Supporting information

Synthesis of Non-symmetrical Alkyl Carbonates from Alcohols and DMC over Nanocrystalline ZSM-5 Zeolite

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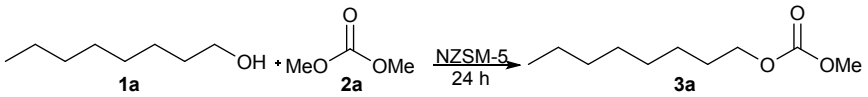
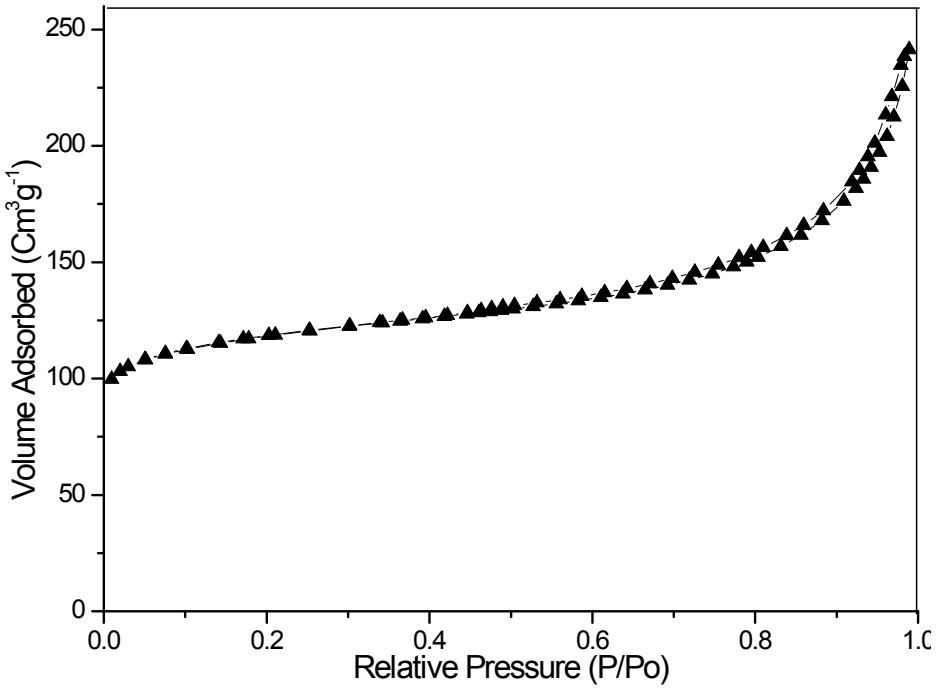
1. Table S1. Effect of amount of **2a**, amount of NZSM-5, and temperature on synthesis of non-symmetrical organic carbonates[a]



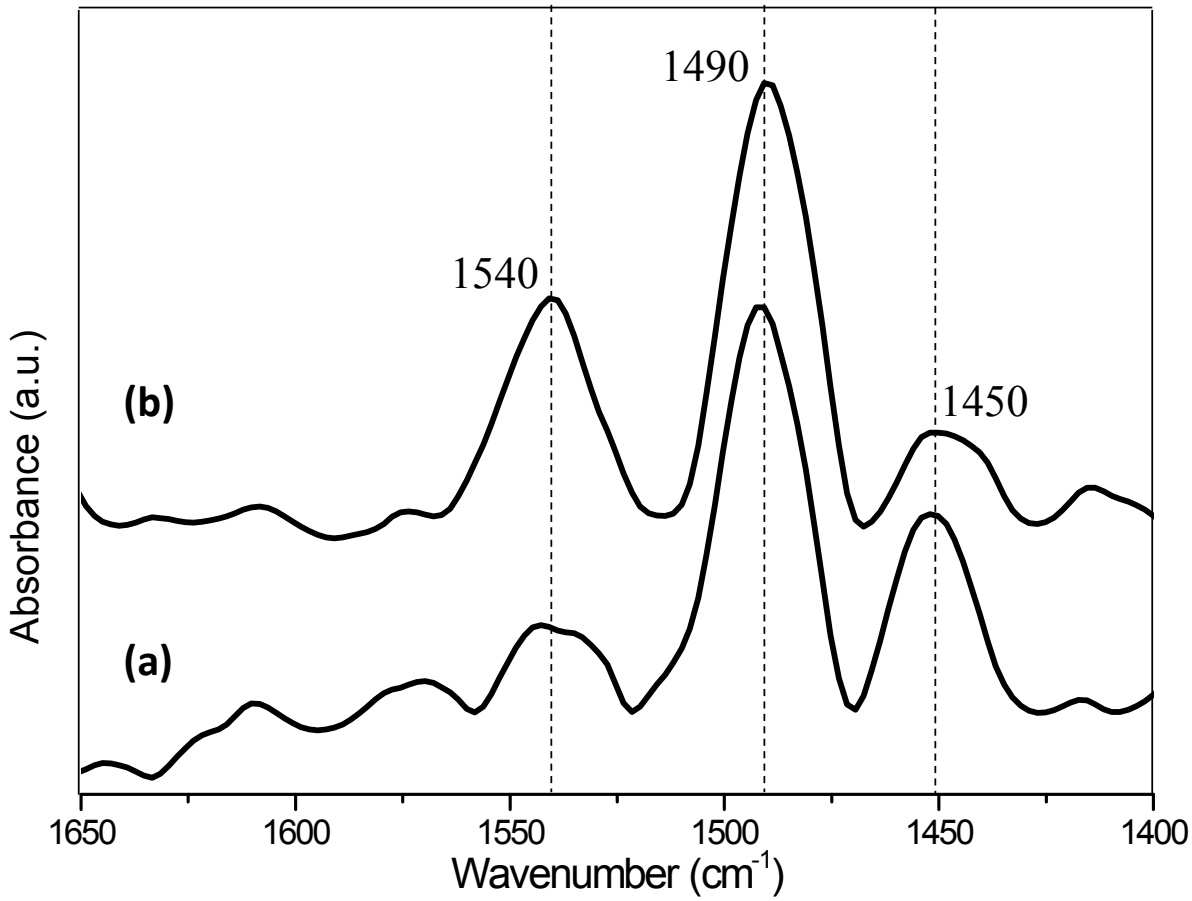
Entry	2a (mL)	NZSM-5 (mg)	Temperature (°C)	Yield[b] (%)
1	0.42	150	110	39
2	0.5	150	110	52
3	1	150	110	79
4	1.5	150	110	89
5	2	150	110	96
6	2	50	110	30
7	2	75	110	47
8	2	100	110	65
9	2	120	110	79
10	2	150	RT	00
11	2	150	50	5
12	2	150	75	18
13	2	150	90	58
14	2	150	100	69
15	2	150	110	95[c]

[a] **1a** (1 mmol). [b] Isolated yields. [c] Reaction conducted in 15 mL RB with condenser.

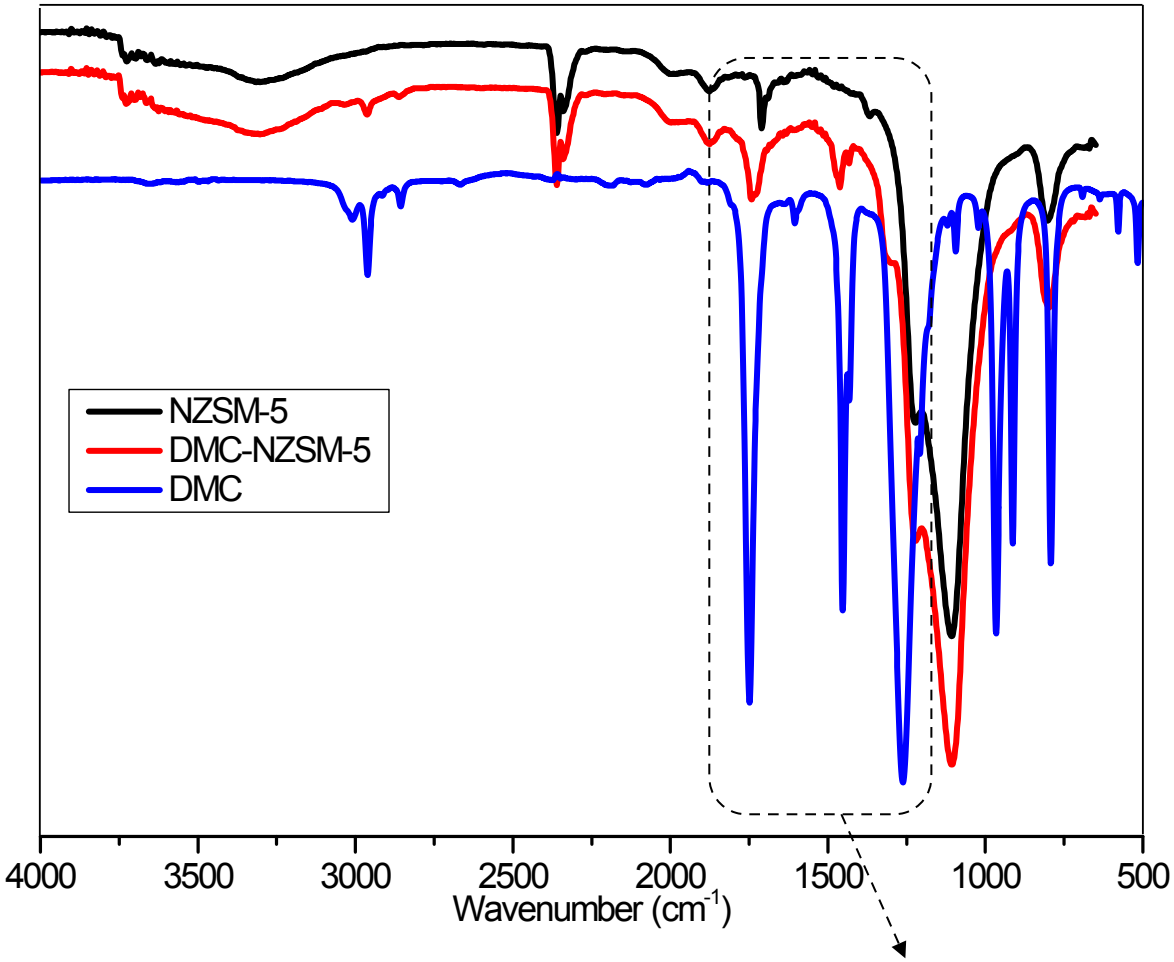
2. **Fig. S1** Nitrogen adsorption/desorption isotherms of NZSM-5

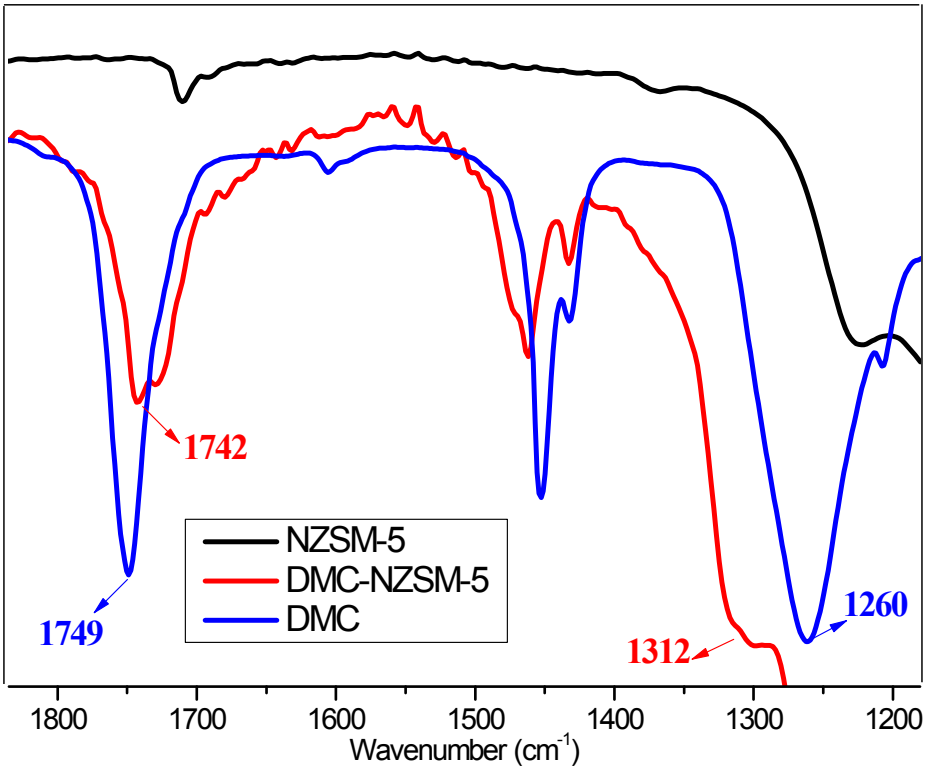


3. Fig. S2 Pyridine-IR spectra of (a) ZSM-5, (b) Synthesized NZSM-5

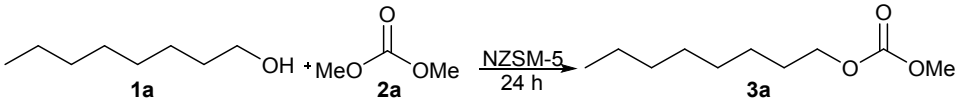


4. Fig. S3 FT-IR spectra of pure DMC, DMC adsorbed NZSM-5 and Pure NZSM-5





5. Table S2. Recyclability of the dried catalyst without calcination[a]



S. No.	Cycle	Yield[b] (%)
1	1st	96
2	2nd	89
3	3rd	86
4	4th	85
5	5th	80

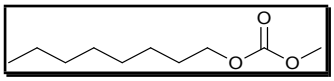
[a] Reaction conditions: 1-Octanol (**1a**, 1 mmol), DMC (2 mL), NZSM-5 (150 mg), 110 °C. [b] Isolated yields based on **1a**.

To regenerate, the NZSM-5 was filtered to separate from the reaction mixture and washed with ethyl acetate, dried at 100 °C for 8 h, without calcination. Recyclability of the NZSM-5 catalyst was tested by performing the reaction of **1a** with **2a** in optimized reaction parameters (Table S2). The activity of the NZSM-5 was decreased with increase in number of cycles, this may be due to the blocking of active sites inside the micropores of zeolite by water

and DMC. In case of recycled NZSM-5 catalyst with calcination showed consistent activity up to five cycles (Table 3).

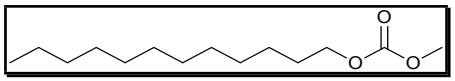
6. Spectroscopic Data

Methyl 1-octyl carbonate (3a)[1]



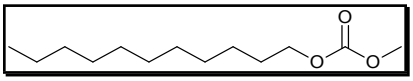
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ = 4.13 (t, J = 6.72 Hz, 2H), 3.78 (s, 3H), 1.71-1.60 (m, 2H), 1.42-1.21 (m, 10H), 0.88 (t, J = 6.72 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.87, 68.25, 54.60, 31.73, 29.12, 28.65, 25.66, 22.60, 14.05; HRMS: m/z calculated for $C_{10}H_{20}O_3$ $[M]^+$ 188.14, found: 188.1440.

