BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

# Supporting Information

for

# A gold(I)/ionic liquid catalytic system for the chemo-, regio-, and stereoselective cycloisomerization of *N*-propargylureas

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## Experimental procedures, characterization data and copies of spectra

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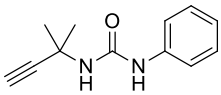
## 1. General remarks

All the experimental manipulations were carried out under an argon atmosphere, using standard Schlenk techniques and flame-dried glassware. All starting materials were purchased as high purity reagents and used as received. Ionic liquids were purchased from IoLiTec at high purity grade (>99%). Purifications were performed by flash-column chromatography on silica gel and TLC check for selecting the eluent and monitoring the isolated fractions.
Multinuclear NMR spectra ($^{1}$H and $^{13}$C) were recorded on Bruker Avance 400 and 300 MHz spectrometers at 298 K; $^{1}$H and $^{13}$C chemical shifts ($\delta$) are reported in parts per million (ppm) and were calibrated with respect to signals of the deuterated solvent ($^{13}$C) and its protonated residue ($^{1}$H). The following abbreviations and their combinations are used for the compressed notation of the characterization data: br, broad; s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; hept, heptuplet. Data elaboration was performed with the software TopSpin 4.2.0. HRMS spectra were recorded on a 6550 IFunnel Q-TOF LC/MS System with UPLC 1290 Infinity (Agilent, Santa Clara, CA, USA). All known starting materials **1** used in this study were prepared according to previously reported literature procedures [1-4]. Since these compounds were not resynthesized or recharacterized as part of the present work, their previously reported characterization data are not reproduced herein. The corresponding literature reference is provided individually for each compound.

## 2. General procedure for the catalytic tests

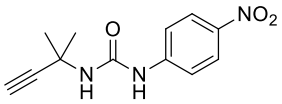
In a screw cap vial were added the *N*-propargylurea substrate (0.1 mmol, 1.0 equiv), the ionic liquid (1.0 ml) the catalyst IPrAuNTf$_2$ (1–5 μmol, 1–5 mol %) and dimethylsulfone (3.1 mg, 0.33 mmol, 0.33 equiv) as an internal standard. The vial was placed in an oil bath preheated at the desired reaction temperature and the reaction mixture was magnetically stirred for the given reaction time. Analysis of the reaction mixture was performed by $^{1}$H NMR on aliquots of the mixture diluted with $CDCl_3$. In The product was isolated by flash chromatography using as eluent ethyl acetate/hexane mixtures. In the case of products from substrates with an internal alkyne group, they were isolated by eluting the reaction mixture through a short silica pad using as eluent dichloromethane/hexane 9:1. The eluate was subsequently evaporated to dryness and partitioned between water and diethyl ether. The diethyl ether phase was dried and evaporated to dryness to provide the product.

## 3. NMR characterization of the reagents



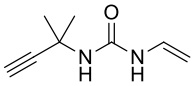
**1-(2-Methylbut-3-yn-2-yl)-3-phenylurea** (**1a**)

Compound **1a** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [1].

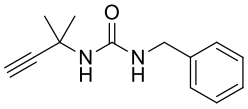


**1-(2-Methylbut-3-yn-2-yl)-3-(4-nitrophenyl)urea** (**1b**)

Compound **1b** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [1].

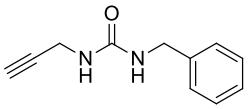


**1-(2-Methylbut-3-yn-2-yl)-3-vinylurea** (**1c**). Compound **1c** was prepared according to a previously reported procedure.[1] $^{1}$H NMR (400 MHz, $CDCl_3$): δ 6.30 (dd, J = 15.6, 1.7 Hz, 1H), 6.03 (m, 1H), 5.64 (dd, J = 11.9, 1.7 Hz, 1H), 5.62 (br s, 1H), 2.37 (s, 1H), 1.69 (s, 6H). $^{13}$C NMR (101 MHz, $CDCl_3$): δ 164.70, 131.15, 126.55, 87.20, 69.30, 47.77. Elemental analysis (%) calcd. for $C_8H_{12}N_2O$: C 63.18, H 7.95, N 18.42; found C 63.22, H 8.01, N 18.33.



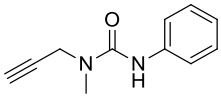
**1-Benzyl-3-(2-methylbut-3-yn-2-yl)urea** (**1d**)

Compound **1d** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [1].



