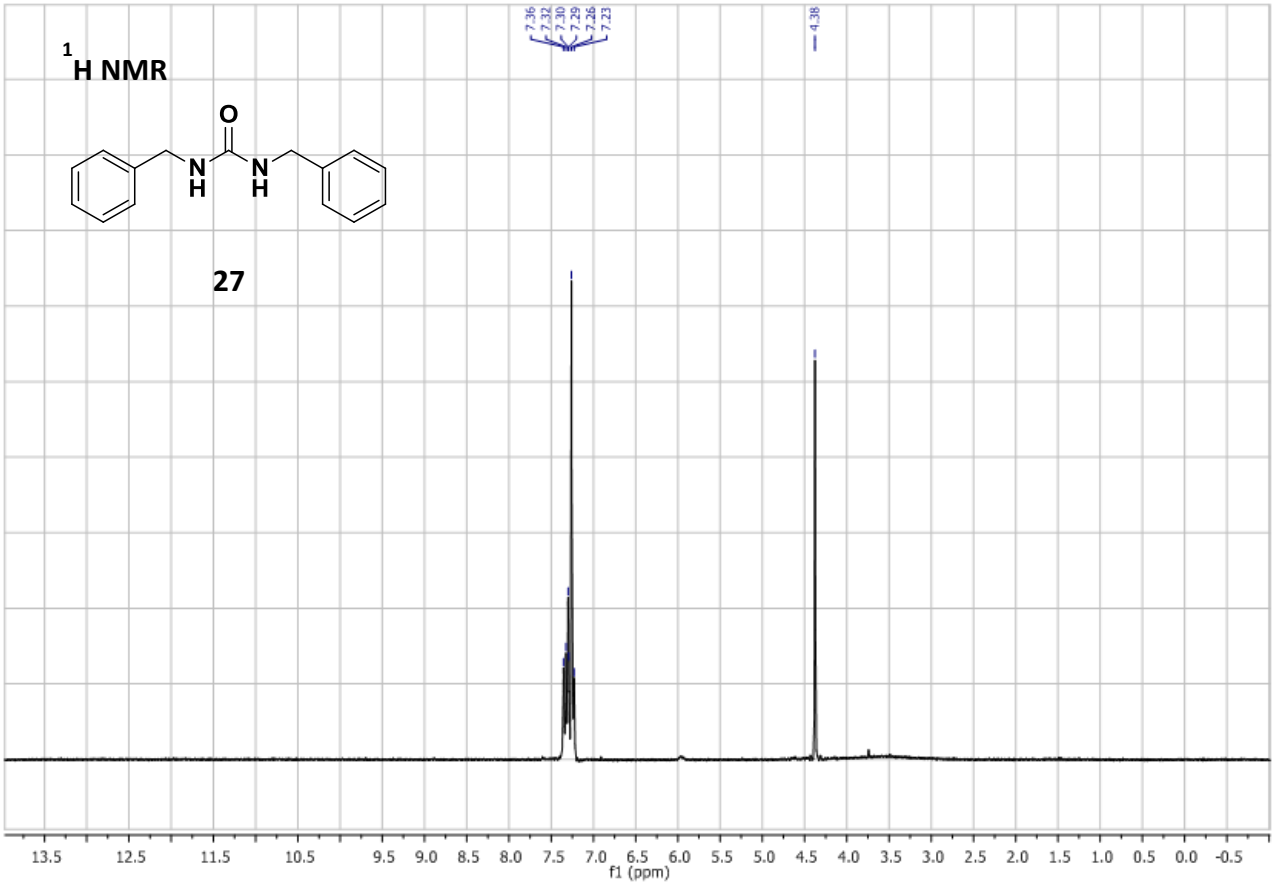
47.09
20.68
18.17
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


1H NMR
16
f1 (ppm)


13C NMR
16
f1 (ppm)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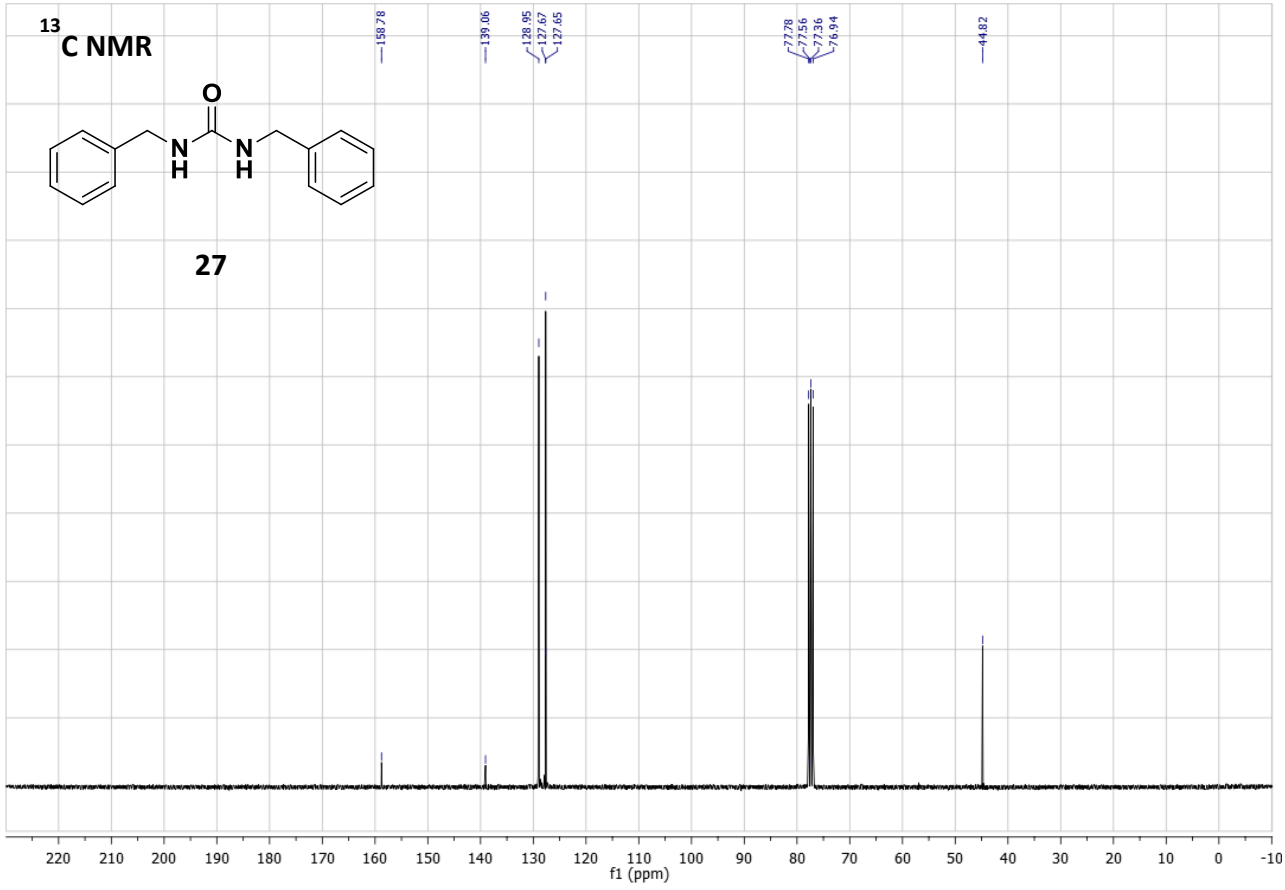
27
7.36
7.32
7.30
7.29
7.26
7.23
4.38
f1 (ppm)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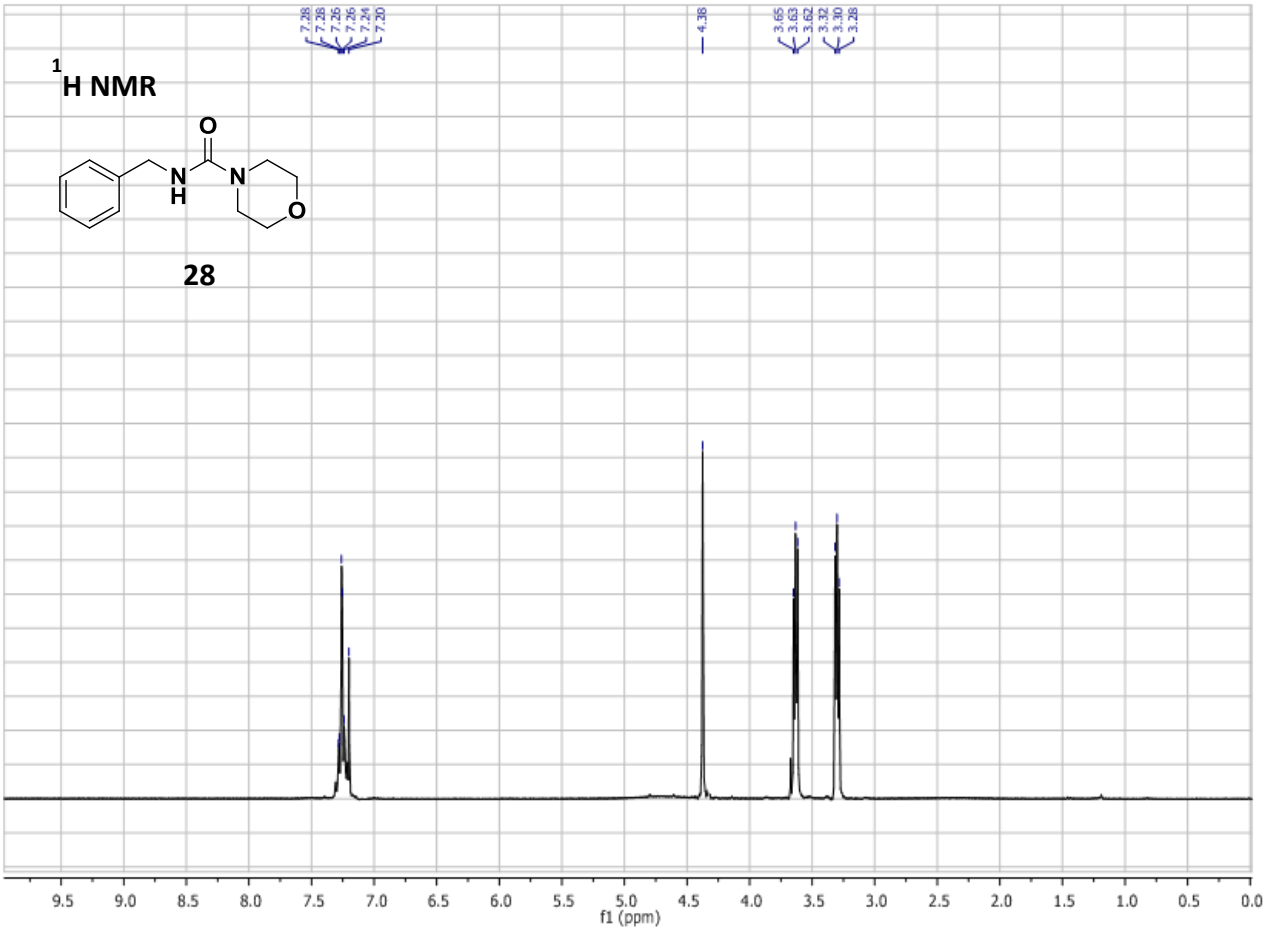
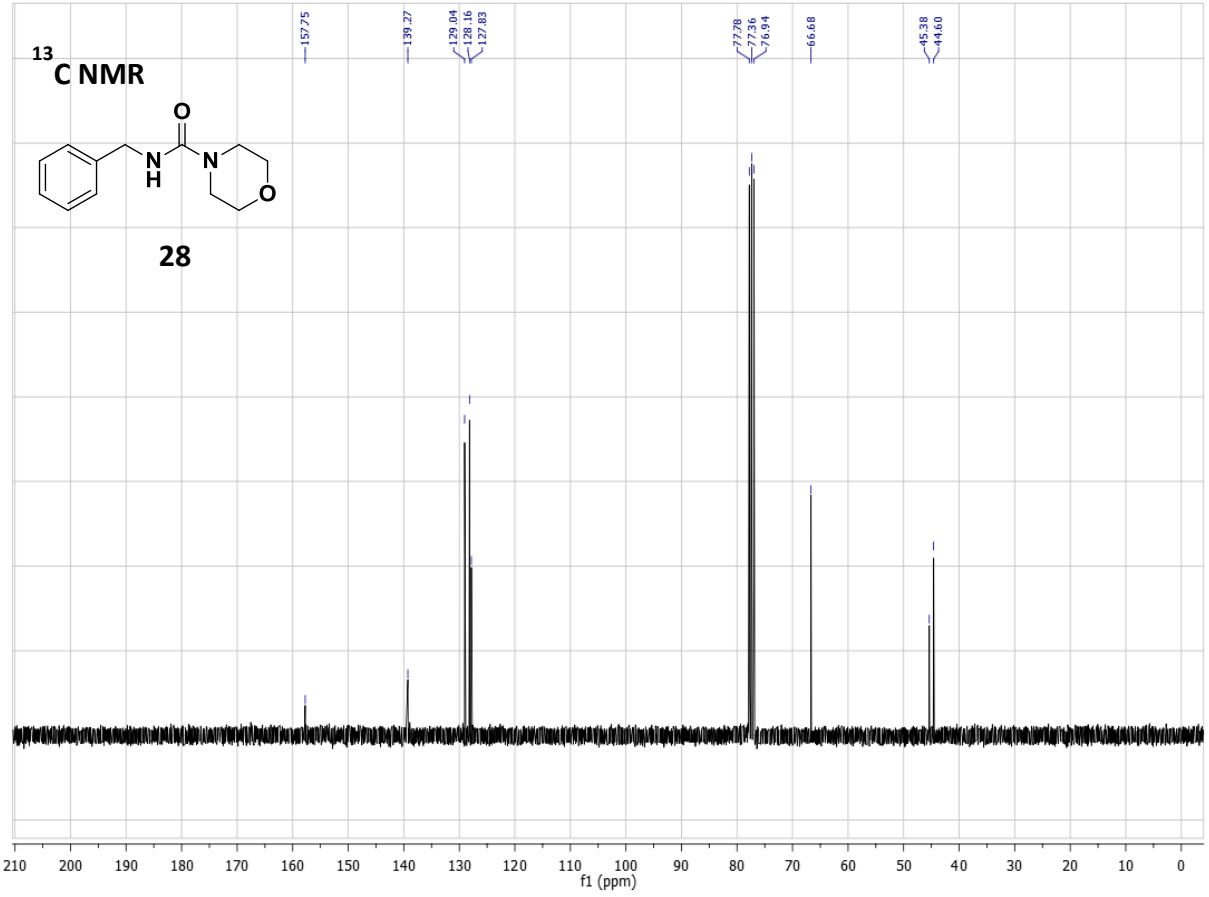
127.65
77.78
77.56
77.36
76.94
44.82
f1 (ppm)


1H NMR
28
7.28
7.26
7.26
7.26
7.24
7.20
4.38
3.65
3.63
3.62
3.32
3.30
3.28
f1 (ppm)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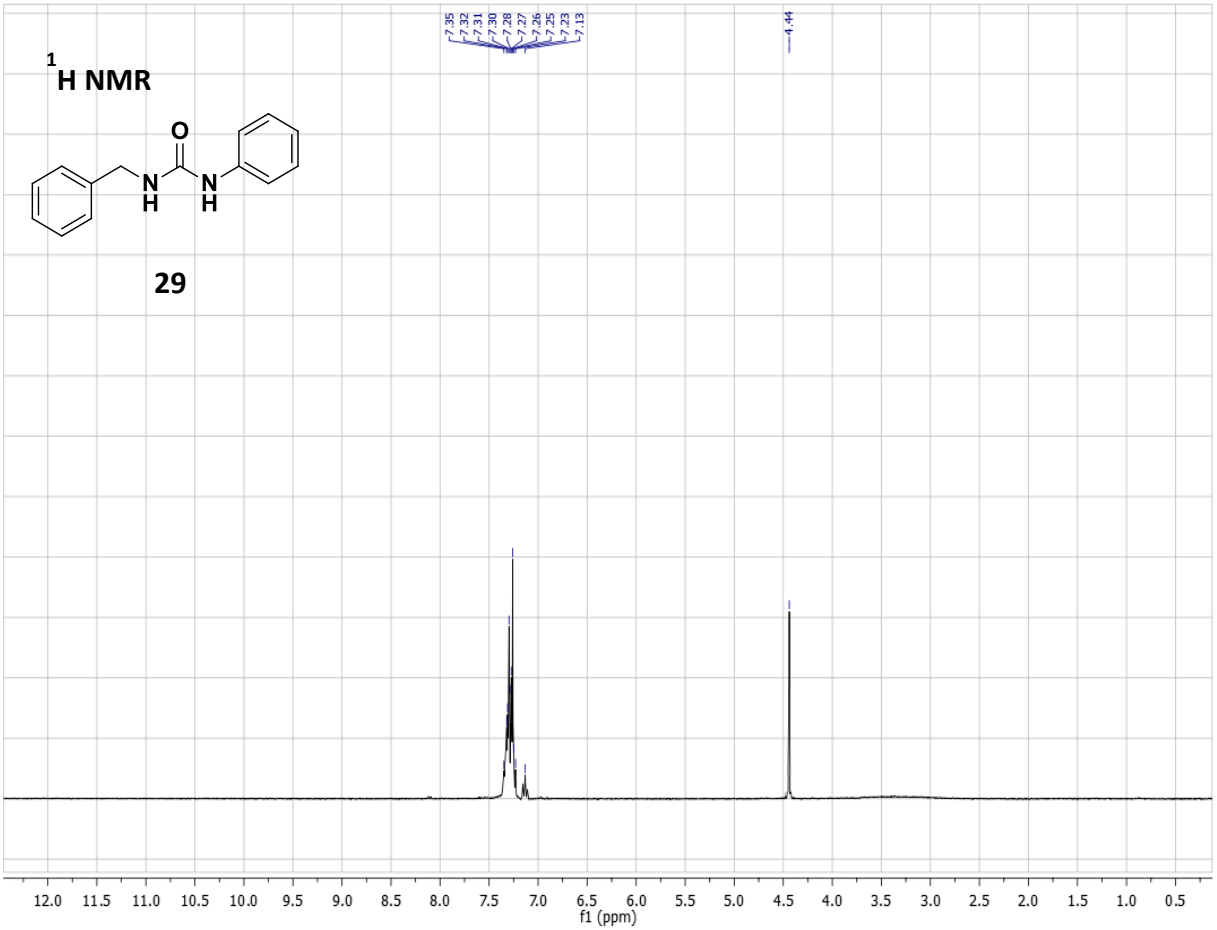
77.36
76.94
66.68
45.38
44.60