1-Dodecyl methyl carbonate (3b)[3]



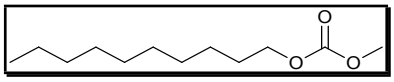
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.13 (t, J = 6.71 Hz, 2H), 3.78 (s, 3H), 1.71-1.61 (m, 2H), 1.41-1.23 (m, 18H), 0.88 (t, J = 6.86 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.87, 68.25, 54.59, 31.89, 29.59, 29.52, 29.46, 29.31, 29.18, 28.65, 25.65, 22.64, 14.09; HRMS: m/z calculated for $C_{14}H_{28}O_3$ $[M+H]^+$ 245.20, found: 245.0792.

Methyl 1-undecyl carbonate (3c)



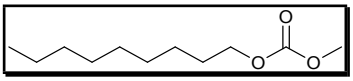
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.13 (t, J = 6.72 Hz, 2H), 3.78 (s, 3H), 1.71-1.61 (m, 2H), 1.40-1.23 (m, 16H), 0.88 (t, J = 6.86 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.88, 68.25, 54.59, 31.88, 29.56, 29.53, 29.46, 29.30, 29.19, 28.65, 25.66, 22.65, 14.08; HRMS: m/z calculated for $C_{13}H_{26}O_3[M+H]^+$ 231.18, found: 231.1966.

1-Decyl methyl carbonate (3d)



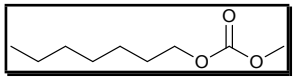
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ = 4.13 (t, J = 6.72 Hz, 2H), 3.78 (s, 3H), 1.71-1.60 (m, 2H), 1.41-1.22 (m, 14H), 0.88 (t, J = 6.72 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.86, 68.26, 54.60, 31.85, 29.46, 29.26, 29.19, 28.65, 25.66, 22.64, 14.08; HRMS: m/z calculated for $C_{12}H_{24}O_3$ $[M]^+$ 216.17, found: 216.1745.

Methyl 1-nonyl carbonate (3e)[2]



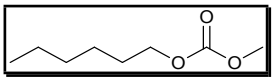
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.13 (t, J = 6.71 Hz, 2H), 3.78 (s, 3H), 1.71-1.61 (m, 2H), 1.40-1.23 (m, 12H), 0.88 (t, J = 6.86 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.87, 68.25, 54.60, 31.82, 29.42, 29.19, 28.65, 25.66, 22.64, 14.08; HRMS: m/z calculated for $C_{11}H_{22}O_3$ $[M+H]^+$ 203.16, found: 203.1061.

1-Heptyl methyl carbonate (3f)[2]



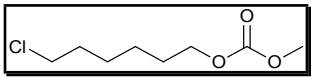
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.13 (t, J = 6.72 Hz, 2H), 3.78 (s, 3H), 1.71-1.61 (m, 2H), 1.41-1.22 (m, 8H), 0.88 (t, J = 6.86 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.86, 68.23, 54.59, 31.65, 28.84, 28.64, 25.60, 22.53, 14.01; HRMS: m/z calculated for $C_9H_{18}O_3$ $[M]^+$ 174.13, found: 174.1298.

1-Hexyl methyl carbonate (3g)[1]



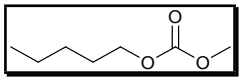
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.13 (t, J = 6.71 Hz, 2H), 3.78 (s, 3H), 1.71-1.61 (m, 2H), 1.41-1.22 (m, 6H), 0.89 (t, J = 6.86 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.87, 68.25, 54.60, 31.36, 28.61, 25.33, 22.48, 13.95; HRMS: m/z calculated for $C_8H_{16}O_3$ $[M+H]^+$ 161.11, found: 161.0427.

6-Chloro-1-hexyl methyl carbonate (3h)



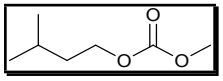
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.14 (t, J = 6.56 Hz, 2H), 3.78 (s, 3H), 3.53 (t, J = 6.56 Hz, 2H), 1.83-1.73 (m, 2H), 1.72-1.64 (m, 2H), 1.52-1.36 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.80, 67.89, 54.63, 44.84, 32.37, 28.49, 26.43, 25.01; HRMS: m/z calculated for $C_8H_{15}O_3Cl$ $[M+H]^+$ 197.07, found: 197.1039.

Methyl 1-pentyl carbonate (3i)[1]



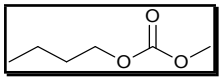
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ = 4.13 (t, J = 6.72 Hz, 2H), 3.78 (s, 3H), 1.72-1.61 (m, 2H), 1.39-1.24 (m, 4H), 0.90 (t, J = 6.96 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.87, 68.22, 54.60, 28.33, 27.78, 22.26, 13.89; HRMS: m/z calculated for $C_7H_{14}O_3$, 147.09 $[M+H]^+$ found: 147.0514.

Isopentyl methyl carbonate (3j)[3]



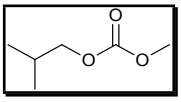
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.17 (t, J = 6.86 Hz, 2H), 3.78 (s, 3H), 1.76-1.68 (m, 1H), 1.59-1.53 (m, 2H), 0.93 (d, J = 6.56 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.85, 66.71, 54.60, 37.28, 24.76, 22.37; HRMS: m/z calculated for $C_7H_{14}O_3$ $[M+H]^+$ 147.09, found: 147.1158.

1-Butyl methyl carbonate (3k)[1]



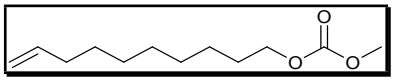
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ = 4.14 (t, J = 6.60 Hz, 2H), 3.78 (s, 3H), 1.71-1.60 (m, 2H), 1.47-1.33 (m, 2H), 0.94 (t, J = 7.33 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.85, 67.89, 54.55, 30.63, 18.85, 13.58; HRMS: m/z calculated for $C_6H_{12}O_3$ $[M]^+$ 132.08, found: 132.0468.

Isobutyl methyl carbonate (3l)



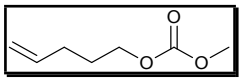
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 3.92 (d, J = 6.71 Hz, 2H), 3.78 (s, 3H), 2.02-1.91 (m, 1H), 0.95 (d, J = 6.71 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.90, 74.12, 54.61, 27.75, 18.84; HRMS: m/z calculated for $C_6H_{12}O_3$ $[M+H]^+$ 133.08, found: 133.0468.

Dec-9-en-1-yl methyl carbonate (3m)



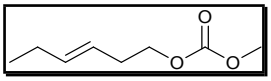
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ = 5.87-5.72 (m, 1H), 5.04-4.90 (m, 2H), 4.13 (t, J = 6.72 Hz, 2H), 3.78 (s, 3H), 2.08-2.00 (m, 2H), 1.71-1.60 (m, 2H), 1.45-1.24 (m, 10H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.87, 139.13, 114.14, 68.23, 54.61, 33.75, 29.29, 29.12, 28.98, 28.85, 28.64, 25.64; HRMS: m/z calculated for $C_{12}H_{22}O_3$ $[M+H]^+$ 125.18, found: 215.1306.

Methyl pent-4-en-1-yl carbonate (3n)[4]



^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 5.86-5.73 (m, 1H), 5.08-4.96 (m, 2H), 4.15 (t, J = 6.56 Hz, 2H), 3.78 (s, 3H), 2.19-2.10 (m, 2H), 1.82-1.72 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.77, 137.17, 115.41, 67.40, 54.64, 29.72, 27.77; HRMS: m/z calculated for $C_7H_{12}O_3$ $[M]^+$ 144.08, found: 144.0798.

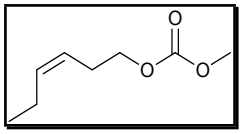
(*E*)-Hex-3-en-1-yl methyl carbonate (3o)



^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 5.62-5.53 (m, 1H), 5.42-5.31 (m, 1H), 4.14 (t, J = 7.02 Hz, 2H), 3.78 (s, 3H), 2.39-2.31 (m, 2H), 2.06-1.96 (m, 2H), 0.97 (t, J = 7.48 Hz, 3H); ^{13}C

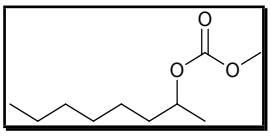
NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.75, 135.43, 123.42, 67.67, 54.63, 31.93, 25.56, 13.62; HRMS: m/z calculated for $C_8H_{14}O_3$ $[M]^+$ 158.09, found: 158.1204.

(*Z*)-Hex-3-en-1-yl methyl carbonate (3p)



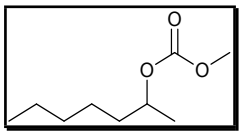
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ = 5.57-5.47 (m, 1H), 5.37-5.26 (m, 1H), 4.13 (t, J = 6.96 Hz, 2H), 3.78 (s, 3H), 2.47-2.37 (m, 2H), 2.11-2.00 (m, 2H), 0.97 (t, J = 7.46 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.79, 134.95, 123.00, 67.44, 54.64, 26.73, 20.58, 14.13; HRMS: m/z calculated for $C_8H_{14}O_3$ $[M]^+$ 158.09, found: 158.1204.

Methyl 2-octyl carbonate (3q)[3]



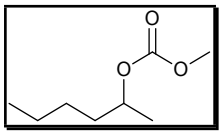
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.80-4.71 (m, 1H), 3.76 (s, 3H), 1.68-1.60 (m, 1H), 1.54-1.45 (m, 1H), 1.38-1.24 (m, 11H), 0.88 (t, J = 6.72 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.47, 75.52, 54.41, 35.86, 31.66, 29.06, 25.21, 22.54, 19.87, 14.03; HRMS: m/z calculated for $C_{10}H_{20}O_3[M]^+$ 188.14, found: 188.1443.

2-Heptyl methyl carbonate (3r)[1]



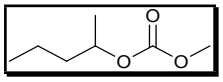
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ = 4.81-4.71 (m, 1H), 3.76 (s, 3H), 1.69-1.59 (m, 1H), 1.55-1.44 (m, 1H), 1.38-1.24 (m, 9H), 0.88 (t, J = 6.72 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, ppm): δ = 155.45, 75.48, 54.37, 35.79, 31.56, 24.89, 22.47, 19.83, 13.93; HRMS: m/z calculated for $C_9H_{18}O_3$ $[M]^+$ 174.13, found: 174.1298.

2-Hexyl methyl carbonate (3s)



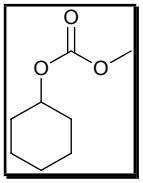
^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ = 4.81-4.71 (m, 1H), 3.77 (s, 3H), 1.70-1.60 (m, 1H), 1.56-1.46 (m, 1H), 1.37-1.24 (m, 7H), 0.90 (t, J = 6.82 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.46, 75.49, 54.40, 35.54, 27.39, 22.47, 19.86, 13.91; HRMS: m/z calculated for $C_8H_{16}O_3$ $[M+H]^+$ 161.11, found: 161.0968.

Methyl 2-pentyl carbonate (3t)



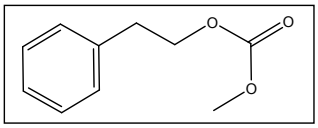
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.81-4.73 (m, 1H), 3.76 (s, 3H), 1.68-1.59 (m, 1H), 1.53-1.44 (m, 1H), 1.43-1.32 (m, 2H), 1.27 (d, J = 6.26 Hz, 3H), 0.92 (t, J = 7.32 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.47, 75.25, 54.42, 37.95, 29.67, 19.87, 16.50, 13.83; HRMS: m/z calculated for $C_7H_{14}O_3$ $[M]^+$ 146.09, found: 146.1158.

Cyclohexyl methyl carbonate (3u)[1]



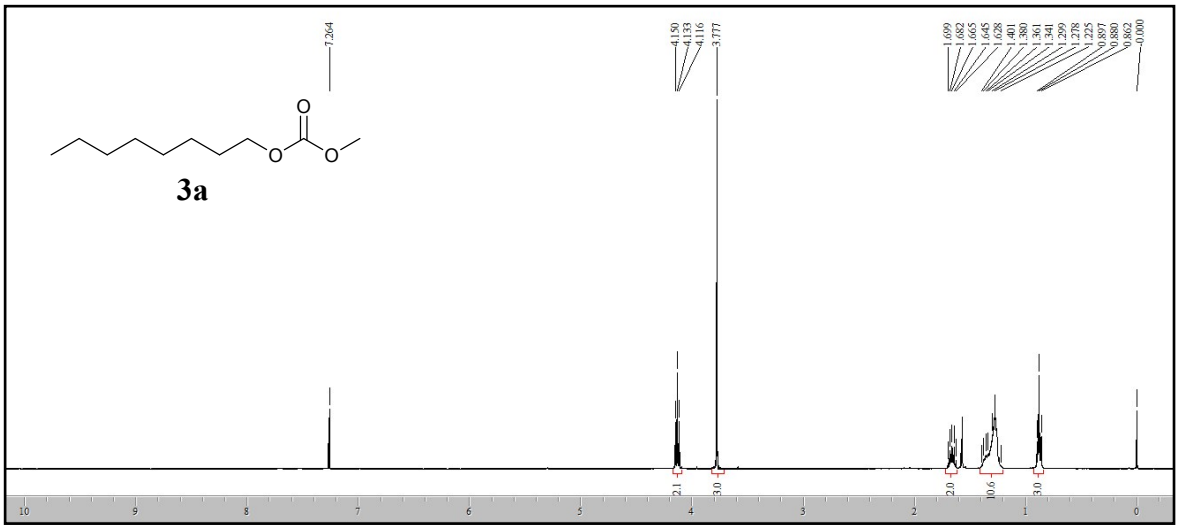
^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ = 4.66-4.57 (m, 1H), 3.77 (s, 3H), 1.95-1.89 (m, 2H), 1.79-1.71 (m, 2H), 1.58-1.43 (m, 3H), 1.41-1.24 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ = 155.23, 76.67, 54.39, 31.51, 25.19, 23.60; HRMS: m/z calculated for $C_8H_{14}O_3$ $[M+H]^+$ 159.09, found: 159.0426.

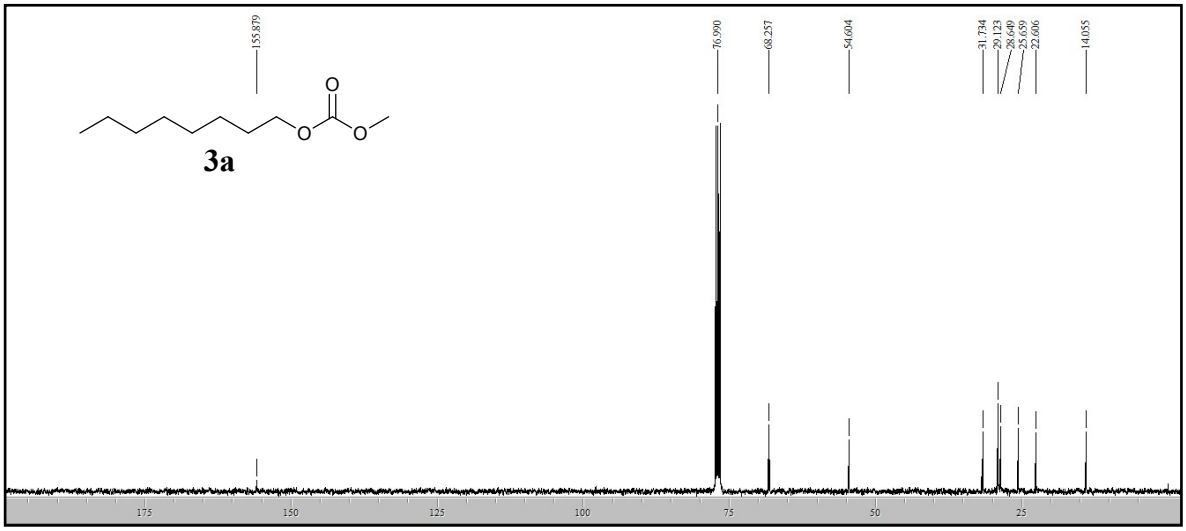
Methyl (2-phenyl)ethyl carbonate (3x)[5]



^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.28-7.34 (m, 2H), 7.21-7.26 (m, 3H), 4.34 (t, 2H, J = 7.21 Hz), 3.76 (s, 3H), 2.98 (t, 2H, J = 7.21 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 155.93, 137.44, 129.13, 128.77, 126.90, 68.61, 54.94, 35.36.