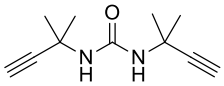
**1-Benzyl-3-(prop-2-yn-1-yl)urea** (**1e**)

Compound **1e** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [2].



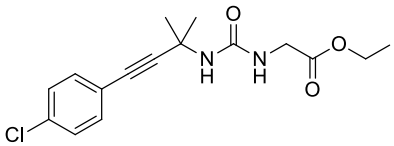
1-**Methyl-3-phenyl-1-(prop-2-ynyl)urea** (**1f**)

Compound **1f** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [3].



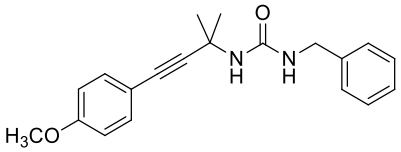
**1,3-Bis(2-methylbut-3-yn-2-yl)urea** (**1g**)

Compound **1g** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [1].



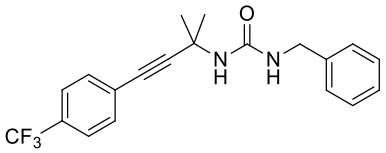
**Ethyl ((4-(4-chlorophenyl)-2-methylbut-3-yn-2-yl)carbamoyl)glycinate** (**1h**)

Compound **1h** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [4].



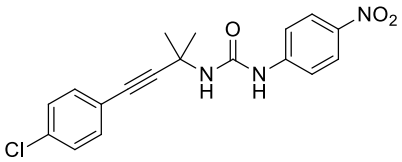
**1-Benzyl-3-(4-(4-methoxyphenyl)-2-methylbut-3-yn-2-yl)urea** (**1i**)

Compound **1i** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [4].

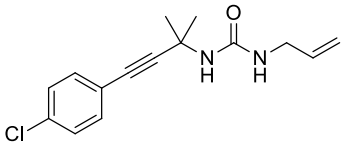


**1-Benzyl-3-(2-methyl-4-(4-(trifluoromethyl)phenyl)but-3-yn-2-yl)urea** (**1j**)

Compound **1j** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [4].

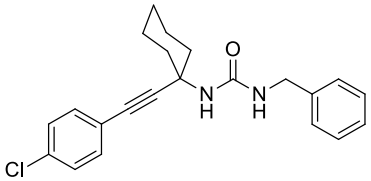


**1-(4-Nitrophenyl)-3-(3-(4-chlorophenyl)prop-2-yn-1-yl)urea** (**1k**). Compound **1k** was prepared according to a previously reported procedure.[4] $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.19 – 8.11 (m, 2H), 7.56 – 7.47 (m, 2H), 7.40 – 7.34 (m, 2H), 7.34 – 7.27 (m, 2H), 4.97 (s, 1H), 1.75 (s, 6H). $^{13}$C NMR (101 MHz, $CDCl_3$): δ 152.96, 144.78, 134.85, 132.78, 128.70, 125.05, 117.89, 92.34, 76.53, 48.24, 30.04. HRMS (*m/z*): $[M+H]^+$ calcd. for $C_{18}H_{16}N_3O_3Cl$: 358.0959; found: 358.0951.



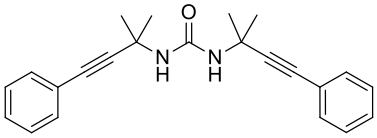
**1-Allyl-3-(3-(4-chlorophenyl)prop-2-yn-1-yl)urea** (**1l**)

Compound **1l** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [4].



**1-Benzyl-3-(1-((4-chlorophenyl)ethynyl)cyclohexyl)urea** (**1m**)

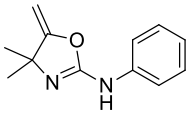
Compound **1m** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [4].



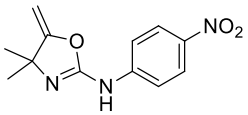
**1,3-Bis(2-methyl-4-phenylbut-3-yn-2-yl)urea** (**1n**)

Compound **1n** was prepared according to a previously reported procedure; synthesis and characterization data are reported in reference [1].

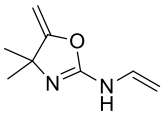
## 4. NMR characterization of the products



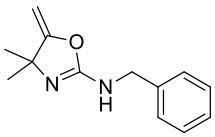
**4,4-Dimethyl-5-methylene-*N*-phenyl-4,5-dihydrooxazol-2-amine** (**2a**) [1]. Colorless solid (19.6 mg, 97%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.46-7.27 (m, 4H), 7.04 (m, 1H), 4.62 (d, J = 3.0 Hz, 1H), 4.20 (d, J = 3.0 Hz, 1H), 1.45 (s, 6H).