f1 (ppm)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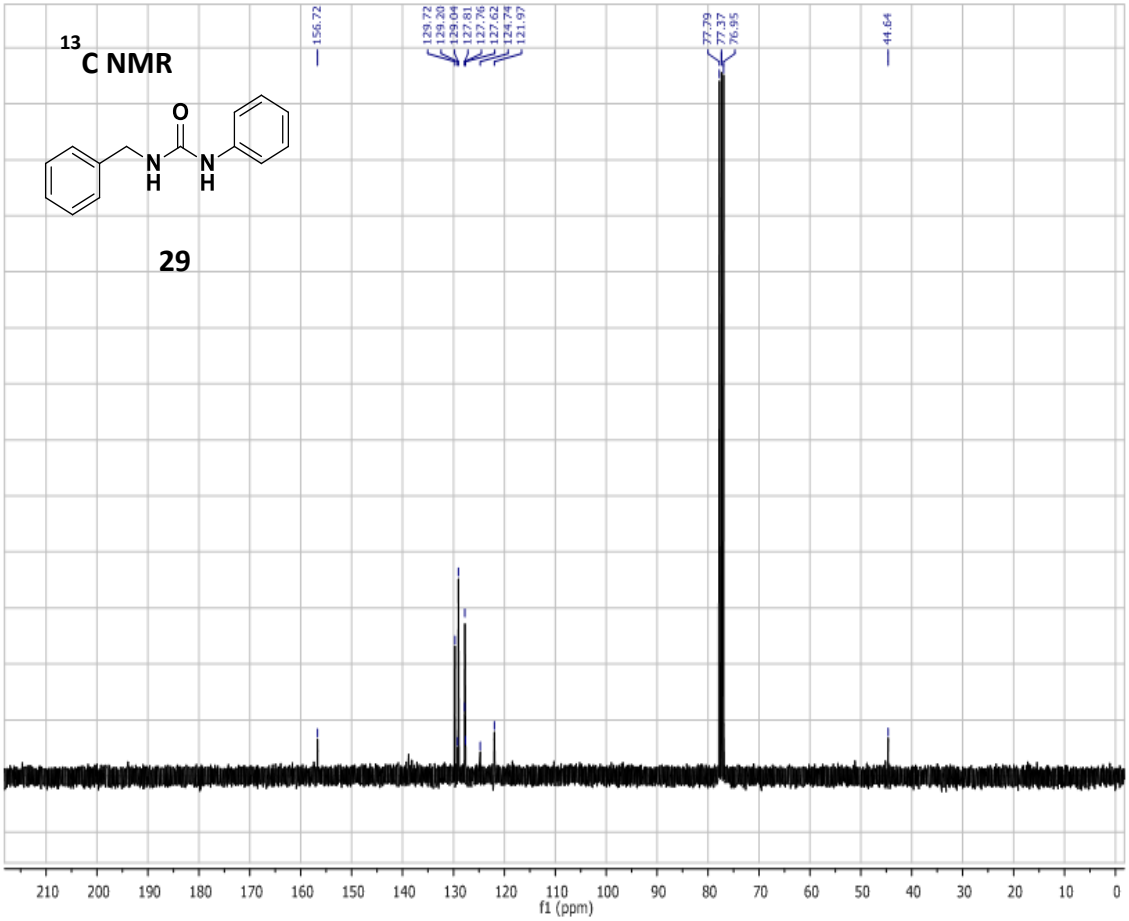
O
N
H
N
H
29
7.35
7.32
7.31
7.30
7.28
7.27
7.26
7.25
7.23
7.13
4.44
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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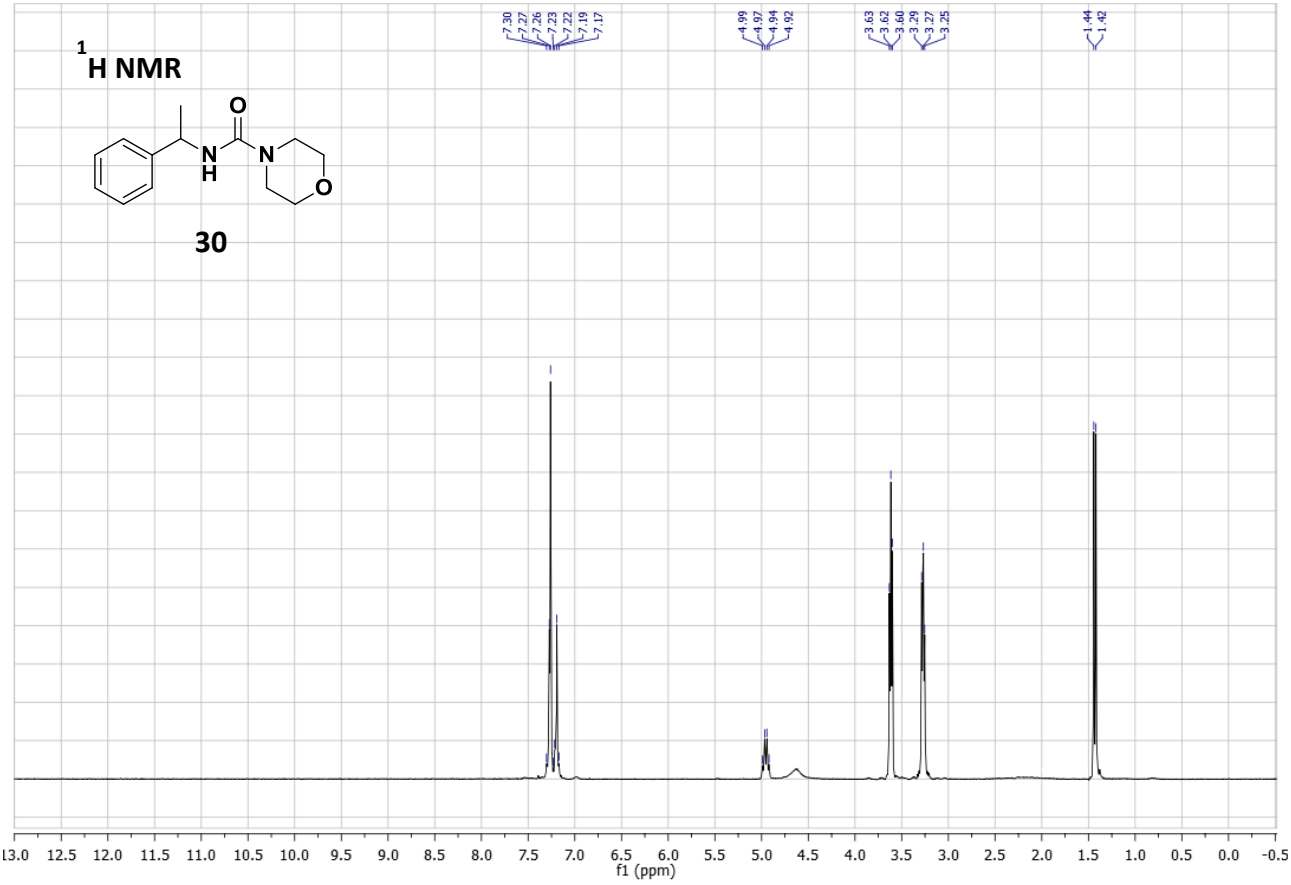
O
N
H
N
H
29
156.72
129.72
129.20
129.04
127.81
127.76
127.62
124.74
121.97
77.39
77.37
76.95
44.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


1H NMR
30
7.30
7.27
7.26
7.23
7.22
7.19
7.17
4.99
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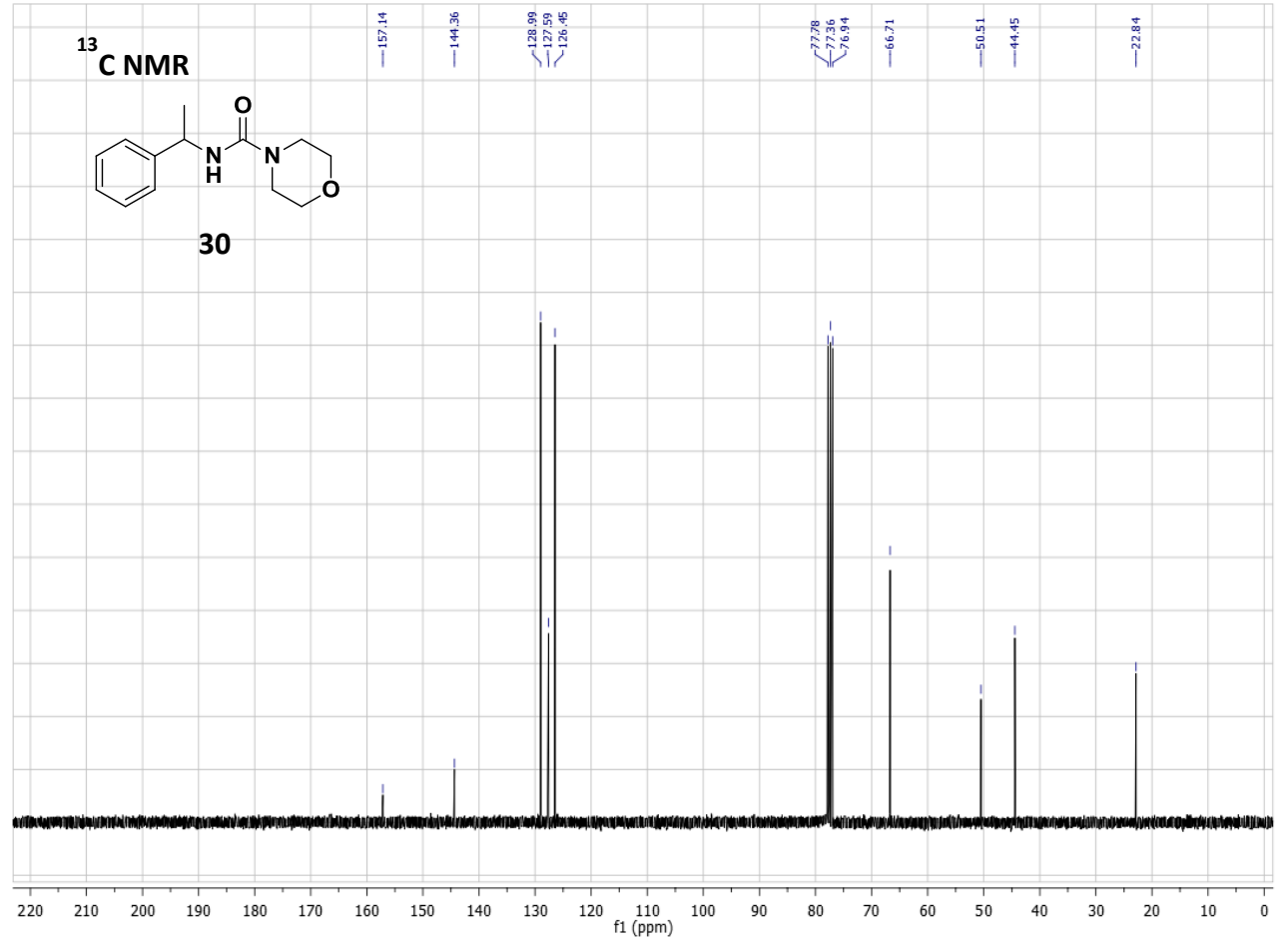
4.94
4.92
3.63
3.62
3.60
3.29
3.27
3.25
1.44
1.42
f1 (ppm)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30
157.14