7. Copies of ^{1}H and ^{13}C NMR Spectra

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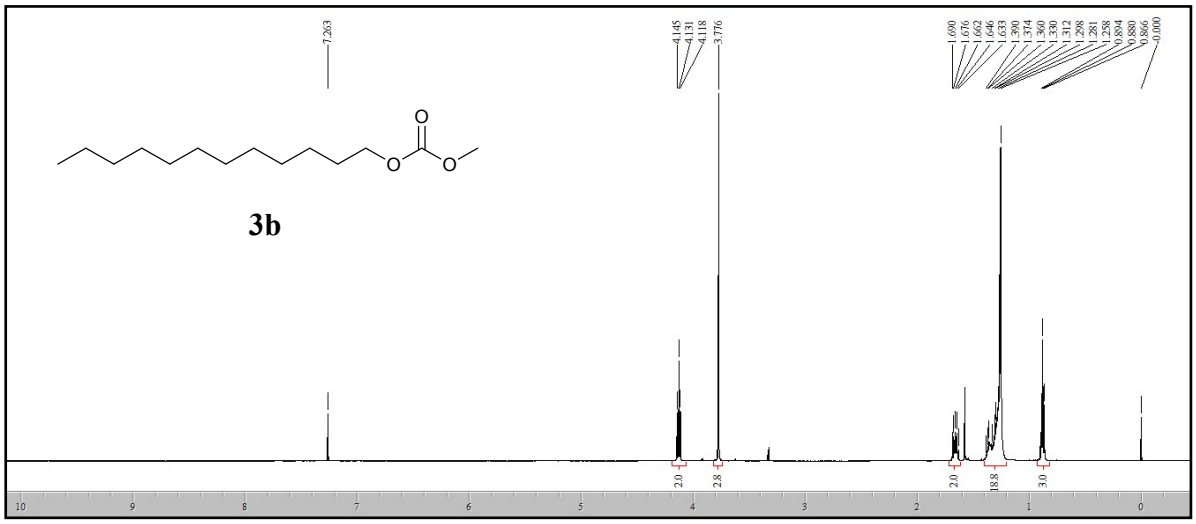
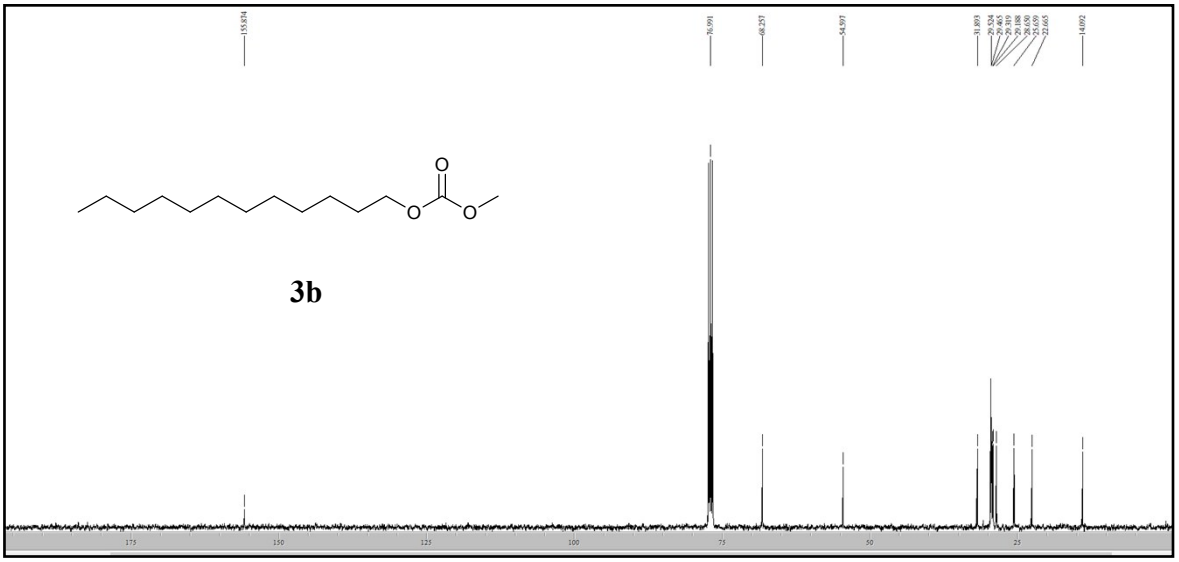
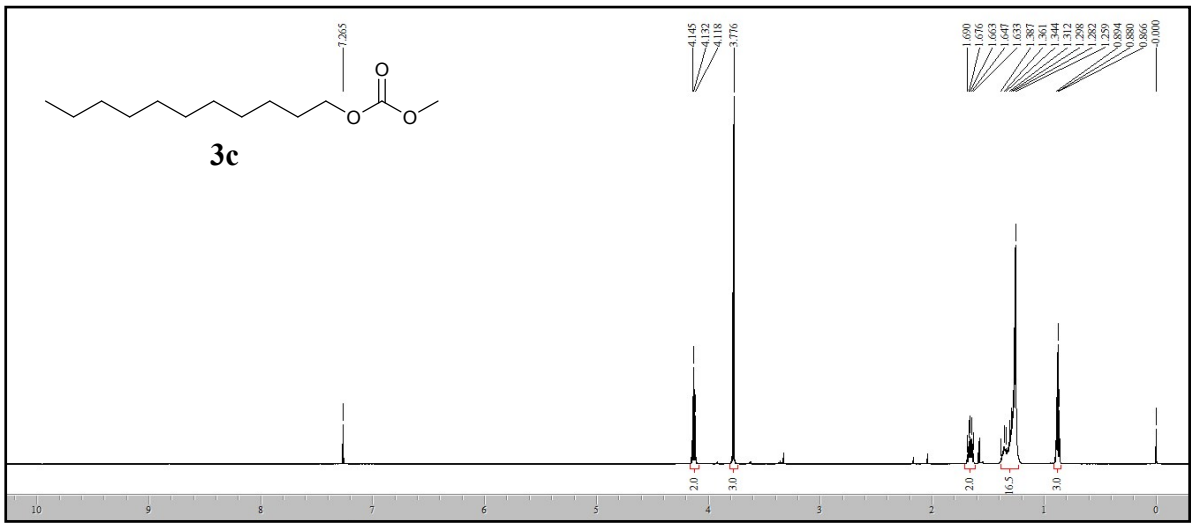
76.990
68.257
54.604
31.734
29.123
28.649
25.659
22.606
14.055
3a
175
150
125
100
75
50
25


7.263
4.145
4.131
4.118
3.776
1.690
1.676
1.662
1.649
1.635
1.300
1.374
1.360
1.330
1.312
1.298
1.281
1.258
0.894
0.880
0.866
0.000
3b
2.0
2.8
2.0
18.8
3.0
10
9
8
7
6
5
4
3
2
1
0