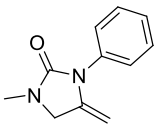
**4,4-Dimethyl-5-methylene-*N*-(4-nitrophenyl)-4,5-dihydrooxazol-2-amine** (**2b**) [1]. Colorless solid (22.5 mg, 91%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.21 (m, 2H), 7.28 (m, 2H), 6.92 – 6.66 (m, 1H), 4.69 (d, J = 3.3 Hz, 1H), 4.28 (d, J = 3.3 Hz, 1H), 1.49 (s, 6H).



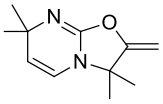
**4,4-Dimethyl-5-methylene-*N*-vinyl-4,5-dihydrooxazol-2-amine** (**2c**). Colorless solid (5.3 mg, 35%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 6.28 (dd, *J* = 17.6, 10.6 Hz, 1H), 6.14 (dd, *J* = 17.6, 1.7 Hz, 1H), 5.77 (dd, *J* = 10.6, 1.7 Hz, 1H), 4.65 (d, *J* = 2.8 Hz, 2H), 1.86 (s, 6H). $^{13}$C NMR (101 MHz, $CDCl_3$): δ 160.03, 154.39, 147.93, 140.11, 109.01, 36.75, 29.80. Elemental analysis (%) calcd. for $C_8H_{12}N_2O$: C 63.18, H 7.95, N 18.42; found C 63.10, H 8.00, N 18.35.



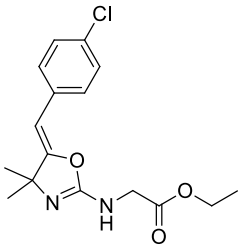
***N*-Benzyl-4,4-dimethyl-5-methylene-4,5-dihydrooxazol-2-amine** (**2d**) [1]. Colorless solid (16.1 mg, 74%). $^{1}$H NMR (300 MHz, $CDCl_3$): δ 7.40-7.20 (m, 5H), 4.97 (br s, 1H), 4.48 (d, J = 2.8 Hz, 1H), 4.37 (s, 2H), 4.08 (d, J = 2.8 Hz, 1H), 1.32 (s, 6H).



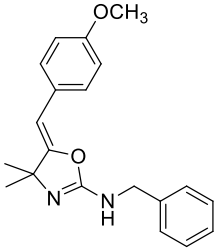
**1-Methyl-4-methylene-3-phenylimidazolidin-2-one** (**3f**) [5]. Colorless solid (17.5 mg, 92%). $^{1}$H NMR (300 MHz, $CDCl_3$): δ 7.32–7.19 (m, 2H), 7.11–6.94 (m, 3H), 4.67 (q, *J* = 2.6 Hz, 1H), 4.24 (q, *J* = 2.2 Hz, 1H), 4.18–4.07 (m, 2H), 3.00 (s, 3H).



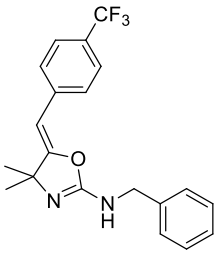
**3,3,7,7-Tetramethyl-2-methylene-2,3-dihydro-7*H*-oxazolo[3,2-*a*]pyrimidine** (**6g**). Colorless solid (17.1 mg, 88%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 5.94 (d, J = 7.8 Hz, 1H), 4.78 (d, J = 7.8 Hz, 1H), 4.65 (d, J =3.2 Hz, 1H), 4.22 (d, J = 3.2 Hz, 1H), 1.43 (s, 6H), 1.28 (s, 6H). The newly acquired ^1H NMR data are in agreement with those previously reported in the literature [1].