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77.36
76.94
66.71
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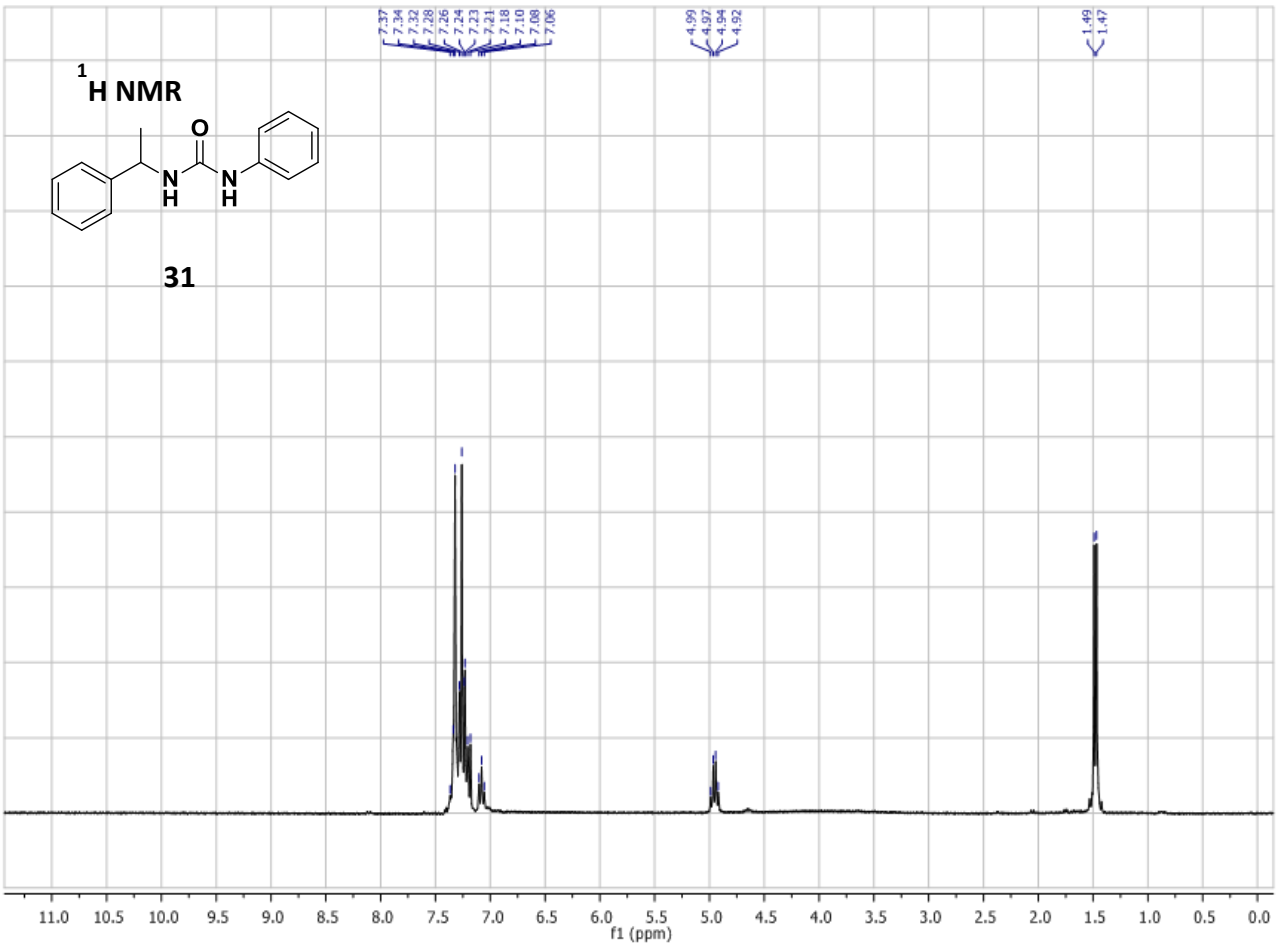
44.45
22.84
f1 (ppm)


1H NMR
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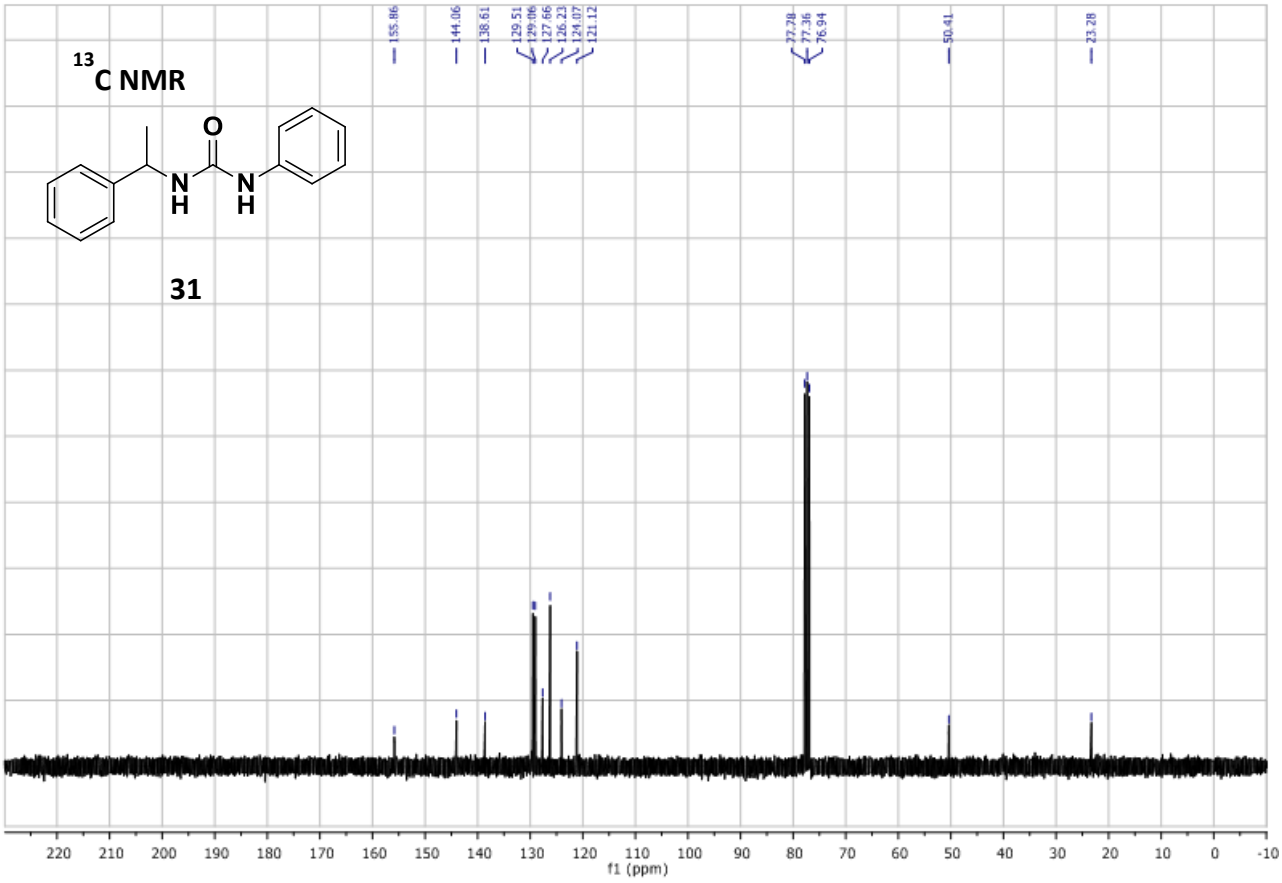
7.37
7.34
7.32
7.28
7.26
7.24
7.23
7.21
7.18
7.10
7.08
7.06
4.99
4.97
4.94
4.92
1.49
1.47
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.06
127.66
126.23
124.07
121.12
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76.94
50.41
23.28
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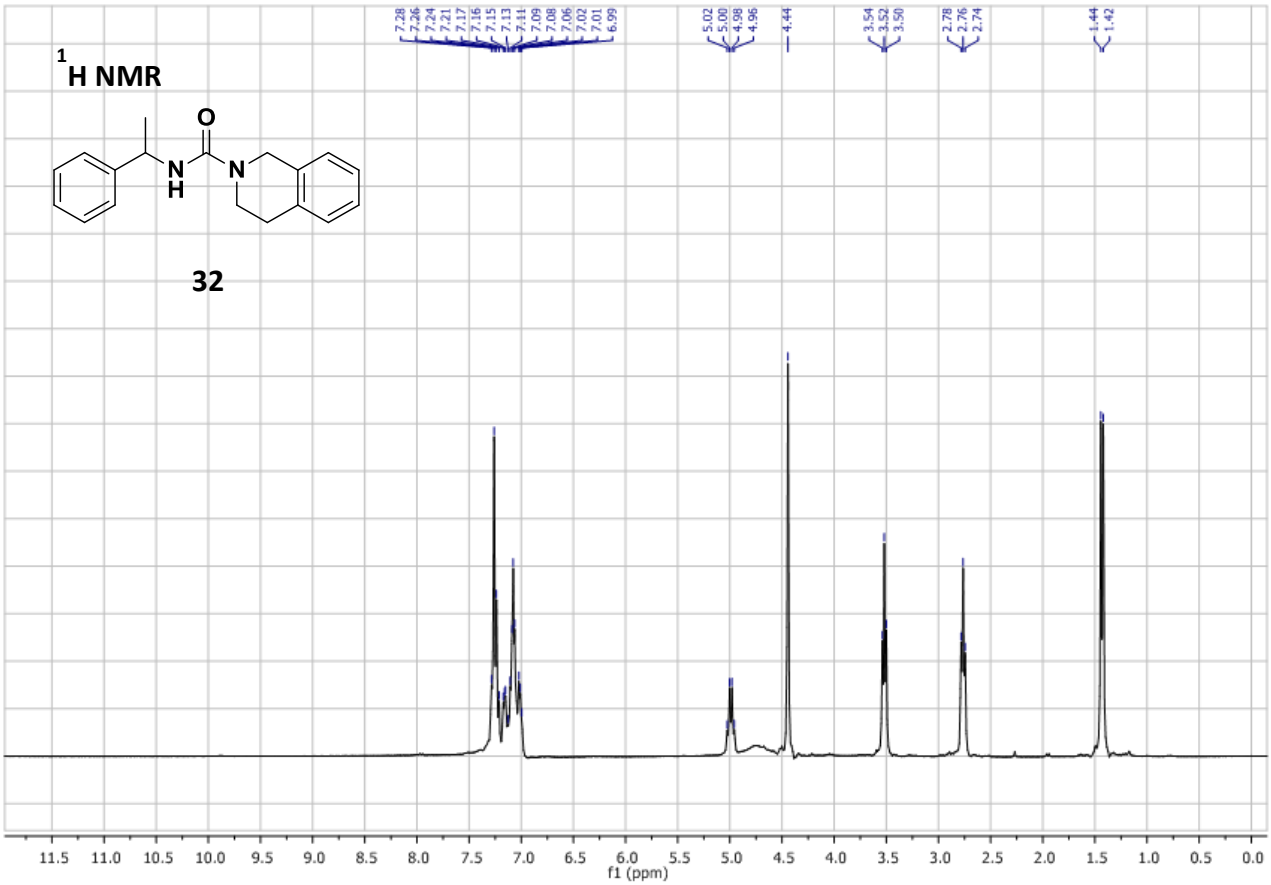
f1 (ppm)


1H NMR
32
7.28
7.26
7.24
7.21
7.17
7.16
7.15
7.13
7.11
7.09