155.834
76.991
68.257
54.507
31.893
29.524
29.465
29.391
29.188
29.049
25.639
22.665
14.092
O
O
O
3b
175
150
125
100
75
50
25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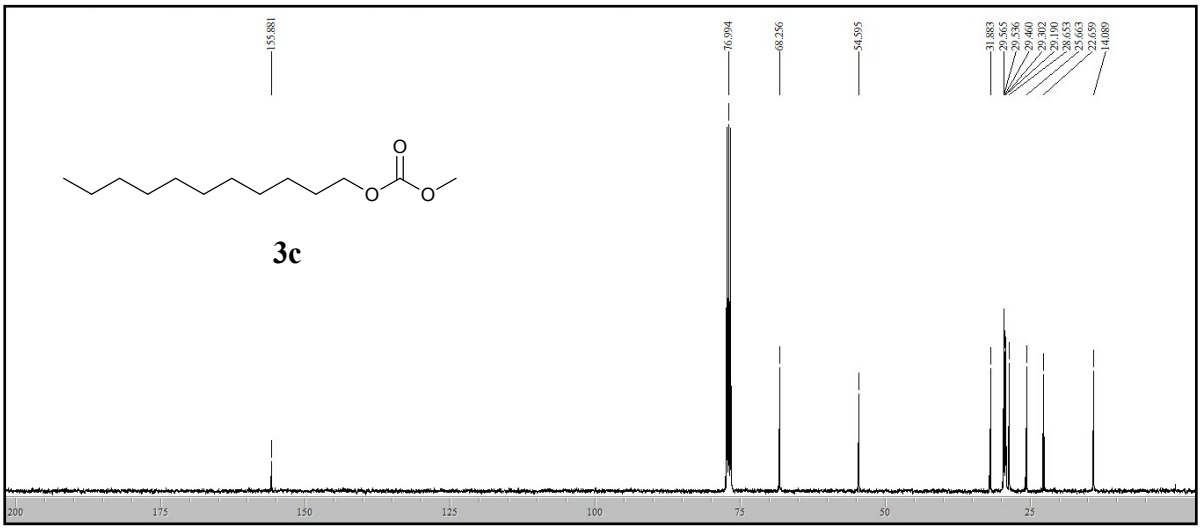
4.145
4.132
4.118
3.776
1.690
1.676
1.663
1.647
1.633
1.387
1.361
1.344
1.312
1.298
1.282
1.259
0.884
0.869
0.866
0.000
O
O
O
3c
2.0
3.0
2.0
16.5
3.0
10
9
8
7
6
5
4
3
2
1
0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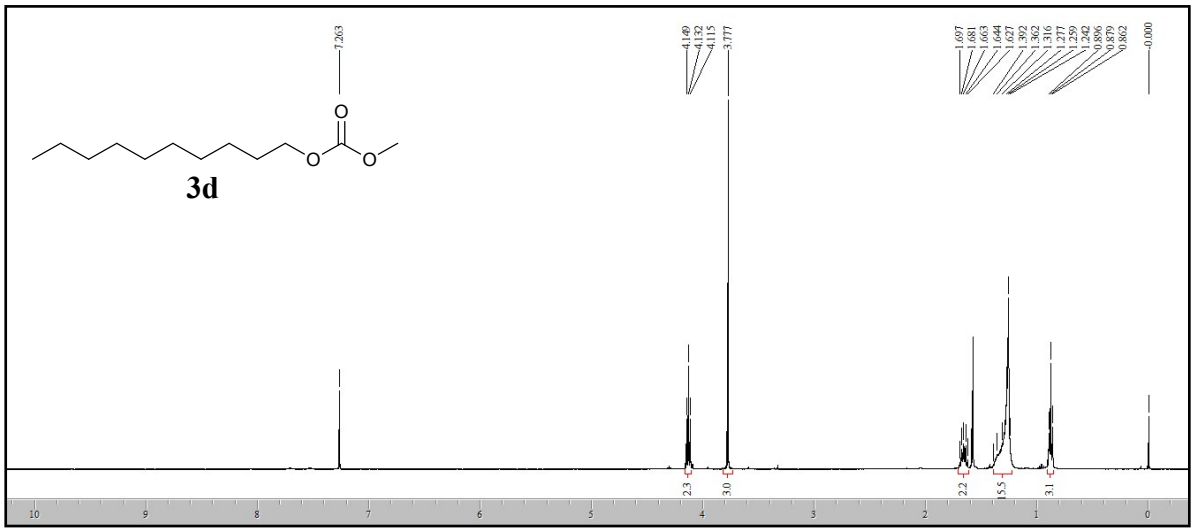
76.994
68.256
54.595
31.883
29.565
29.536
29.460
29.307
29.190
28.653
25.663
22.659
14.089
O
O
O
3c
200
175
150
125
100
75
50
25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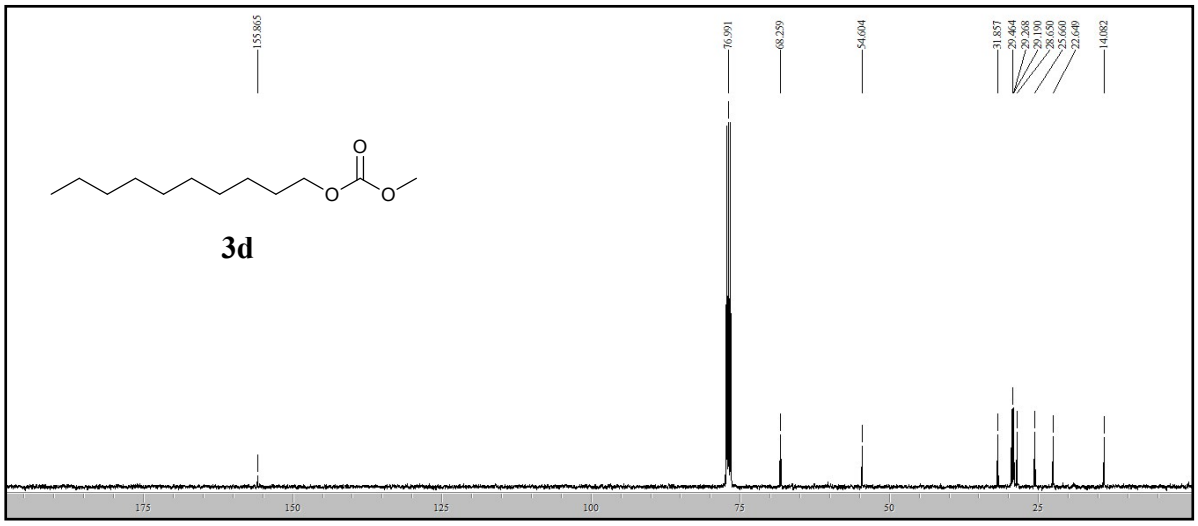
4.149
4.132
4.115
3.777
1.697
1.681
1.663
1.644
1.627
1.392
1.362
1.316
1.277
1.259
1.242
0.896
0.879
0.862
0.000
O
O
O
3d
2.3
3.0
2.2
15.5
3.1
10
9
8
7
6
5
4
3
2
1
0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54.604
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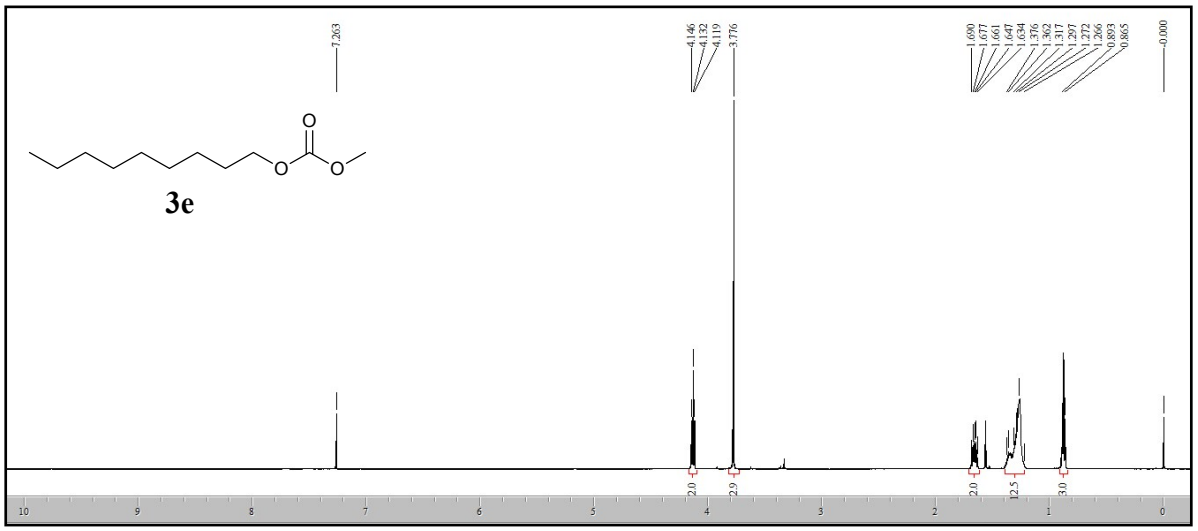
29.464
29.268
29.190
28.650
25.660
22.649
14.082
O
O
O
3d
175
150
125
100
75
50
25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4.132
4.119
3.776
1.690
1.677
1.661
1.647
1.634
1.376
1.362
1.317
1.297
1.272
1.266
0.893
0.865
0.000
O
O
O
3e
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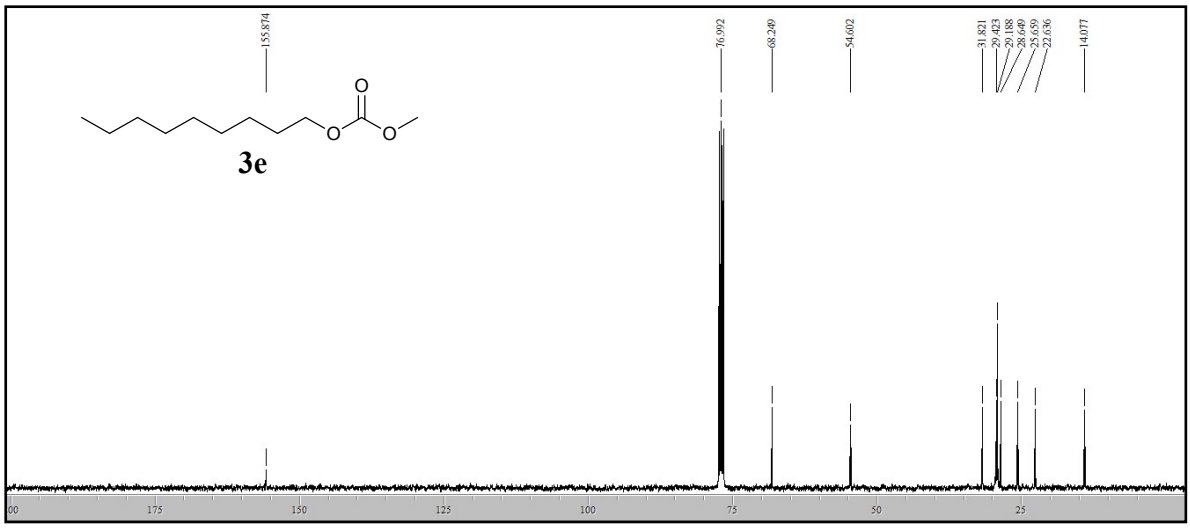
2.9
2.0
12.5
3.0
10
9
8
7
6
5
4
3
2
1
0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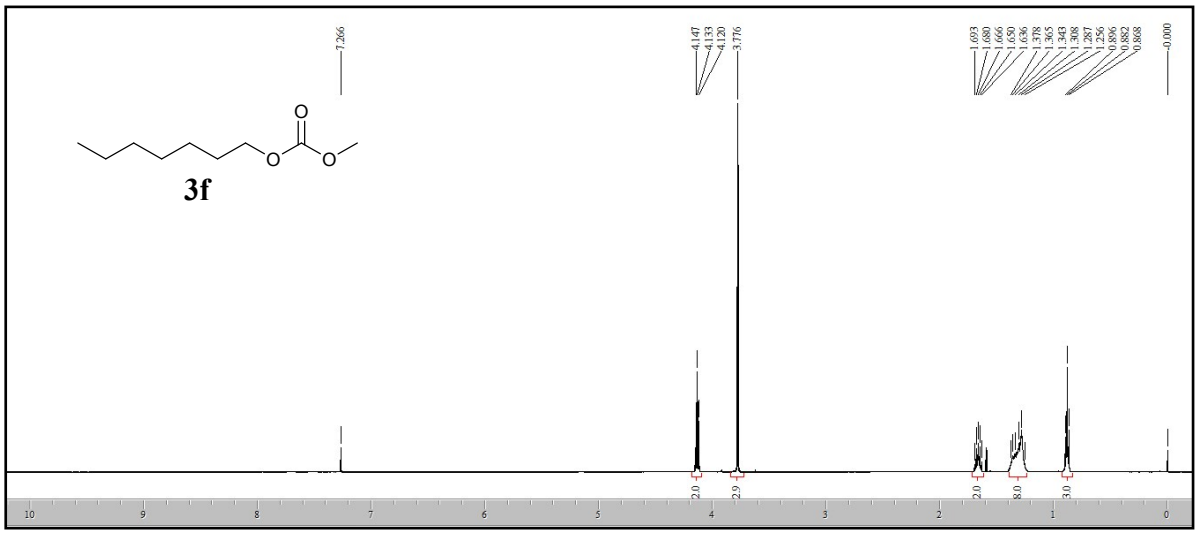
68.249
54.602
31.821
29.423
29.188
28.649
25.639
22.656
14.077
3e
175
150
125
100
75
50
25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4.147
4.133
4.120
3.776
1.693
1.680
1.666
1.650
1.636
1.378
1.365
1.343
1.308
1.287
1.256
0.896
0.882
0.868
0.000
3f
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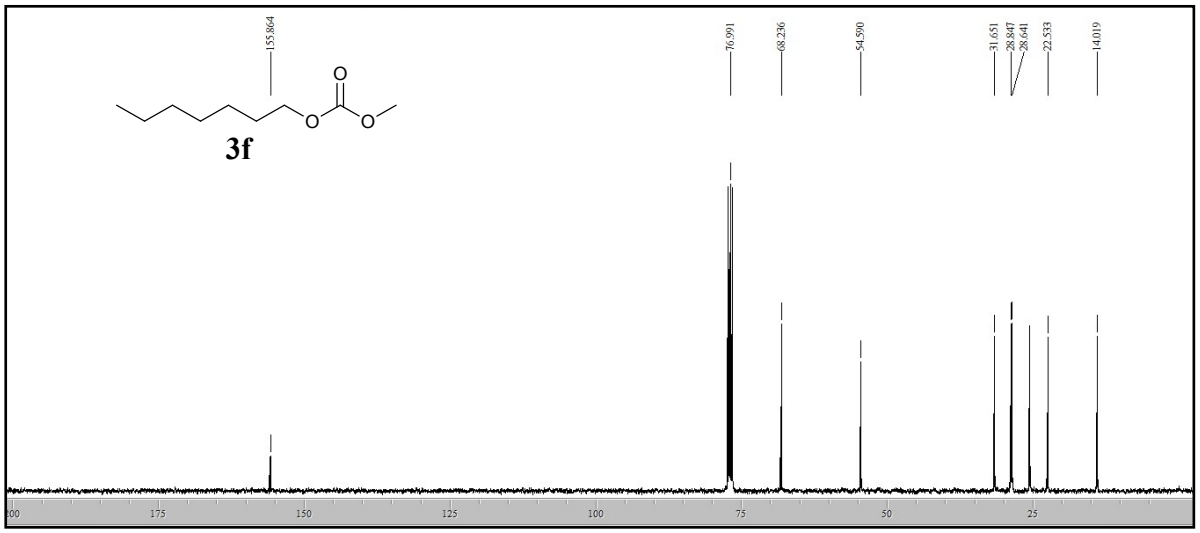
2.9
2.0
8.0
3.0
10
9
8
7
6
5
4
3
2
1
0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28.847
28.641
22.533
14.019
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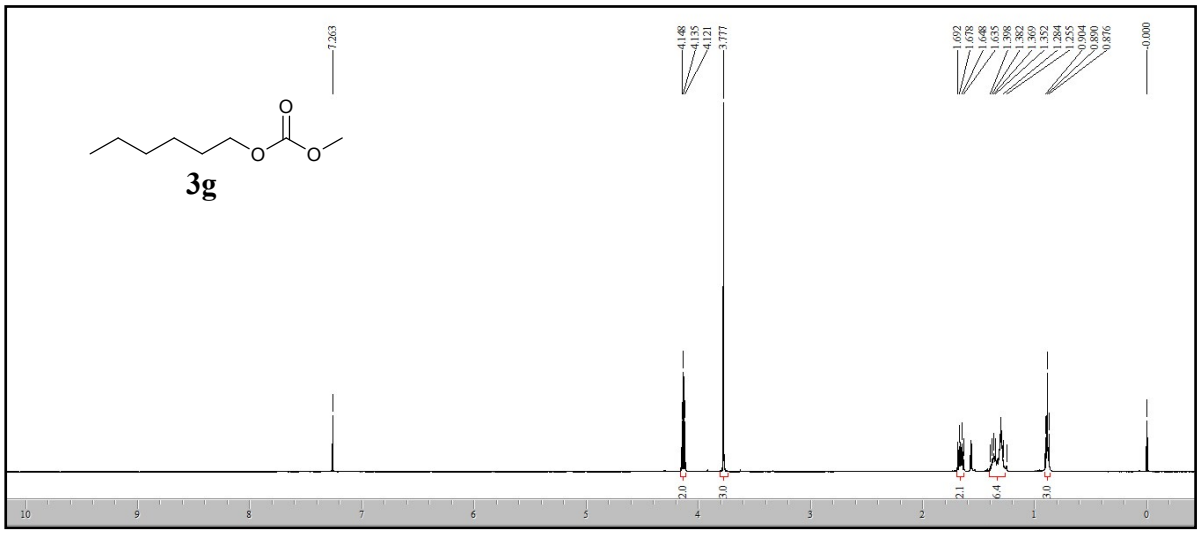
200
175
150
125
100
75
50
25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4.148
4.135
4.121
3.777
1.692
1.678
1.648
1.635
1.398
1.382
1.369
1.352
1.284
1.255
0.904
0.890
0.876
0.000
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2.1
6.4
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10
9
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7
6
5
4
3
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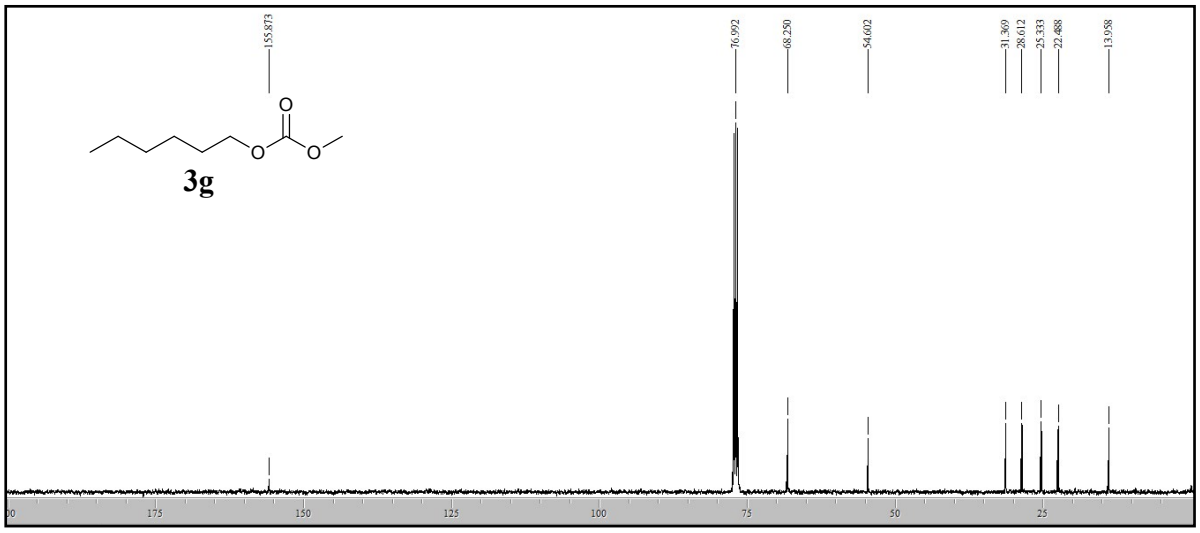
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68.250
54.602
31.369
28.612
25.333
22.488
13.958
O
O
O
3g
175
150
125
100
75
50
25