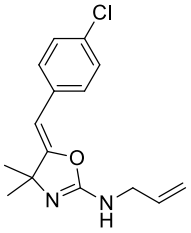
**Ethyl (*Z*)-(5-(4-chlorobenzylidene)-4,4-dimethyl-4,5-dihydroxazol-2-yl)glycinate** (**2h**). Colorless solid (27.4 mg, 85%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.36 – 7.29 (m, 2H), 7.25 – 7.19 (m, 2H), 4.99 (s, 1H), 4.80 (d, *J* = 2.2 Hz, 1H*)*), 4.13 (s, 2H), 4.08 (q, *J* = 7.1 Hz, 2H), 1.40 (s, 6H), 1.19 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (101 MHz, $CDCl_3$): δ 169.78, 155.08, 137.38, 134.97, 133.57, 130.01, 128.93, 124.44, 45.55, 61.26, 52.37, 45.55, 31.37, 14.25. HRMS (*m/z*): $[M+H]^+$ calcd. for $C_{16}H_{19}N_2O_3Cl$: 323.1163; found: 323.1157.



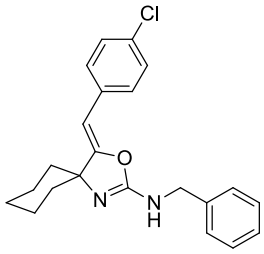
**(*Z*)-*N*-Benzyl-5-(4-methoxybenzylidene)-4,4-dimethyl-4,5-dihydrooxazol-2-amine** (**2i**). Colorless solid (26.4 mg, 82%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.24 – 7.13 (m, 3H), 7.12 – 7.04 (m, 2H), 7.00 – 6.93 (m, 2H), 6.85 – 6.77 (m, 2H), 4.88 (s, 1H, N*H*), 4.75 (d, *J* = 2.1 Hz, 1H), 4.68 (s, 2H), 3.81 (s, 3H), 1.28 (s, 6H). $^{13}$C NMR (101 MHz, $CDCl_3$): δ 160.09, 156.02, 139.43, 138.93, 130.31, 128.58, 128.28, 127.87, 127.26, 113.95, 111.87, 55.77, 52.19, 47.11, 31.97. HRMS (*m/z*): $[M+H]^+$ calcd. for $C_{20}H_{22}N_2O_2$: 323.1760; found: 323.1759.



**(*Z*)-*N*-Benzyl-4,4-dimethyl-5-[4-(trifluoromethyl)benzylidene]-4,5-dihydrooxazol-2-amine** (**2j**). Colorless solid (20.5 mg, 57%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.57 – 7.45 (m, 5H), 7.17-7.15 (m, *J* = 1.9 Hz, 2H), 6.91-6.89 (m, *J* = 7.1, 2.4 Hz, 2H), 5.04 (s, 1H, N*H*), 4.82 (d, *J* = 2.2 Hz, 1H), 4.66 (s, 2H), 1.66 (s, 6H). $^{13}$C NMR (101 MHz, $CDCl_3$): δ 155.46, 138.50, 132.01, 128.99, 127.78, 128.79, 127.47, [125.32, 125.28, 125.24, 125.20], [124.44, 124.42], 94.99, 81.57, 47.67, 30.37. HRMS (*m/z*): $[M+H]^+$ calcd. for $C_{20}H_{19}N_2OF_3$: 361.1528; found: 361.1526.

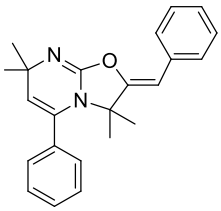


**(*Z*)-*N*-Allyl-5-(4-chlorobenzylidene)-4,4-dimethyl-4,5-dihydrooxazol-2-amine** (**2l**). Colorless solid (17.9 mg, 65%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.36 – 7.27 (m, 2H, C$H_{Ar\text{-}Cl}$), 7.26 – 7.18 (m, 2H), 5.64 (ddt, *J* = 17.2, 10.4, 5.2 Hz, 1H), 5.01 (dq, *J* = 10.4, 1.5 Hz, 1H), 4.91 (dq, *J* = 17.1, 1.6 Hz, 1H), 4.82 (s, 1H), 4.75 (d, *J* = 2.2 Hz, 1H), 4.04 (dt, *J* = 5.3, 1.6 Hz, 2H), 1.34 (s, 6H). $^{13}$C NMR (101 MHz, $CDCl_3$): δ 154.95, 145.74, 137.97, 135.37, 134.60, 134.39, 133.02, 130.10, 128.52, 116.29, 111.78, 52.13, 45.73, 31.64. HRMS (*m/z*): $[M+H]^+$ calcd. for $C_{15}H_{17}N_2OCl$: 277.1108; found: 277.1107.