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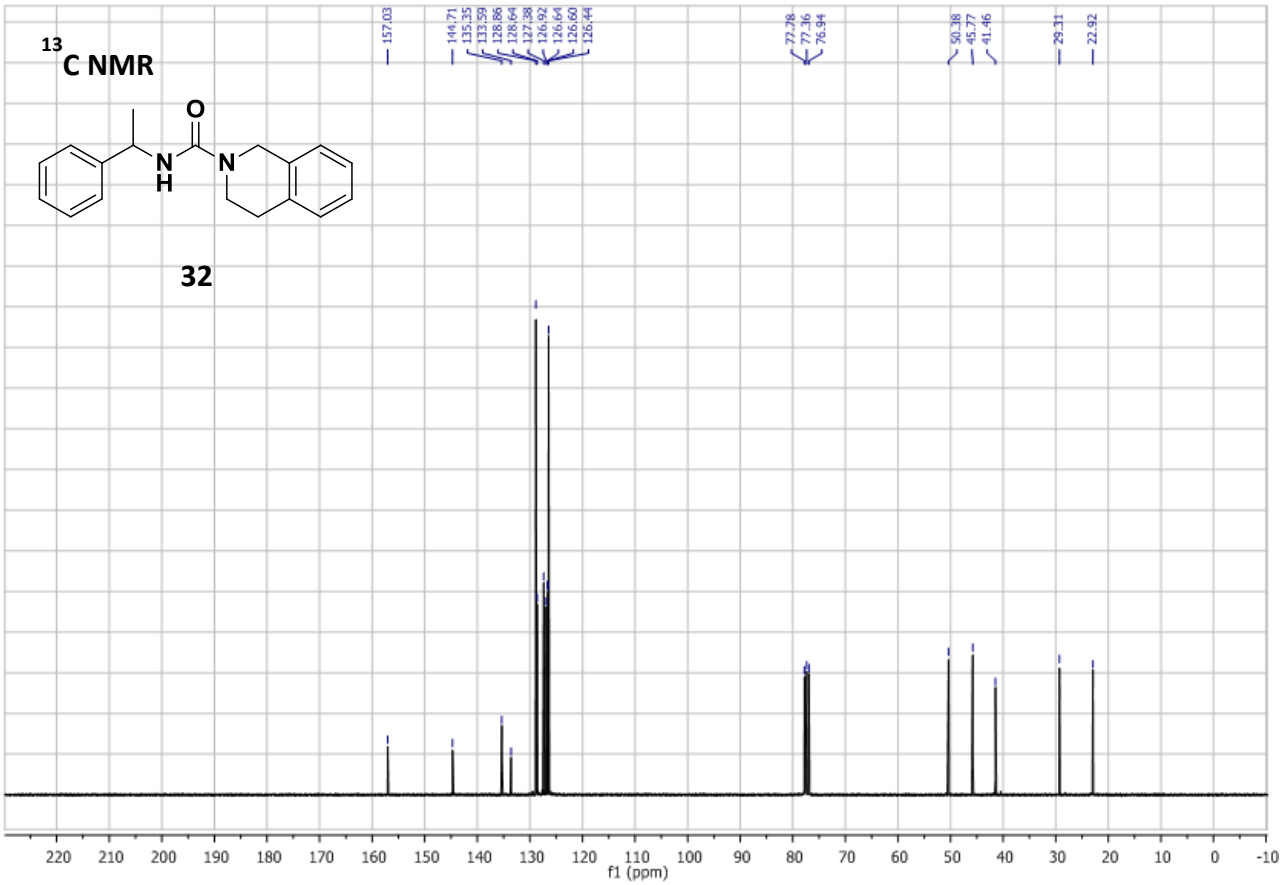
7.06
7.02
7.01
6.99
5.02
5.00
4.98
4.96
4.44
3.54
3.52
3.50
2.78
2.76
2.74
1.44
1.42
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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157.03
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127.38
126.92
126.64
126.60
126.44
77.28
77.16
76.94
50.38
45.77
41.46
29.31
22.92
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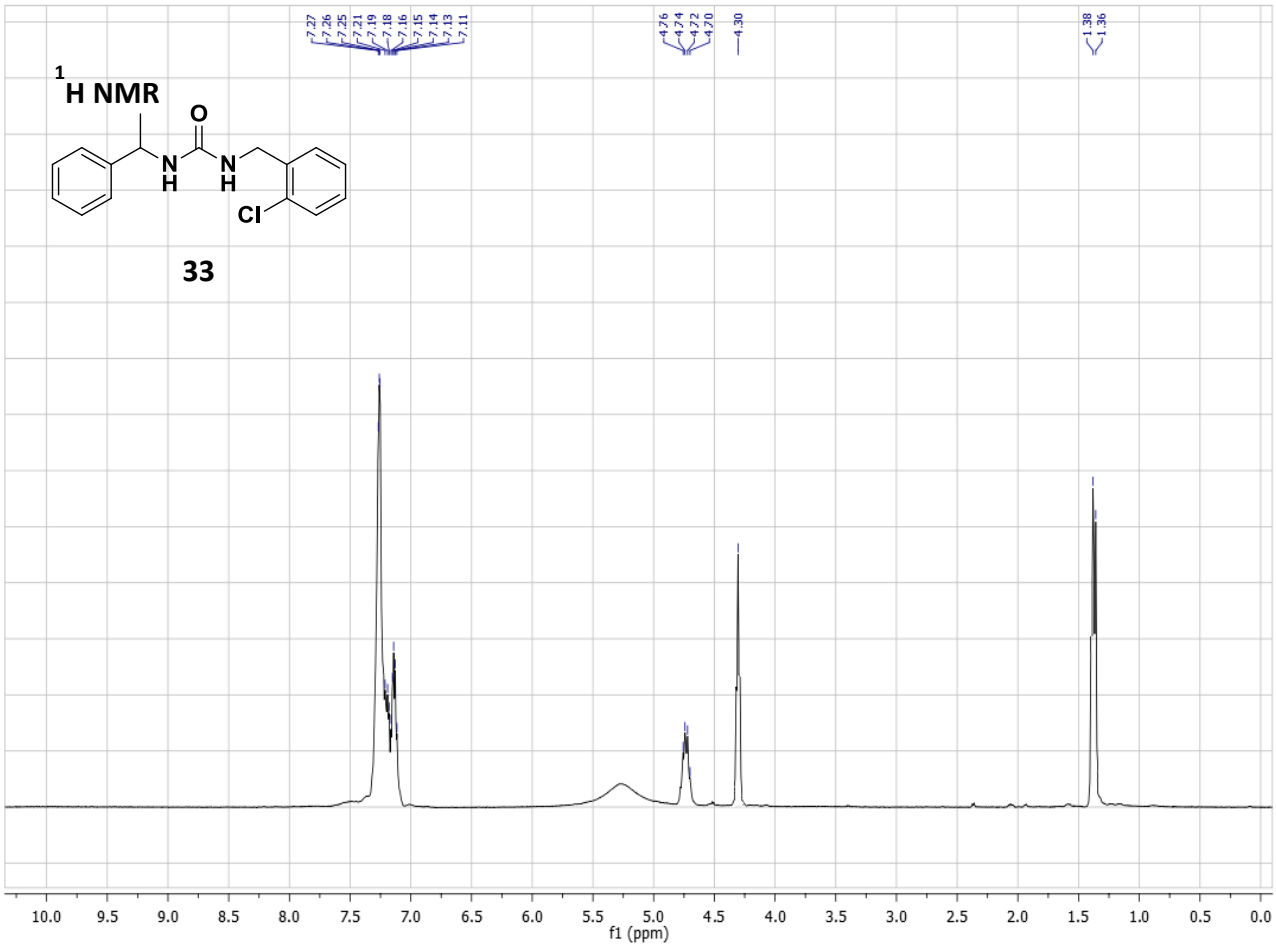
f1 (ppm)


1H NMR
O
N
H
N
H
Cl
33
7.27
7.26
7.25
7.21
7.19
7.18
7.16
7.15
7.14
7.13
7.11
4.76
4.74
4.72
4.70
4.30
1.38
1.36
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
3.5
3.0
2.5
2.0