7.266
4.158
4.145
4.132
3.781
3.548
3.535
3.522
1.812
1.799
1.785
1.770
1.757
1.719
1.705
1.691
1.677
1.664
1.507
1.492
1.477
1.463
1.448
1.438
1.422
1.409
1.381
0.000
Cl
O
O
O
3h
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2.0
2.1
2.1
4.2
9
8
7
6
5
4
3
2
1
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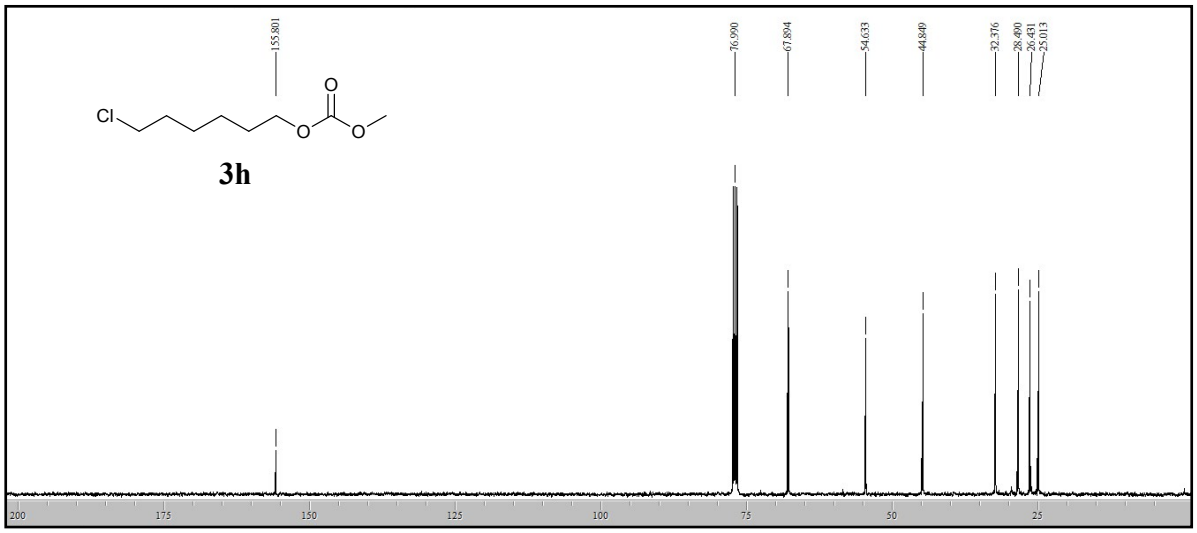
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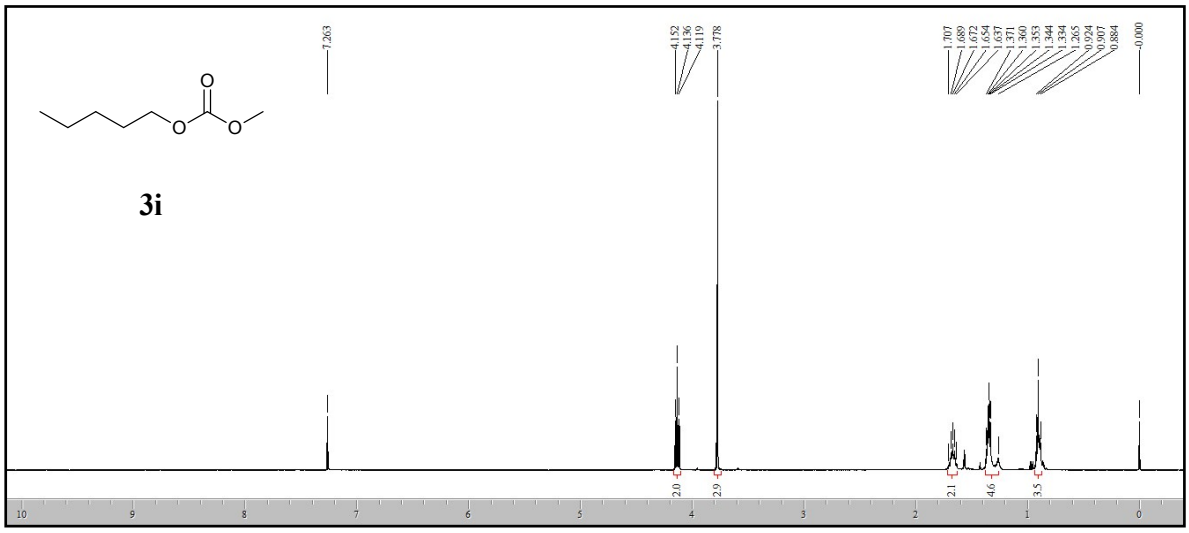
67.894
54.633
44.849
32.376
28.400
26.431
25.013
Cl
O
O
O
3h
200
175
150
125
100
75
50
25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4.119
3.778
1.707
1.689
1.672
1.654
1.637
1.371
1.360
1.353
1.344
1.334
1.325
0.904
0.907
0.884
0.000
O
O
O
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2.1
4.6
3.5
10
9
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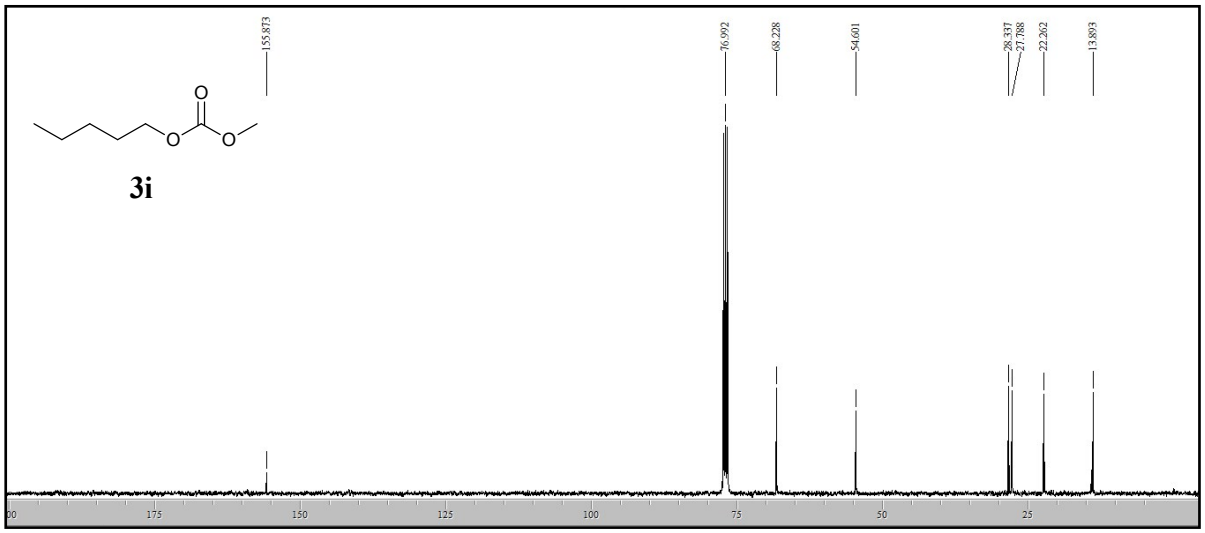
7
6
5
4
3
2
1
0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27.788
22.262
13.893
3i
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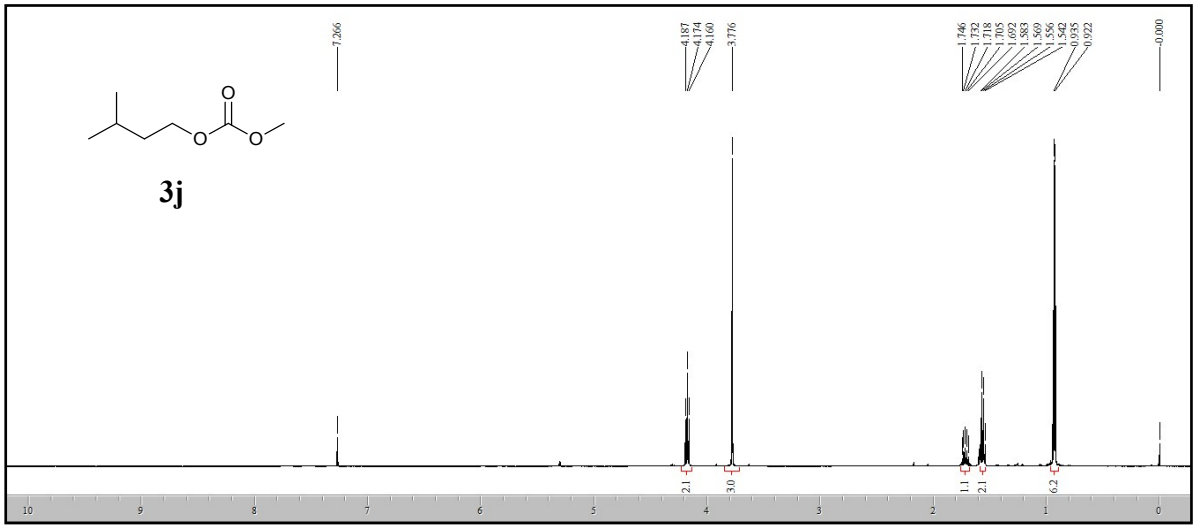
150
125
100
75
50
25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3.776
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1.732
1.718
1.705
1.692
1.583
1.569
1.556
1.542
1.095
0.922
0.000
3j
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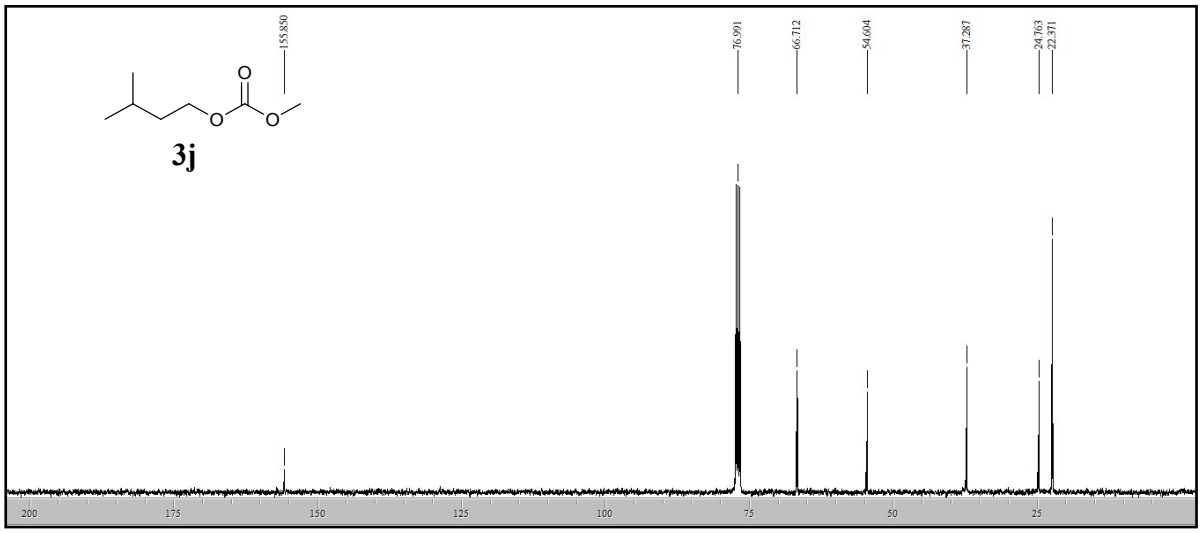
1.1
2.1
6.2
10
9
8
7
6
5
4
3
2
1
0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66.712
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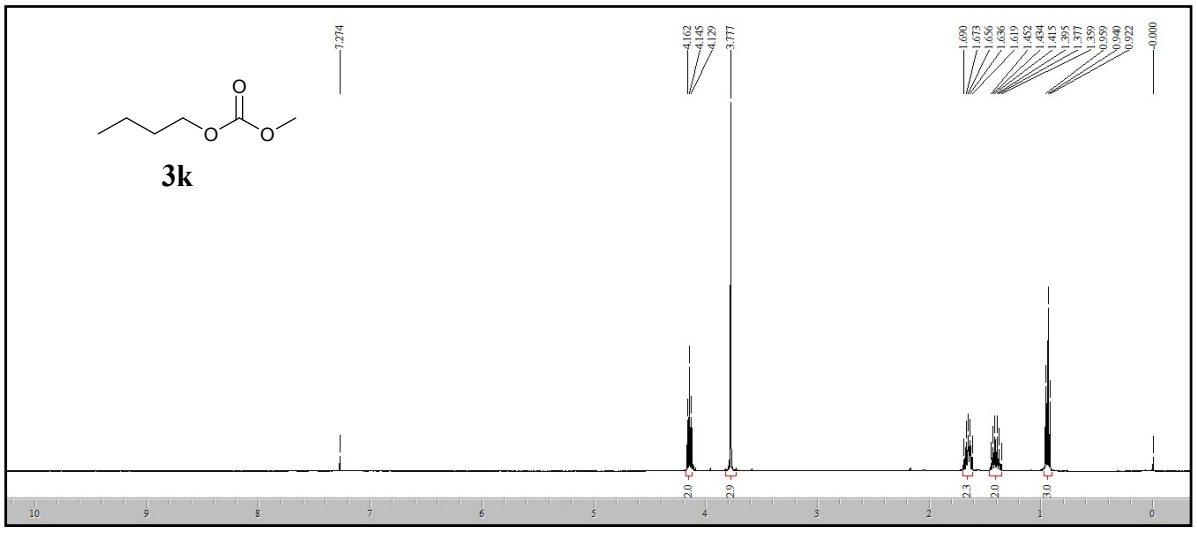
37.287
24.763
22.371
200
175
150
125
100
75
50
25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1.673
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1.636
1.619
1.452
1.434
1.415
1.395
1.377
1.359
0.949
0.940
0.922
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3.0
10
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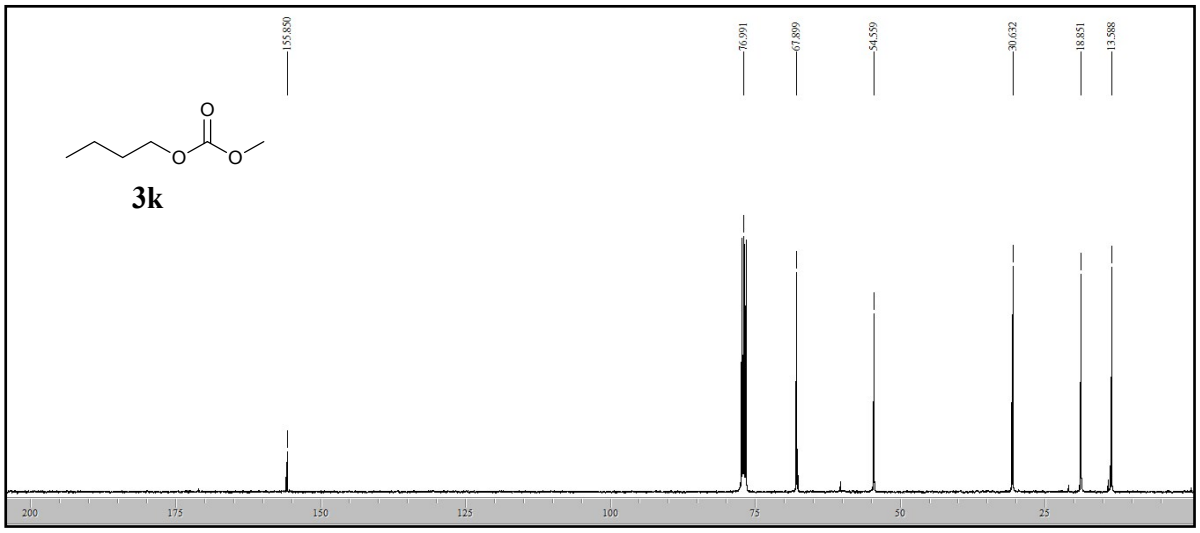
7
6
5
4
3
2
1
0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13.588
3k
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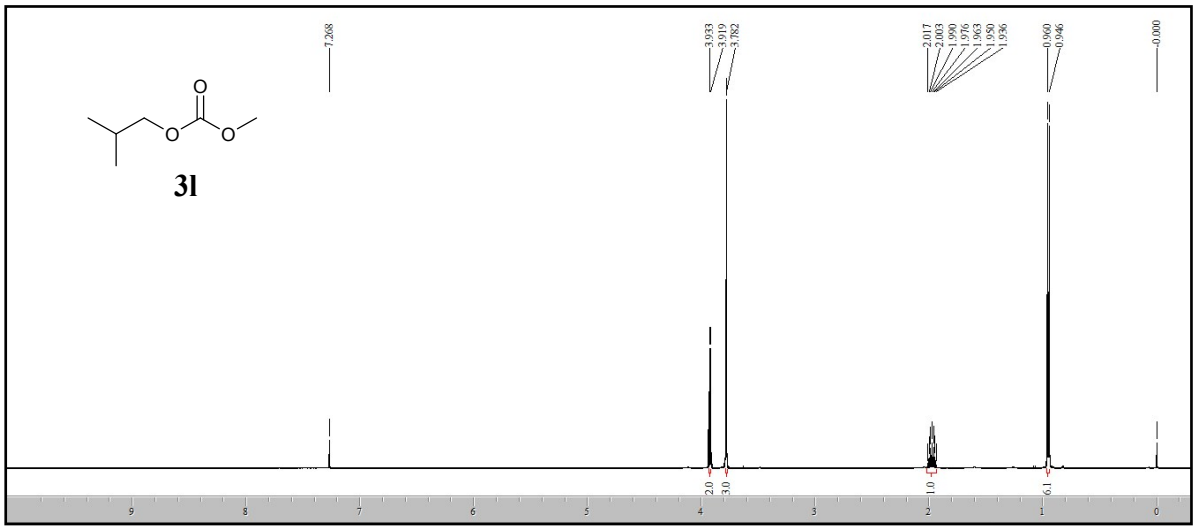
175
150
125
100
75
50
25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3.782