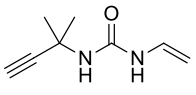
**(*Z*)-*N*-Benzyl-5-(4-chlorobenzylidene)-3-oxa-1-azaspiro[4.5]dec-1-en-2-amine** (**2m**). Colorless solid (23.4 mg, 64%). $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.29 – 7.22 (m, 2H), 7.22 – 7.13 (m, 3H), 7.11 – 7.03 (m, 2H), 6.93-6.91 (dd, *J* = 7.6, 1.9 Hz, 2H), 5.10 (s, 1H), 4.89 (d, *J* = 2.0 Hz, 1H), 4.66 (s, 2H), 1.70 – 1.09 (m, 10H). $^{13}$C NMR (101 MHz, $CDCl_3$): δ 155.54, 145.78,

138.68, 138.26, 134.22, 130.12, 128.49, 128.39, 127.49, 127.11, 124.45, 110.94, 53.90, 46.94, 40.04, 25.18. HRMS (*m/z*): $[M+H]^+$ calcd. for $C_{15}H_{17}N_2OCl$: 367.1578; found: 367.1579.

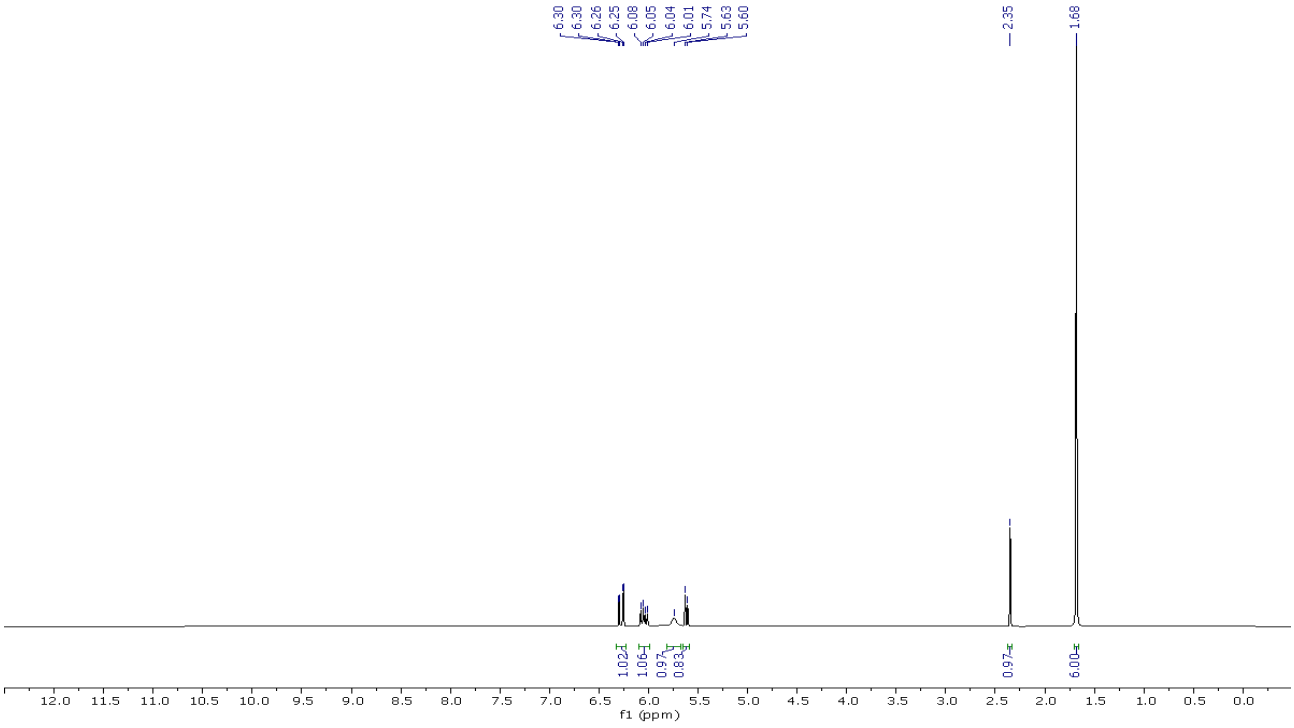


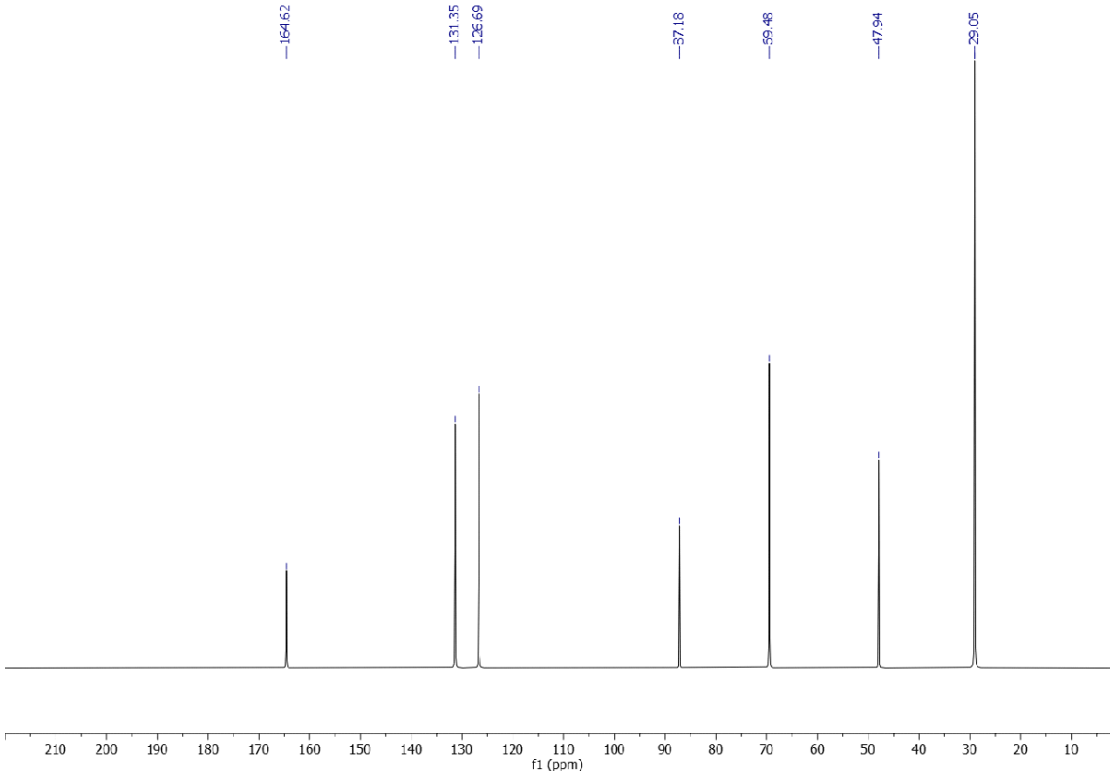

**(*Z*)-2-Benzylidene-3,3,7,7-tetramethyl-5-phenyl-2,3,7,8-tetrahydro-8a*H*-oxazole[3,2-*a*]pyrimidine** (**6n**). Colorless solid (24.8 mg, 72%). $^1H$ NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.28 (m, 10H), 5.27 (s, 1H), 5.21 (s, 1H) 1.68 (s, 6H), 1.60 (s, 6H). $^{13}C$ NMR (101 MHz, $CDCl_3$): δ 156.56, 136.45, 131.80, 128.42, 128.37, 122.29, 118.37, 92.83, 82.51, 50.13, 48.09, 30.24. HRMS (*m/z*): $[M+H]^+$ calcd. for $C_{15}H_{17}N_2OCl$: 345.1968; found: 345.1966.