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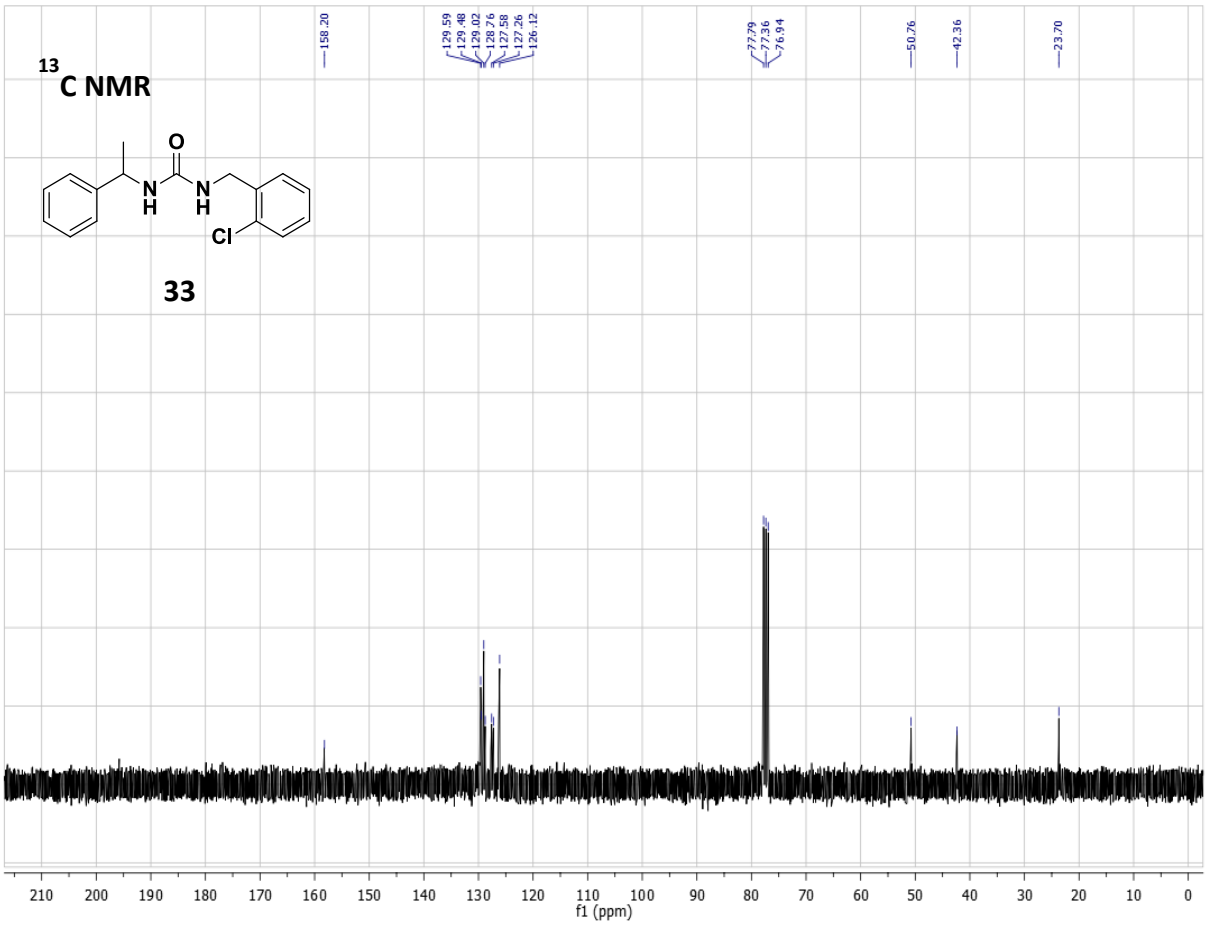
1.0
0.5
0.0
f1 (ppm)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129.02
128.76
127.58
127.26
126.12
77.79
77.36
76.94
50.76
42.36
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O
N H
N H
Cl
33
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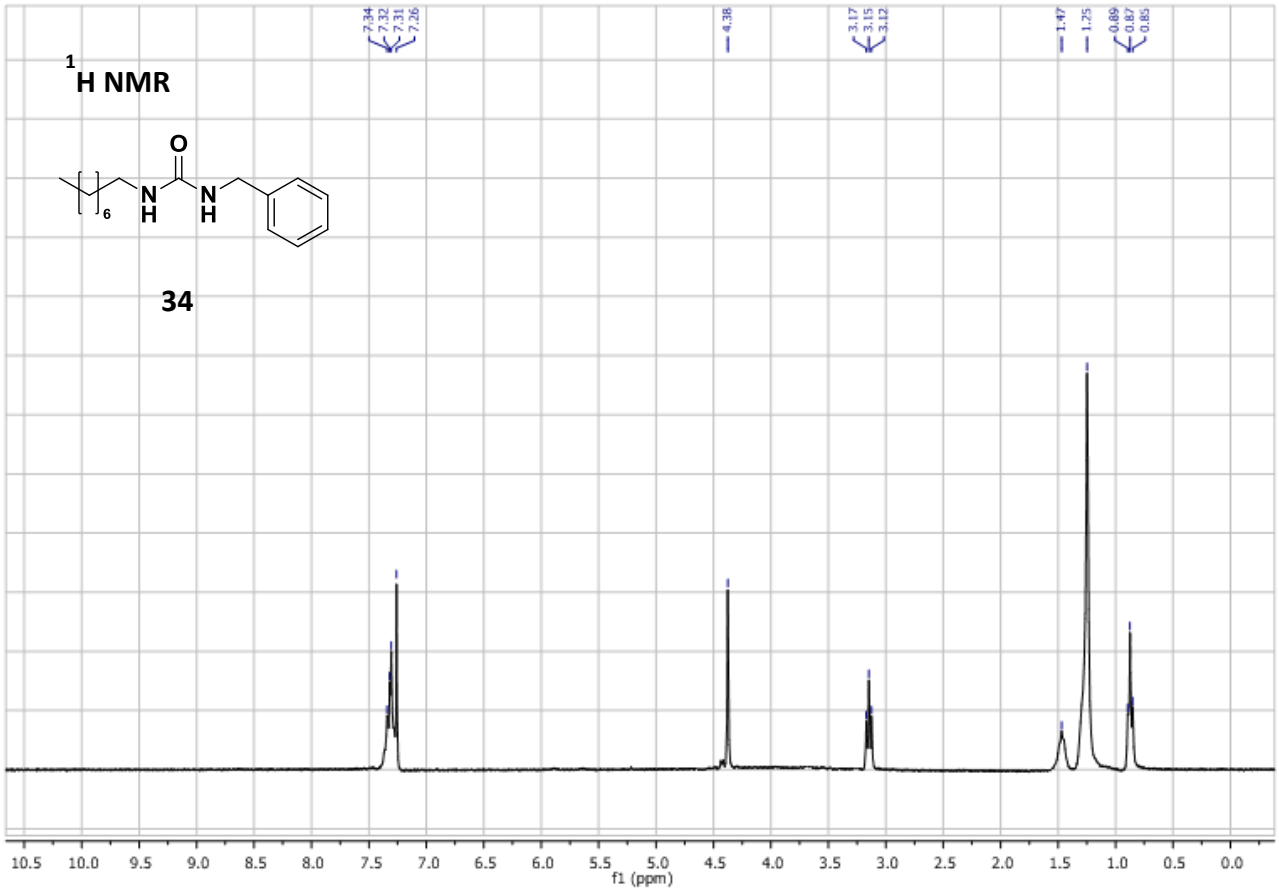
f1 (ppm)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7.31
7.26
4.38
3.17
3.15
3.12
1.47
1.25
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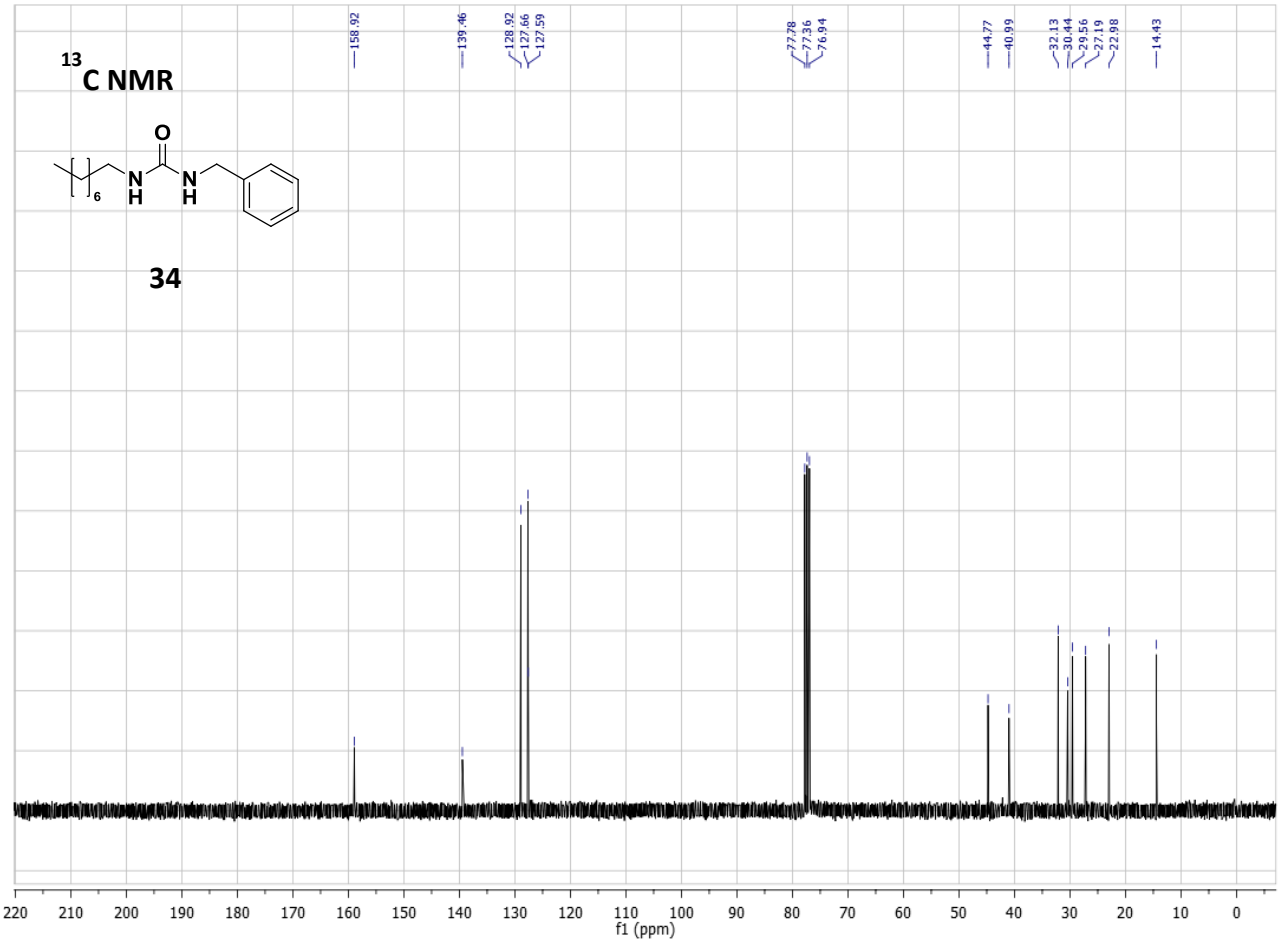
0.87
0.85
O
N H
N H
6
34
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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27.19