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1.990
1.976
1.963
1.950
1.936
0.960
0.946
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1.0
6.1
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7
6
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3
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1
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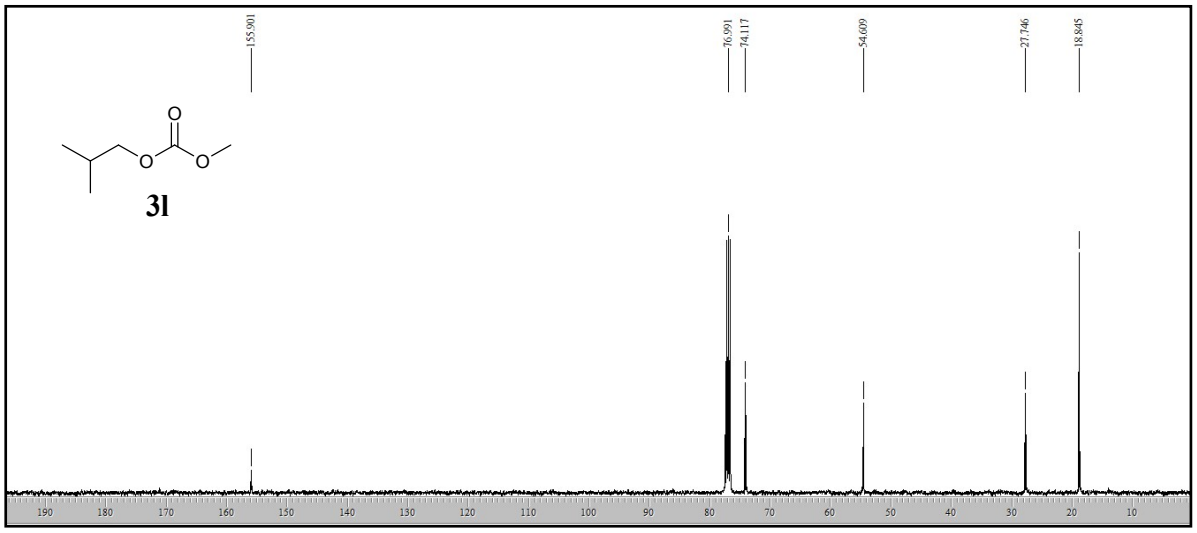
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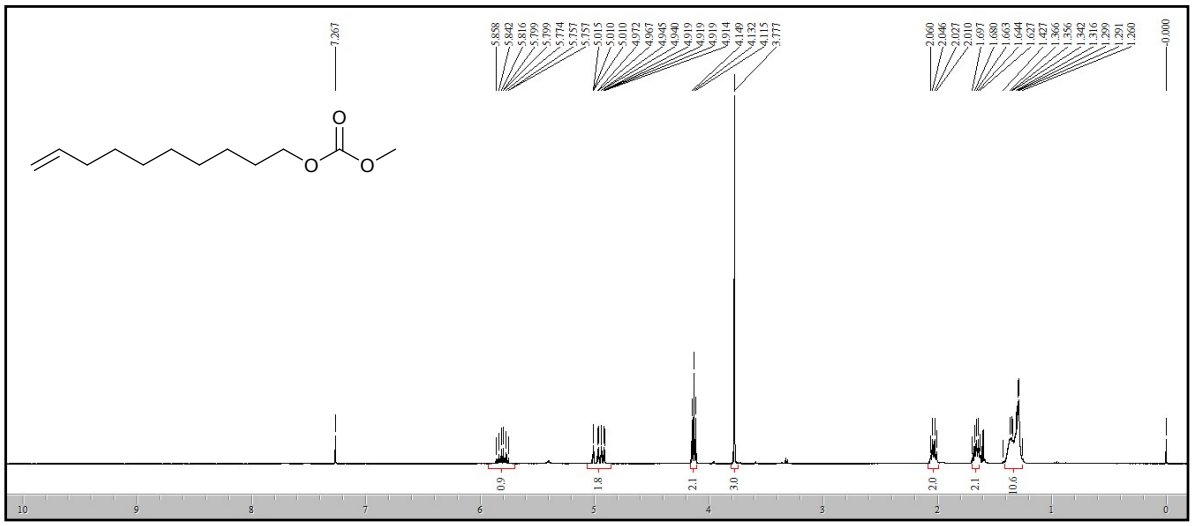
27.746
18.845
3l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5.015
5.015
5.010
5.010
4.972
4.967
4.945
4.940
4.919
4.919
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4.132
4.115
3.777
2.060
2.046
2.027
2.010
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1.644
1.627
1.427
1.366
1.356
1.342
1.316
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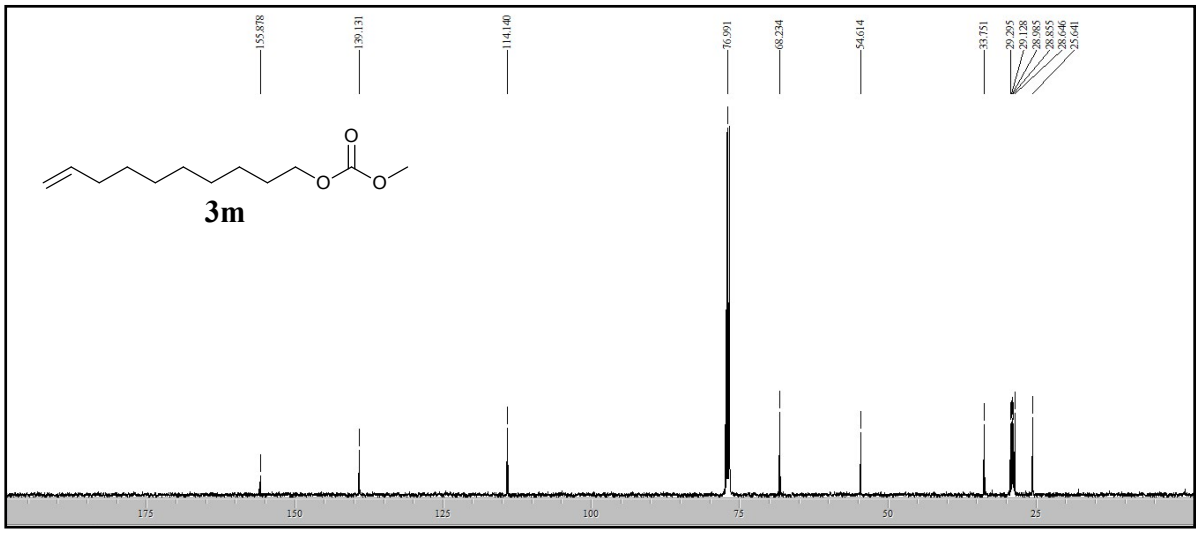
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28.855
28.646
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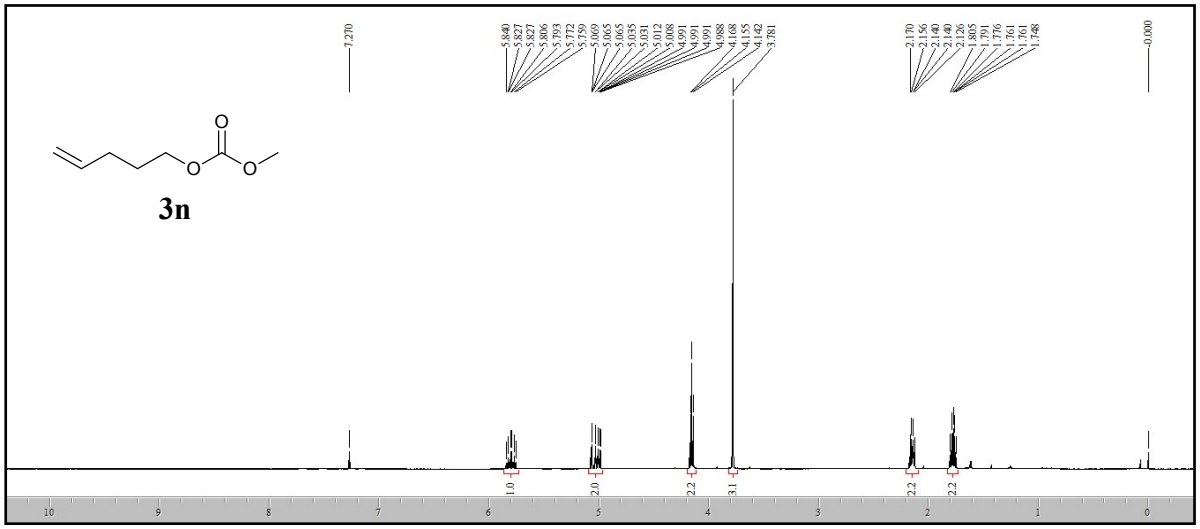
3m
175
150
125
100
75
50
25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5.031
5.012
5.008
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4.991
4.991
4.988
4.168
4.155
4.142
3.781
2.170
2.156
2.140
2.140
2.126
1.805
1.791
1.776
1.761
1.761
1.748
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3n
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3.1
2.2
2.2
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7
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2
1
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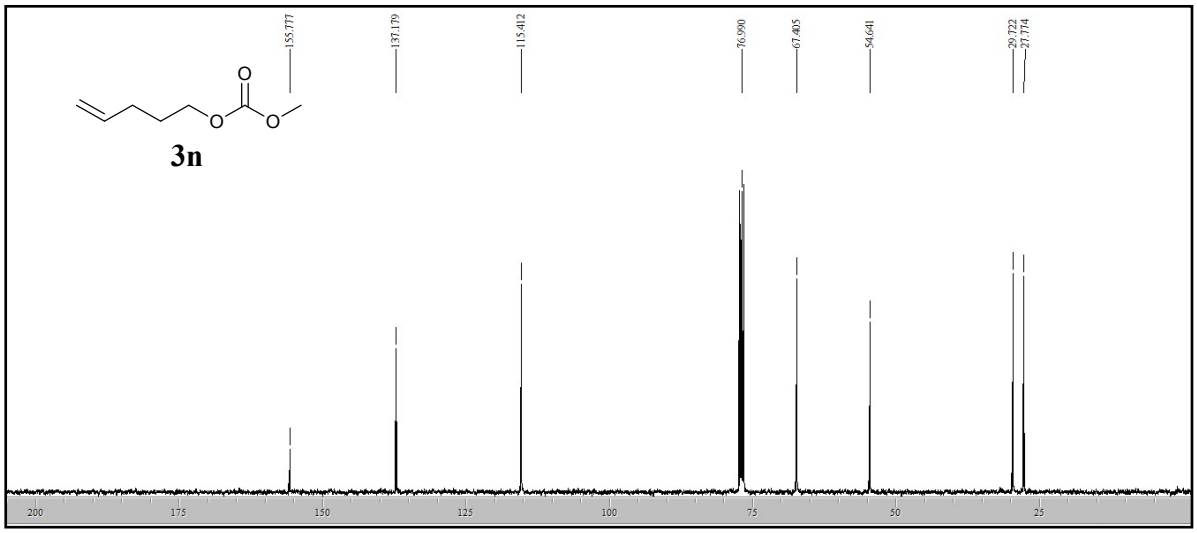
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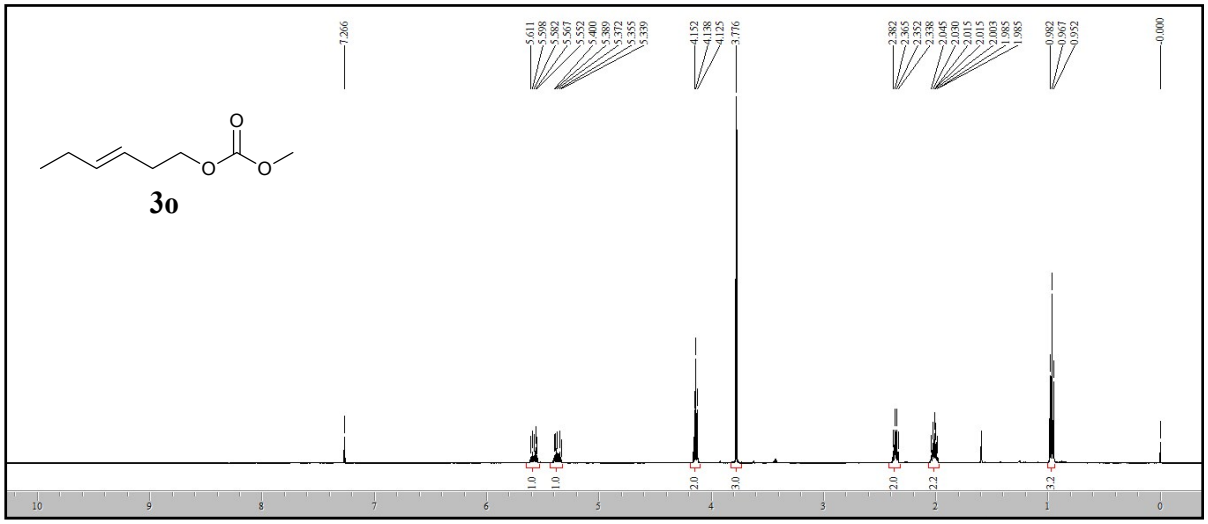
27.774
3n
200
175
150
125
100
75
50
25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2.015
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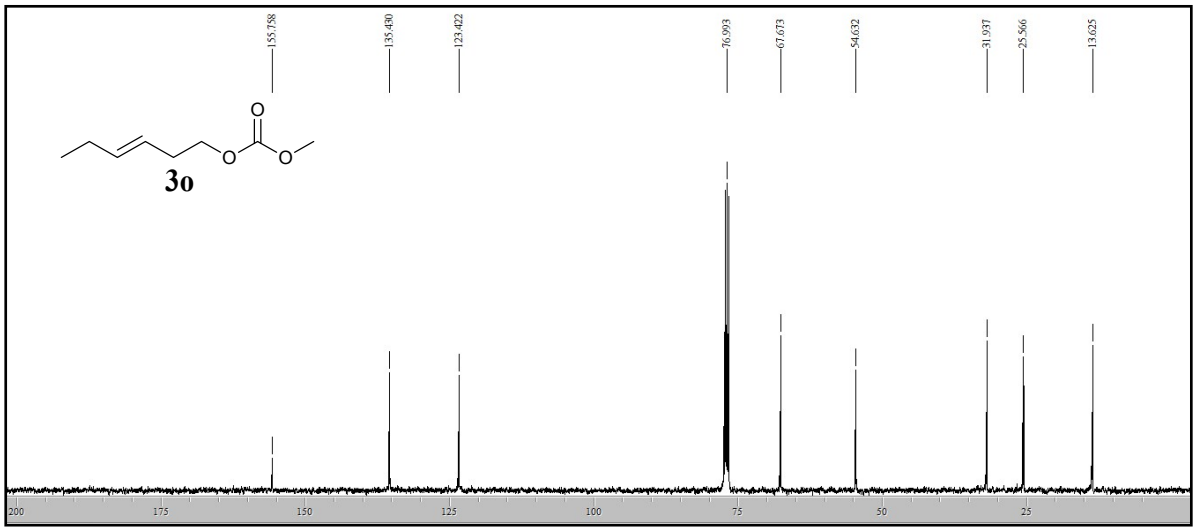
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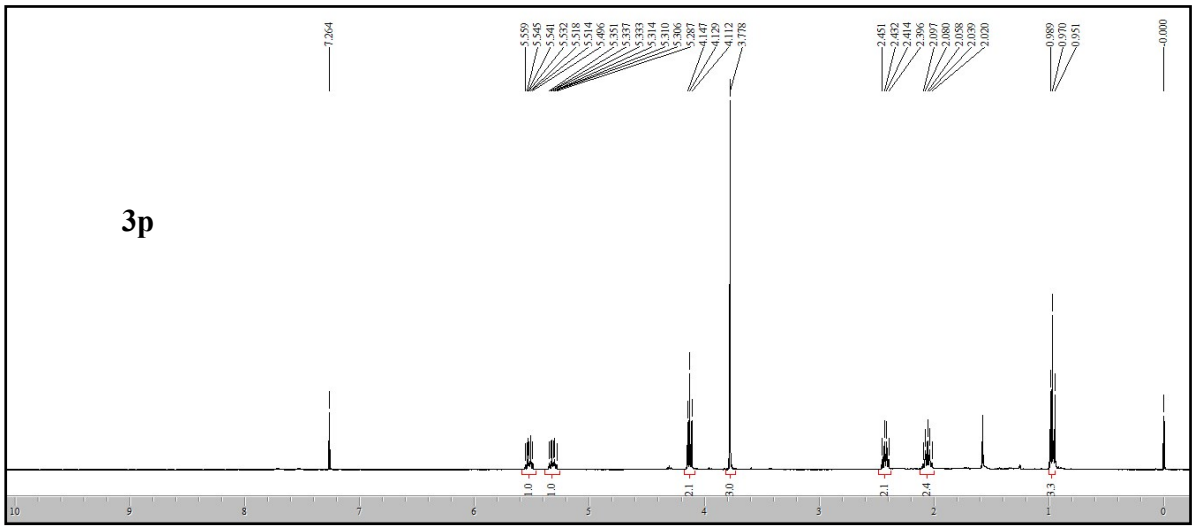
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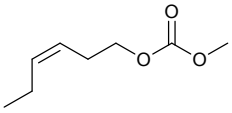