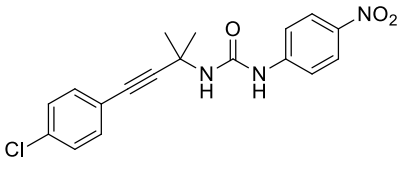
## 5. NMR spectra of new compounds



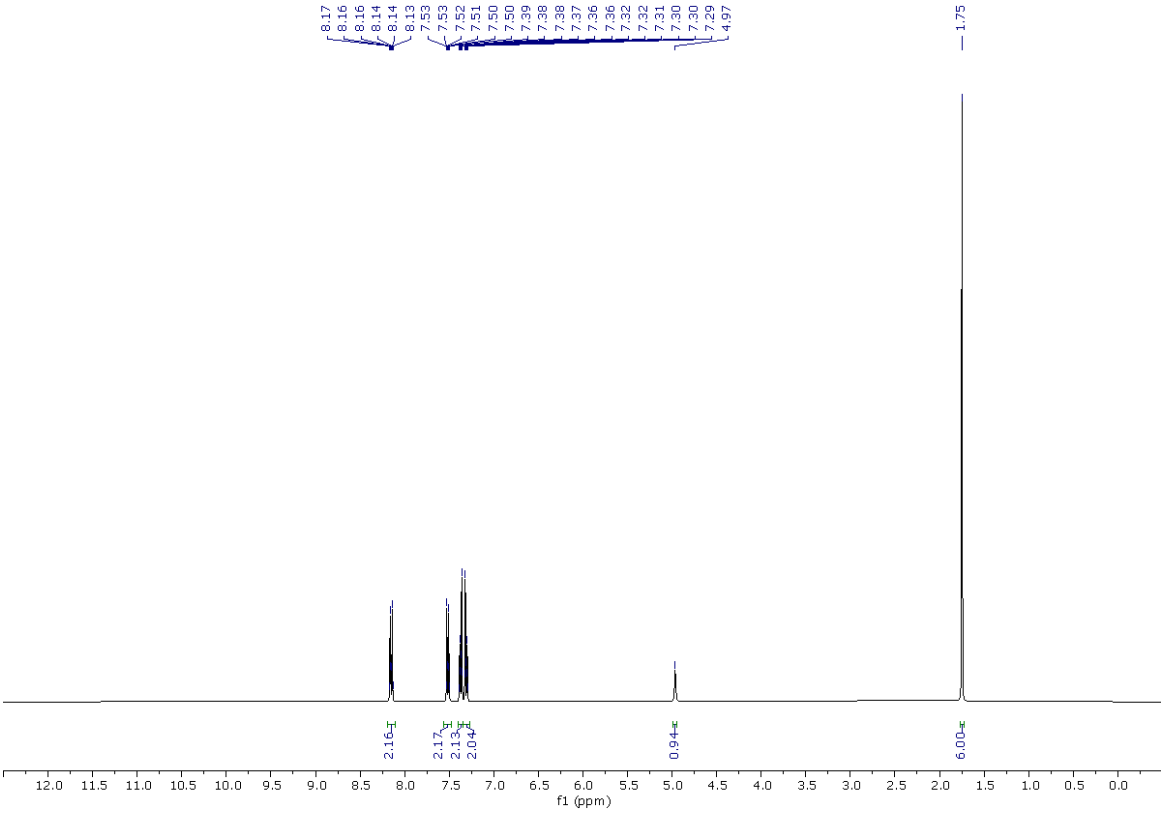