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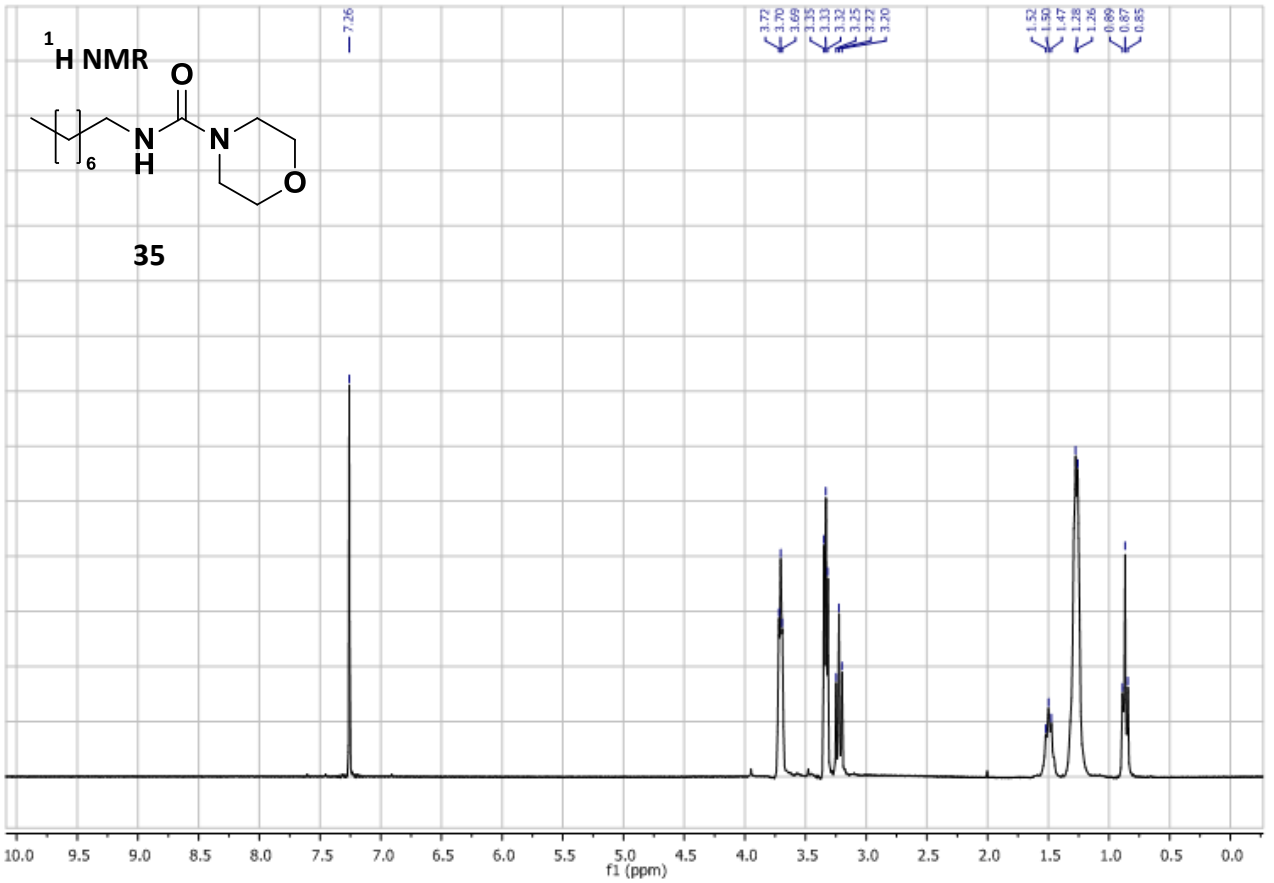
f1 (ppm)


1 H NMR
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3.33
3.32
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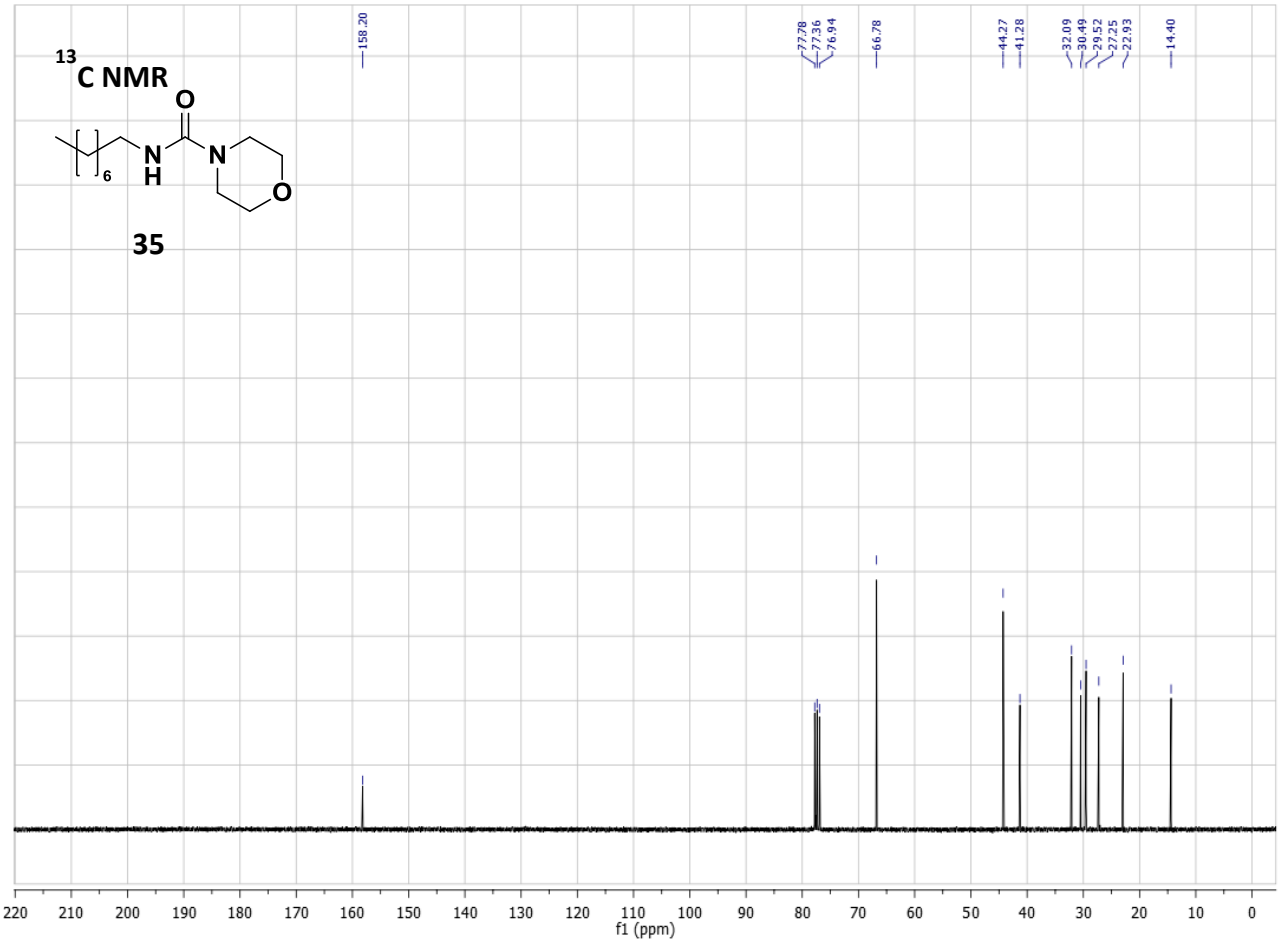
1.52
1.50
1.47
1.28
1.26
0.89
0.87
0.85
f1 (ppm)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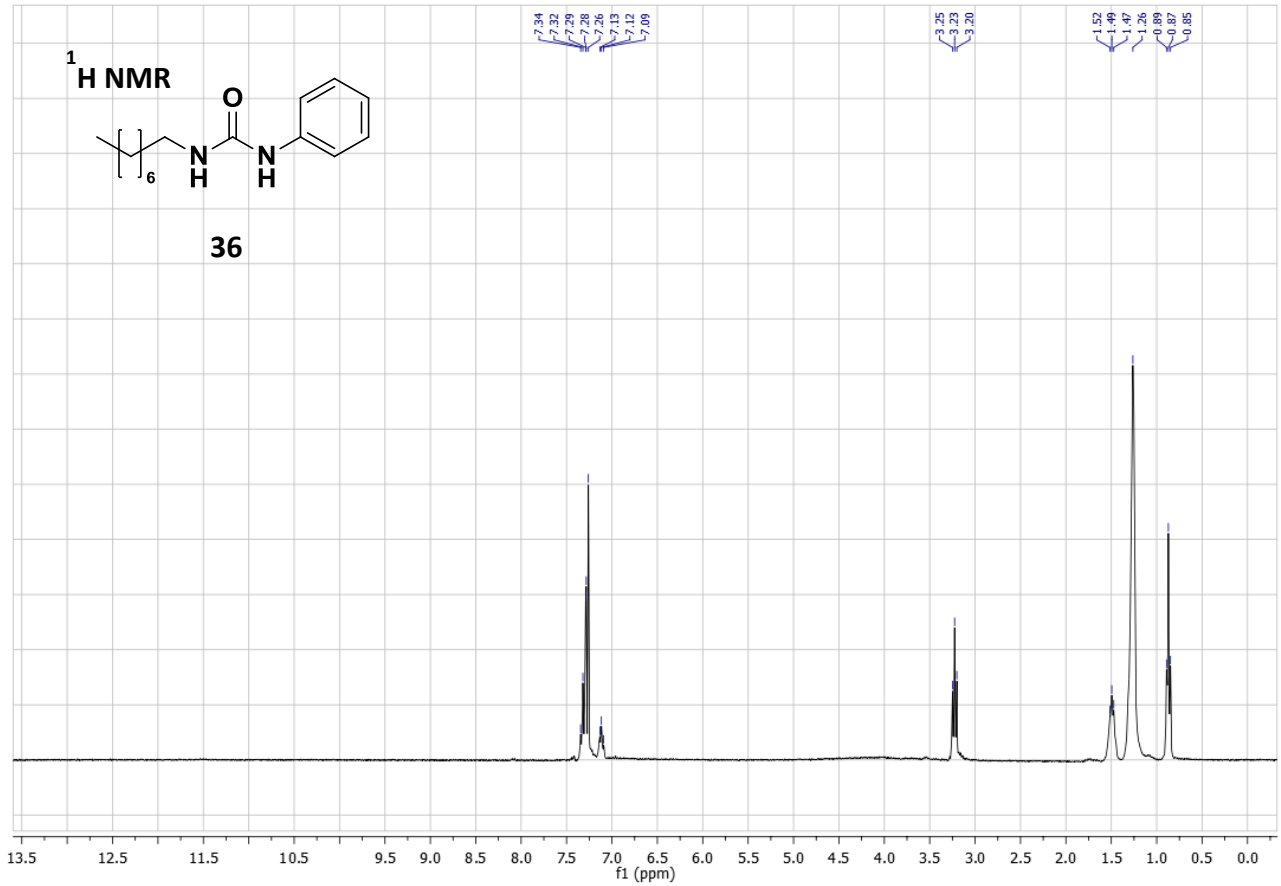
f1 (ppm)


1 H NMR
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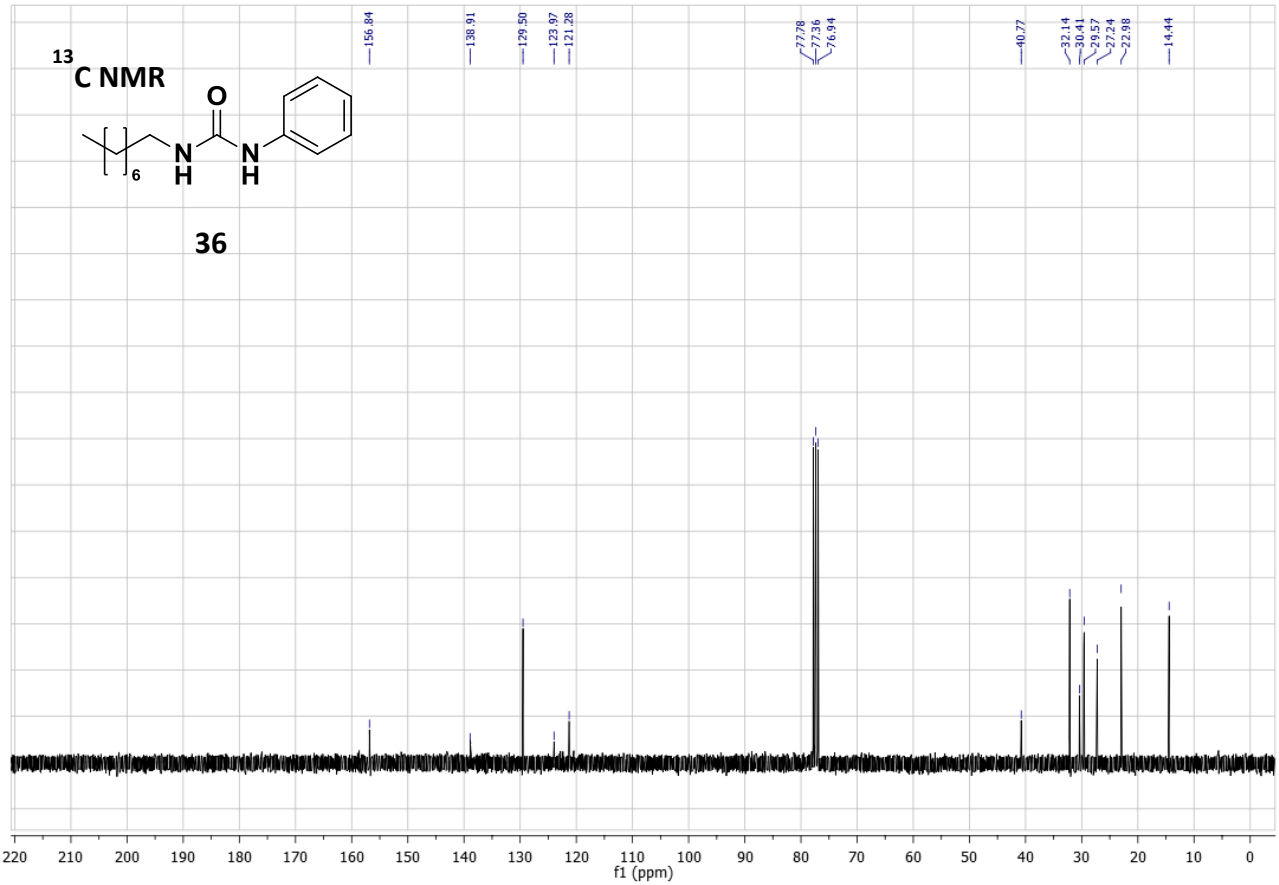
7.34
7.32
7.29
7.28
7.26
7.13
7.12
7.09
3.25
3.23
3.20
1.52
1.49
1.47
1.26
0.89
0.87
0.85
f1 (ppm)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40.77
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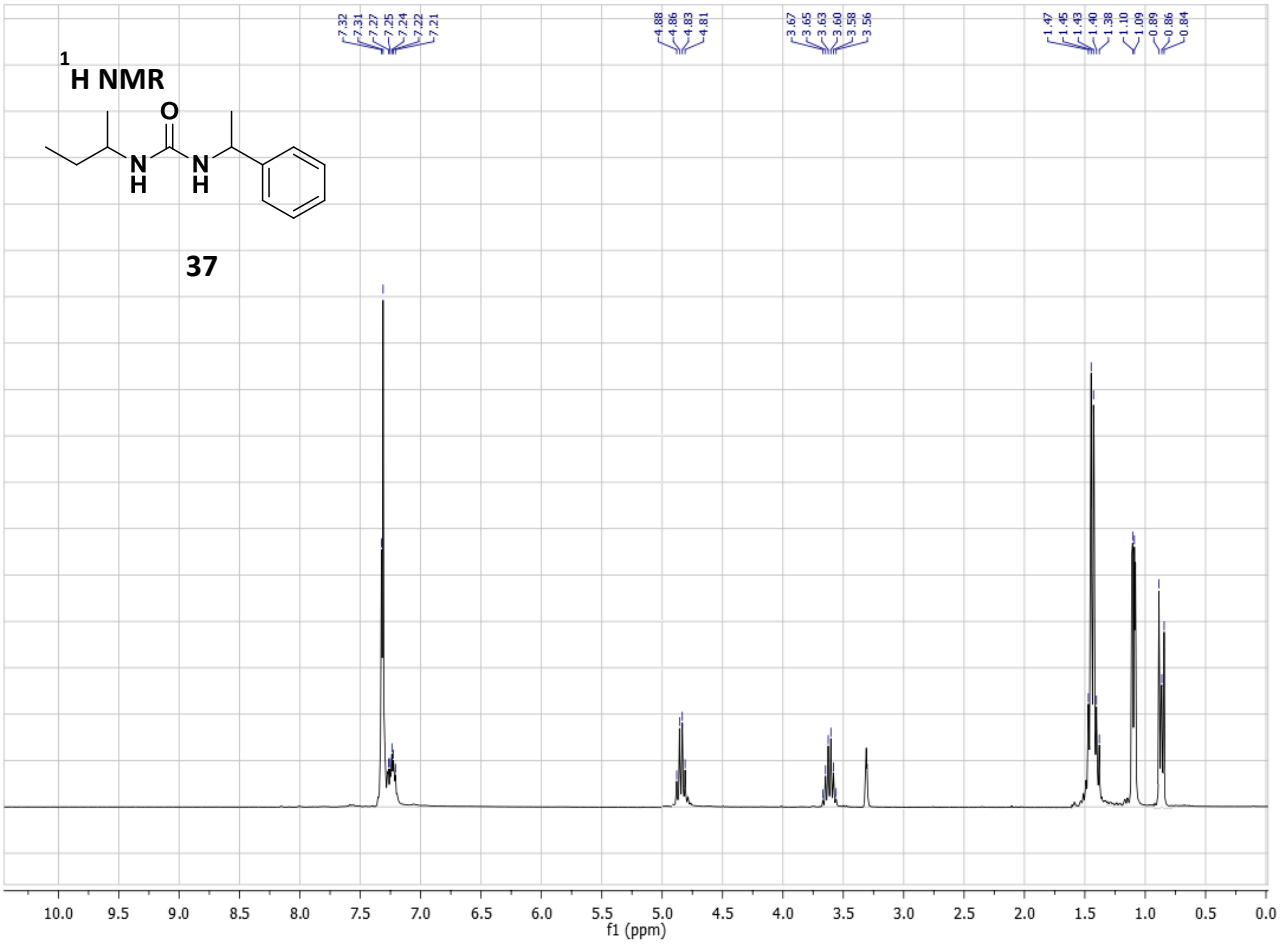