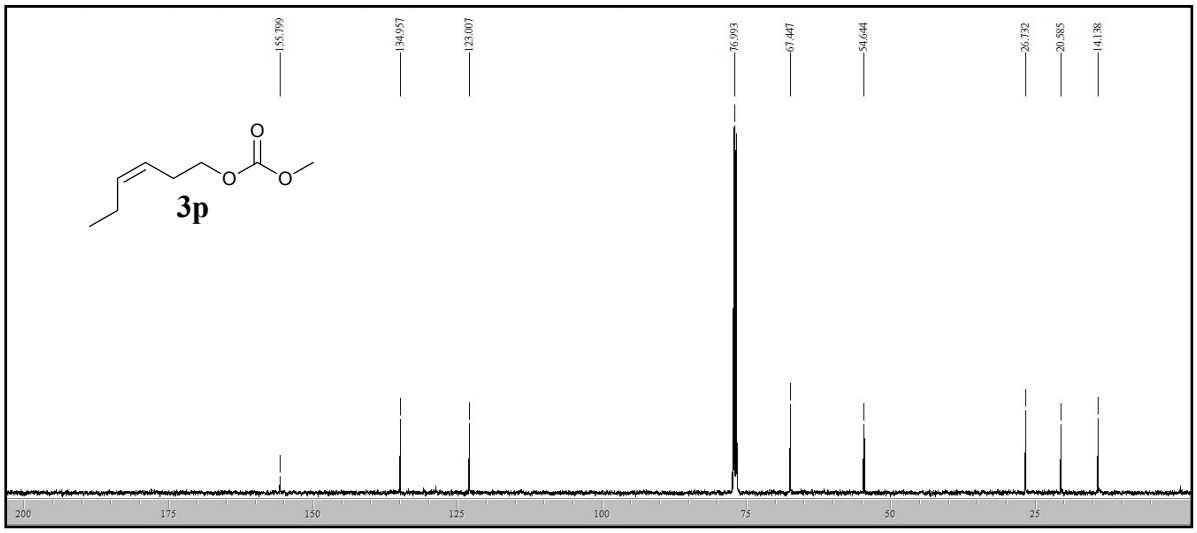
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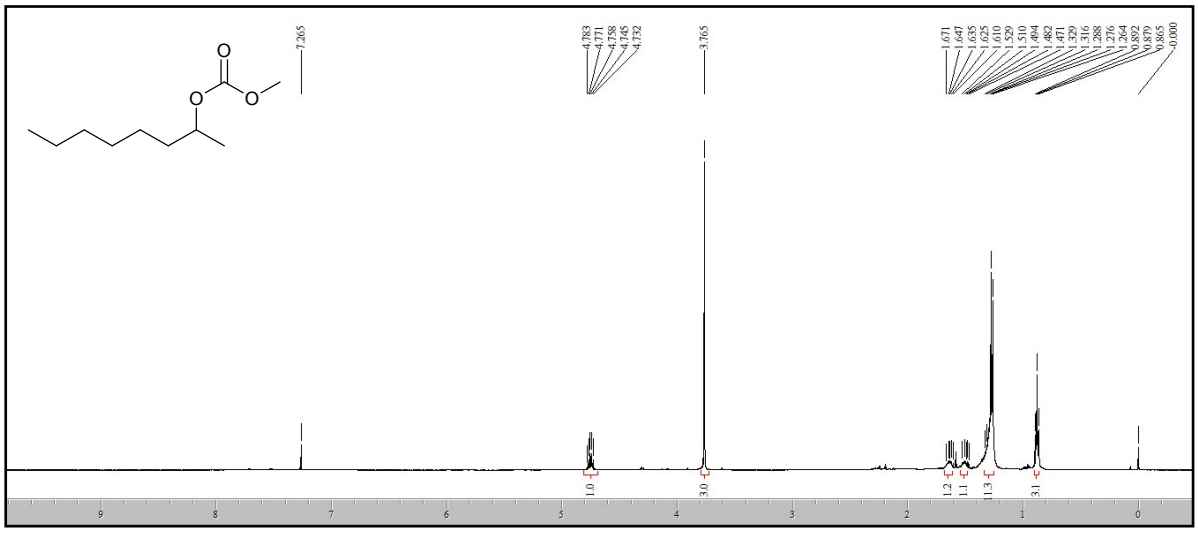
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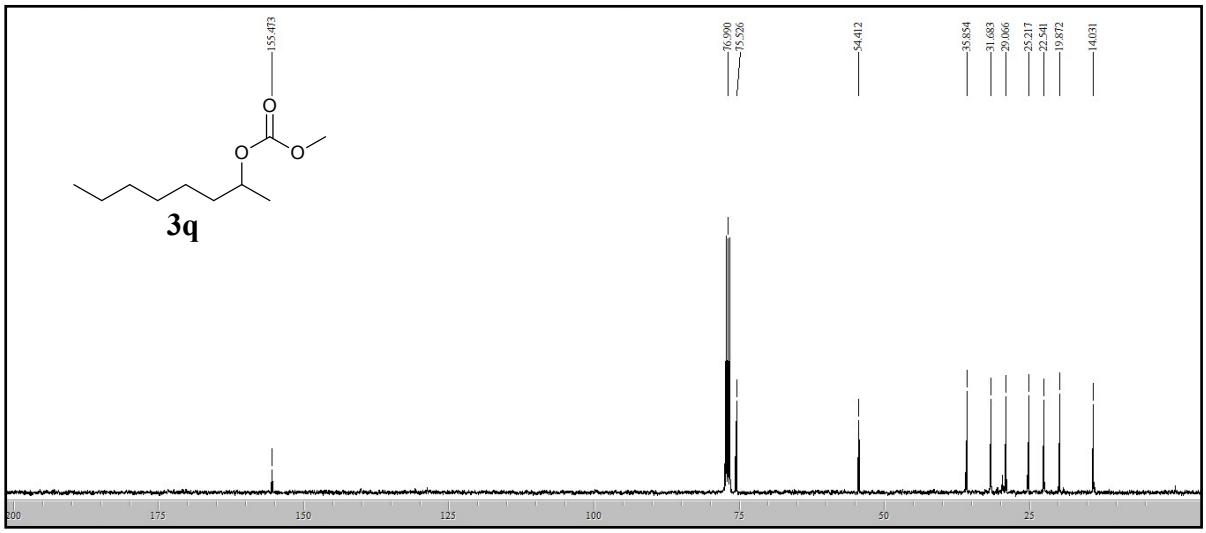
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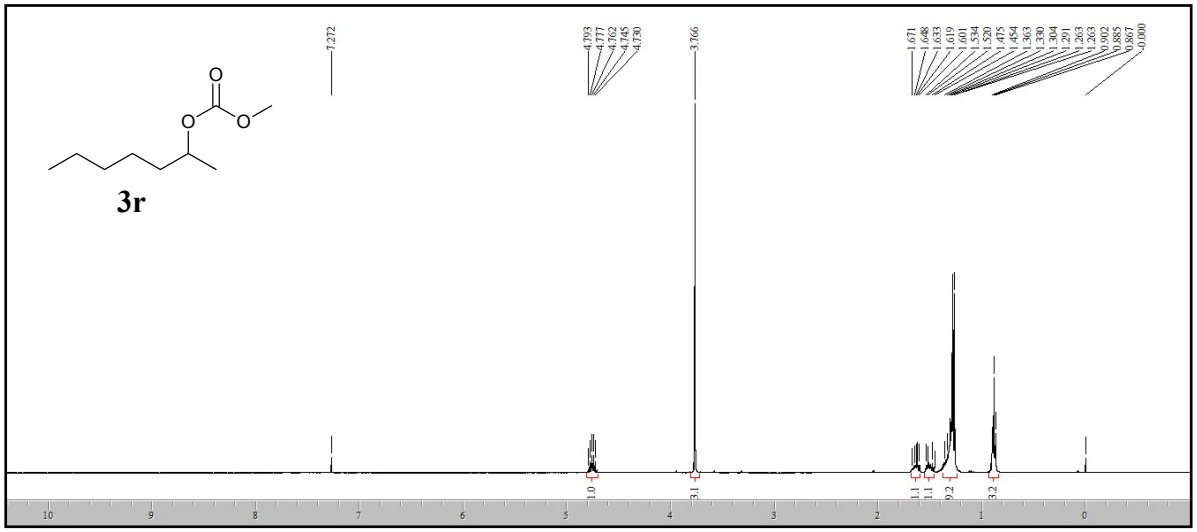
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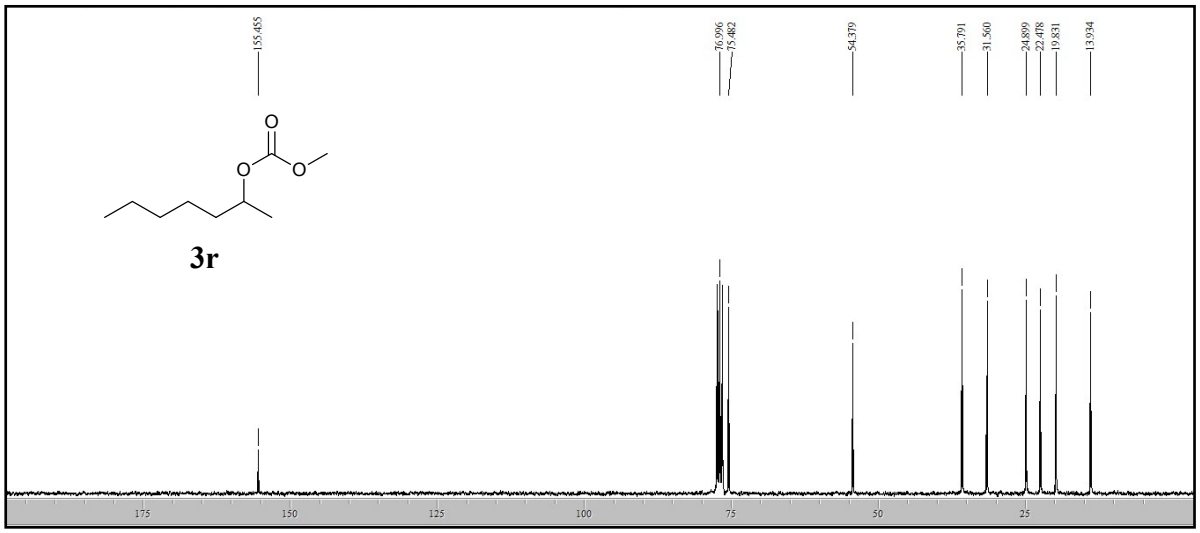
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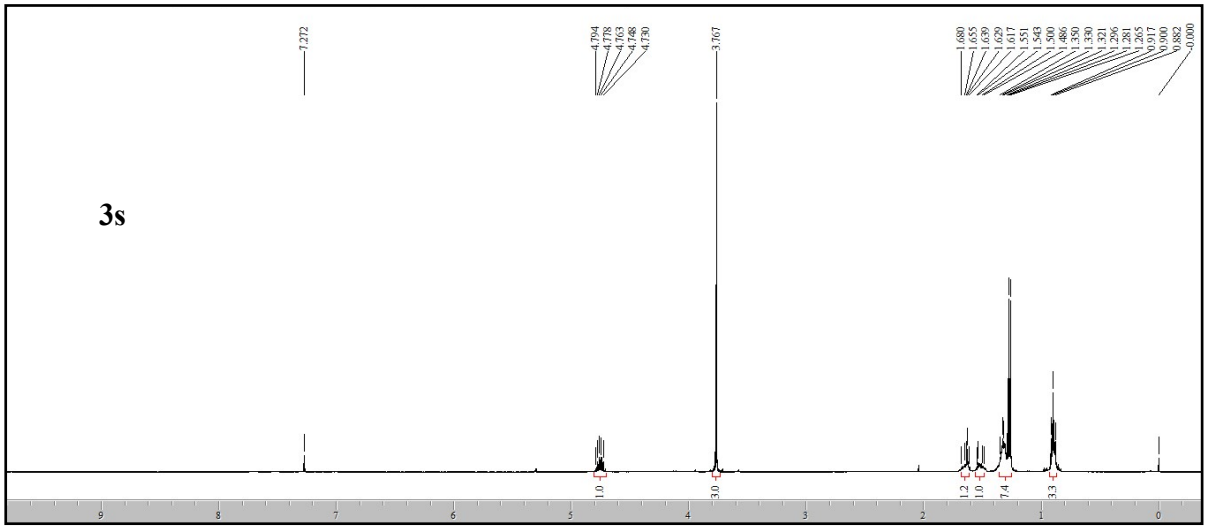
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1.543
1.500
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1.321
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1.265
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0.900
0.882
-0.000
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1.0
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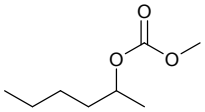