22.98
14.44
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


1H NMR
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7.25
7.24
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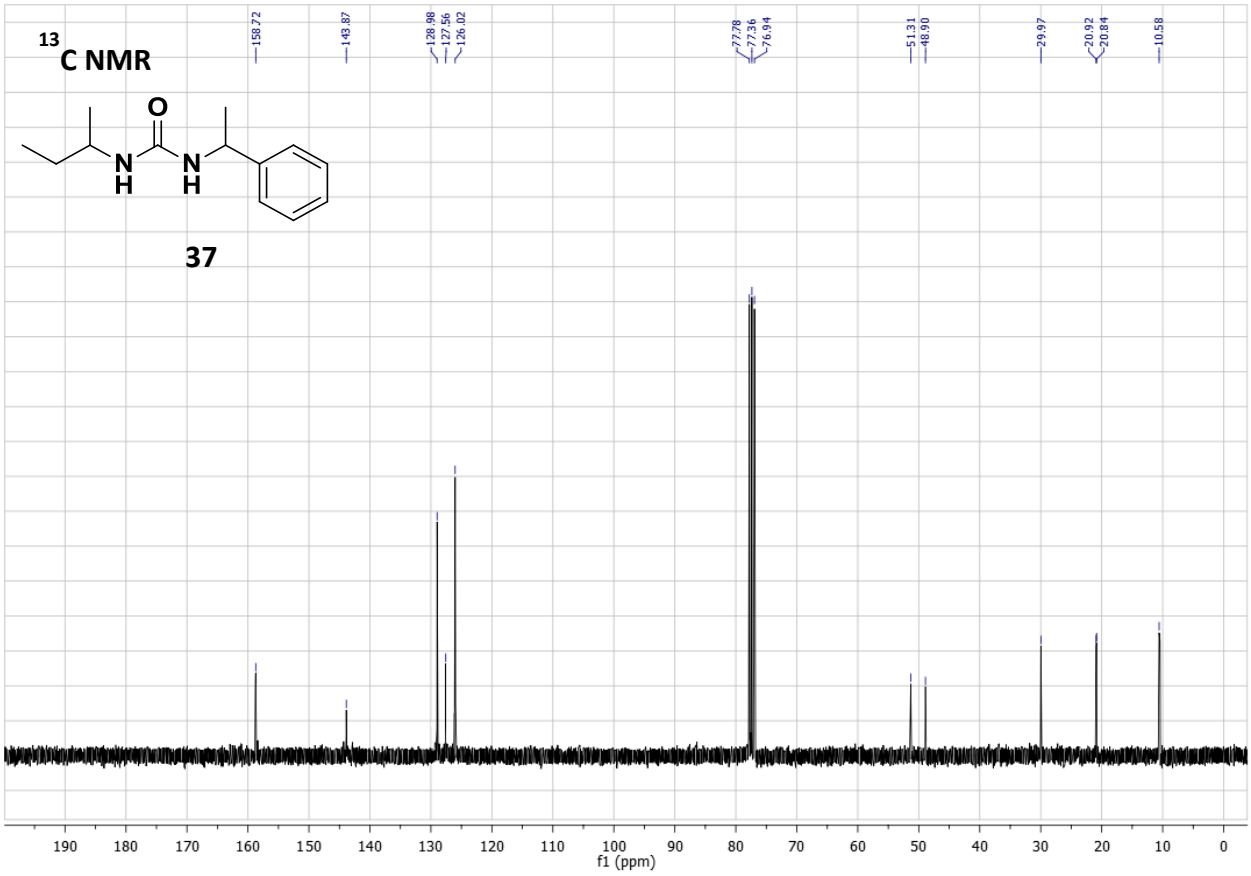
4.81
3.67
3.65
3.63
3.60
3.58
3.56
1.47
1.45
1.43
1.40
1.38
1.10
1.09
0.89
0.86
0.84
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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158.72
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126.02
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77.36
76.94
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20.92
20.84
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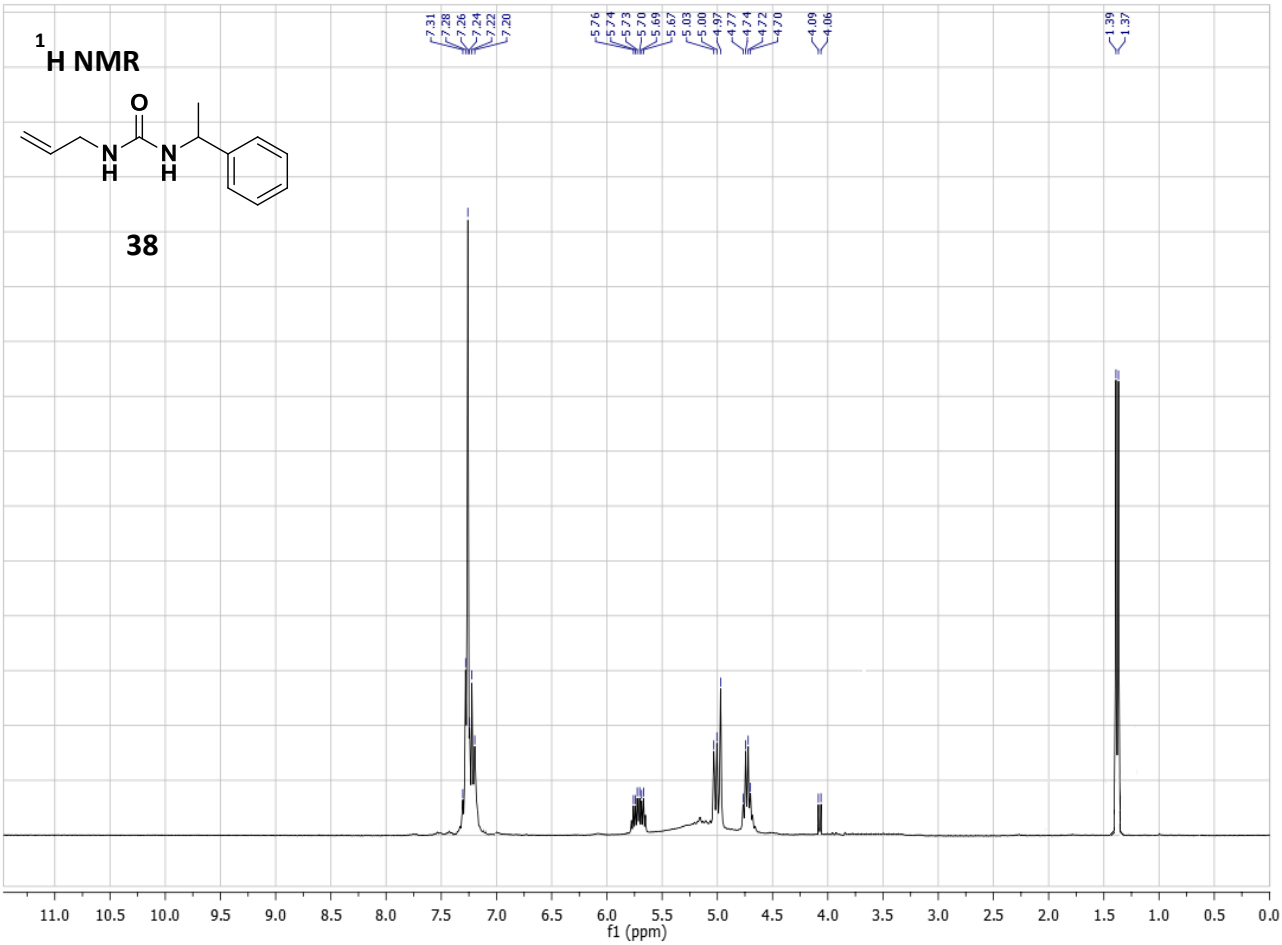