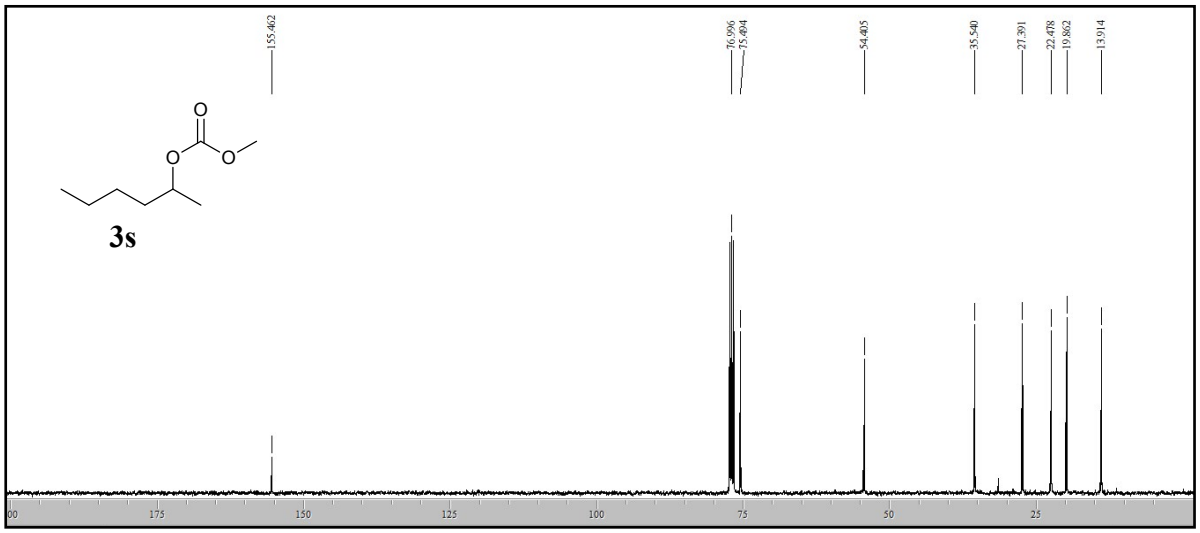
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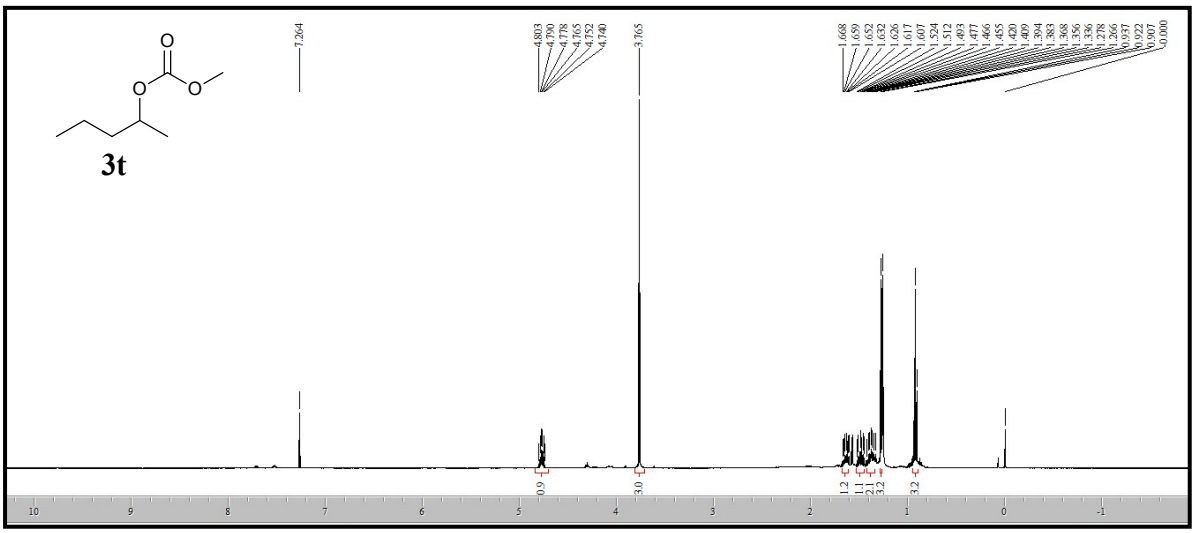
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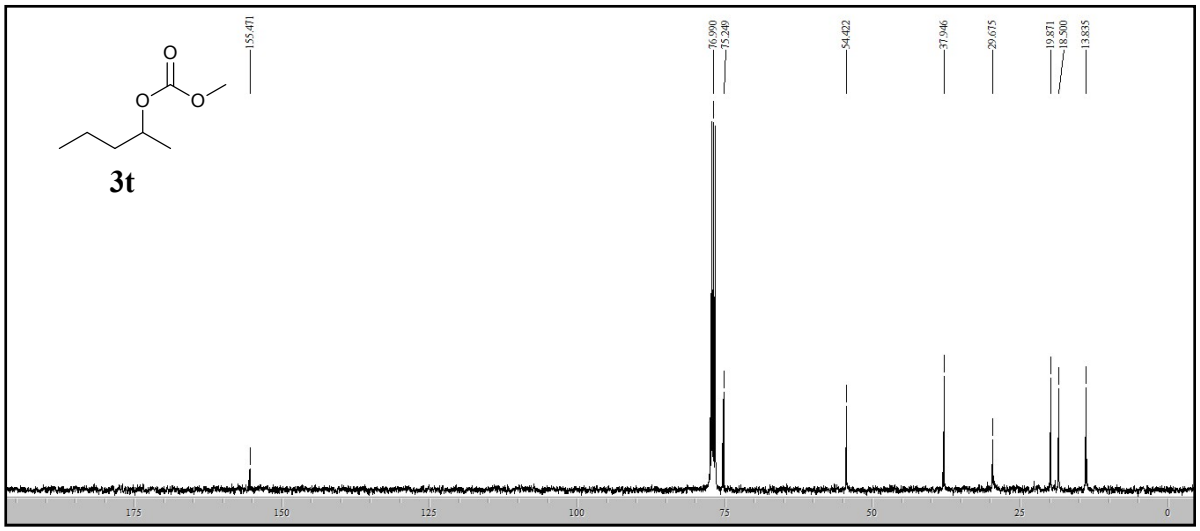
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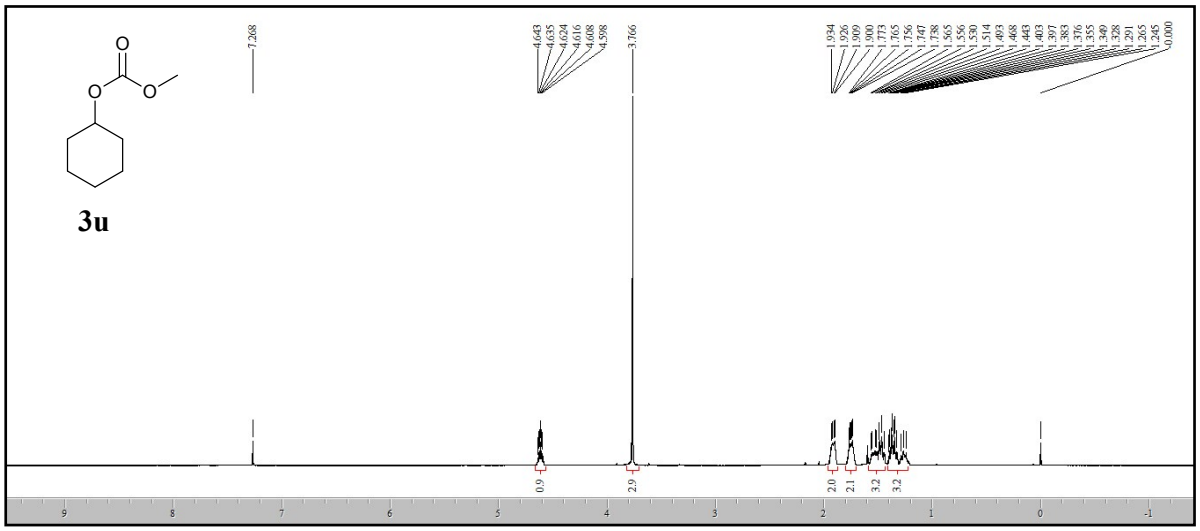
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1.556
1.530
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1.468
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1.403
1.397
1.383
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1.355
1.349
1.328
1.291
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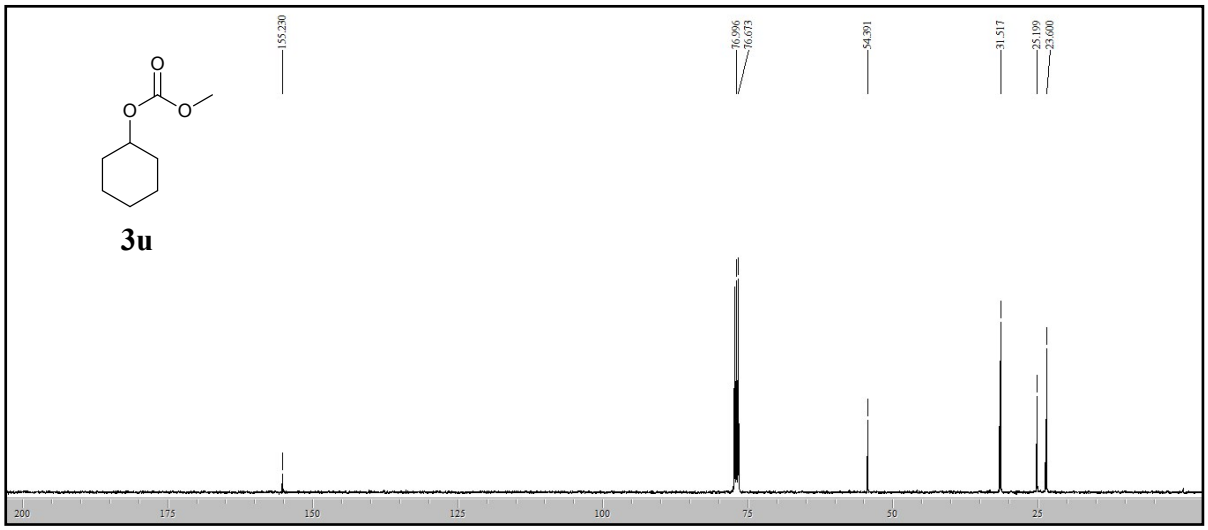
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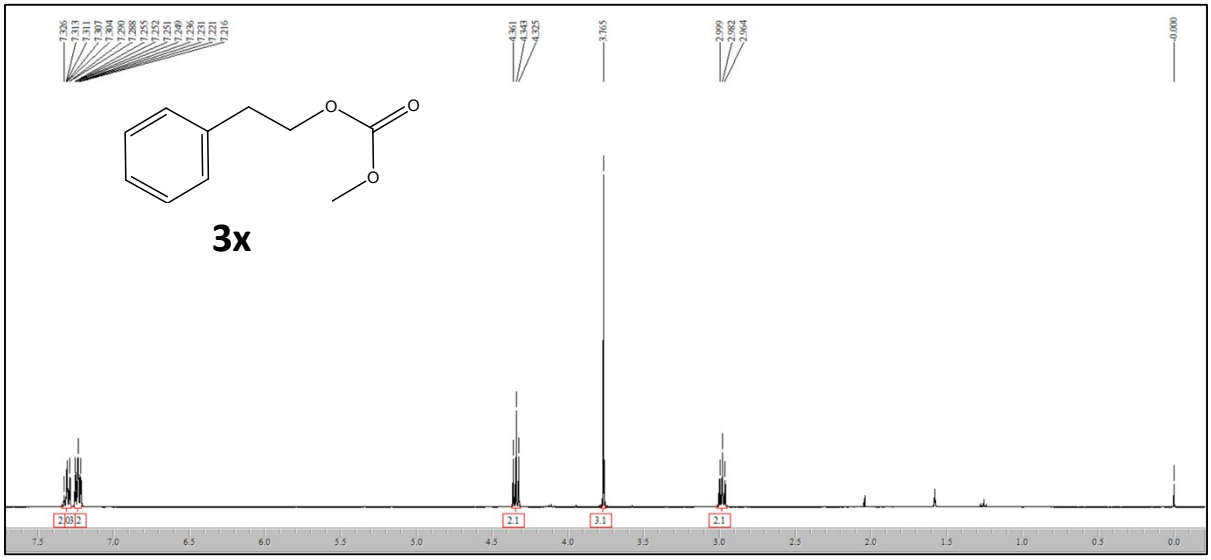
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7.221
7.216
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4.343
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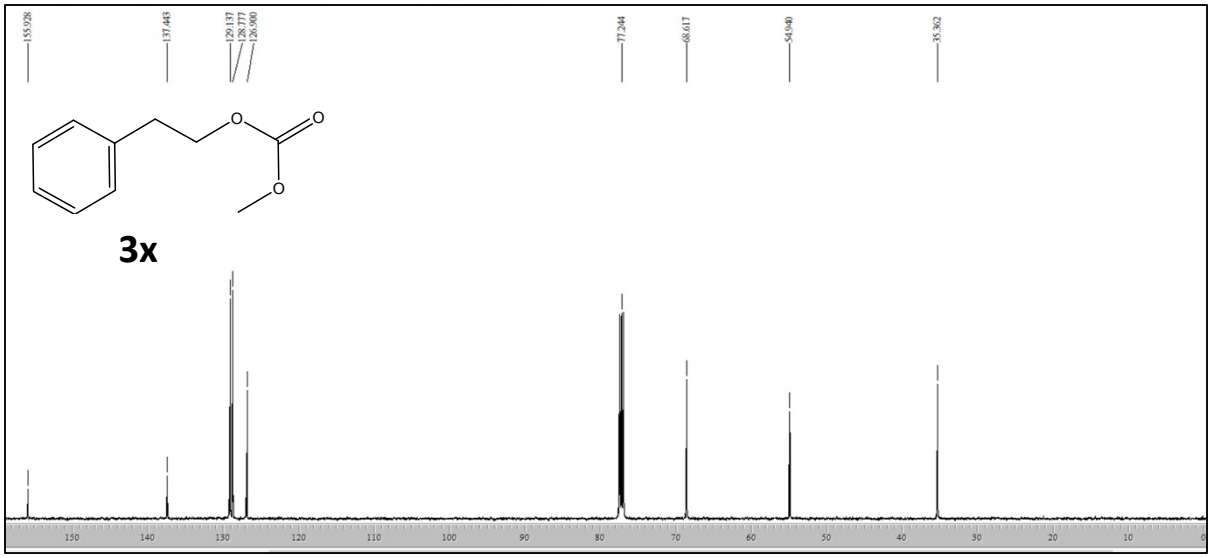

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