f1 (ppm)


1H NMR
38
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7.20
5.76
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5.73
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4.09
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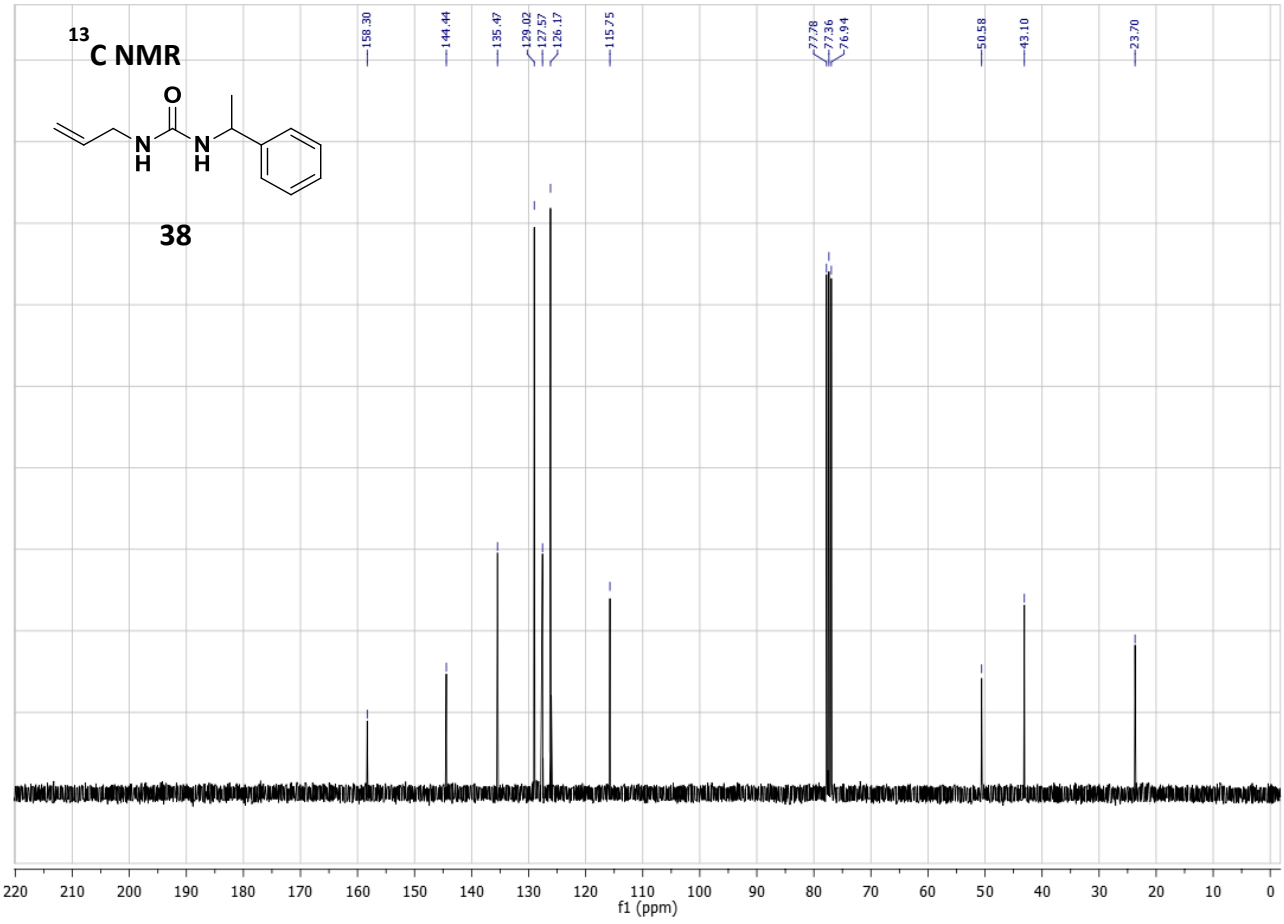
1.39
1.37
f1 (ppm)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129.02
127.57
126.17
115.75
77.78
77.36
76.94
50.58
43.10
23.70
f1 (ppm)

References

1. Tsopmo, A.; Ngnokam, D.; Ngamga, D.; Ayafor, J. F.; Sterner, O. *J. Nat. Prod.* **1999,** *62*, 1435–1436.
2. Park, J. H.; Yoon, J. C.; Chunga, Y. K. *Adv. Synth. Catal.* **2009,** *351*, 1233–1237.
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