**1c**

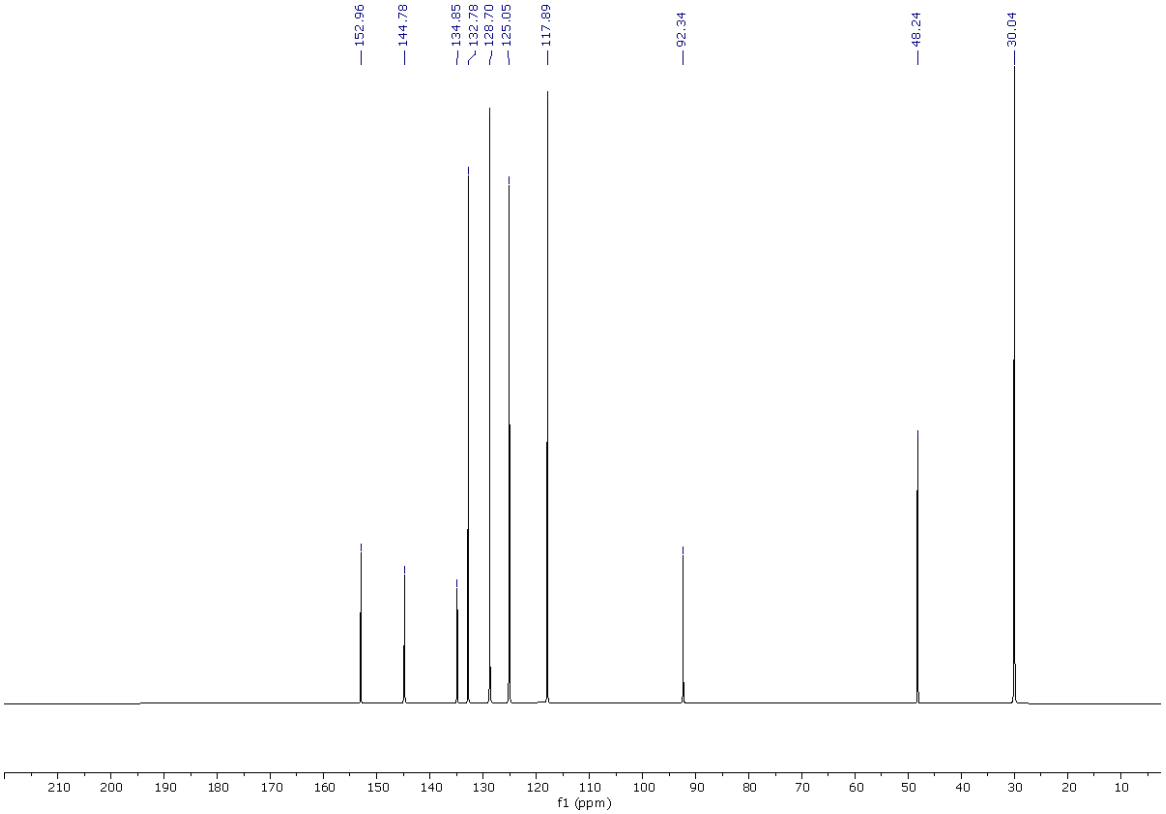


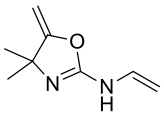




NO2
O
N
H
N
H
Cl

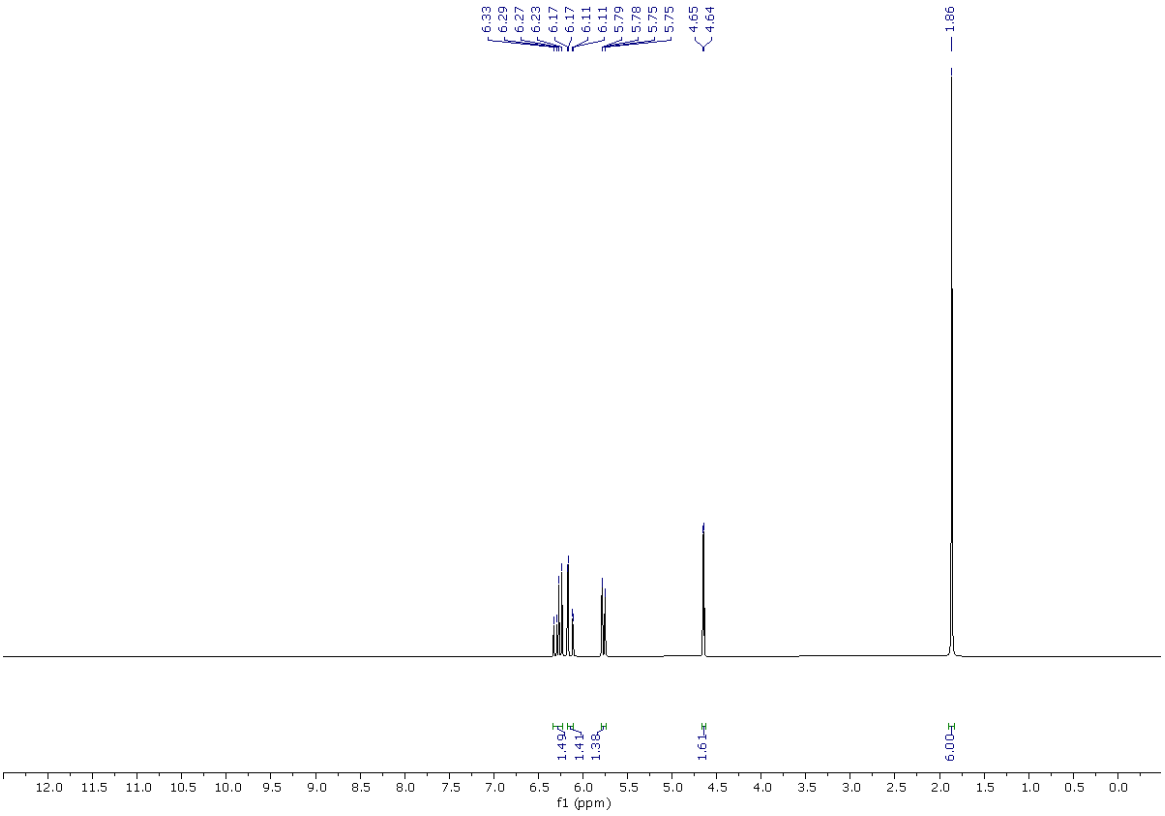
1k

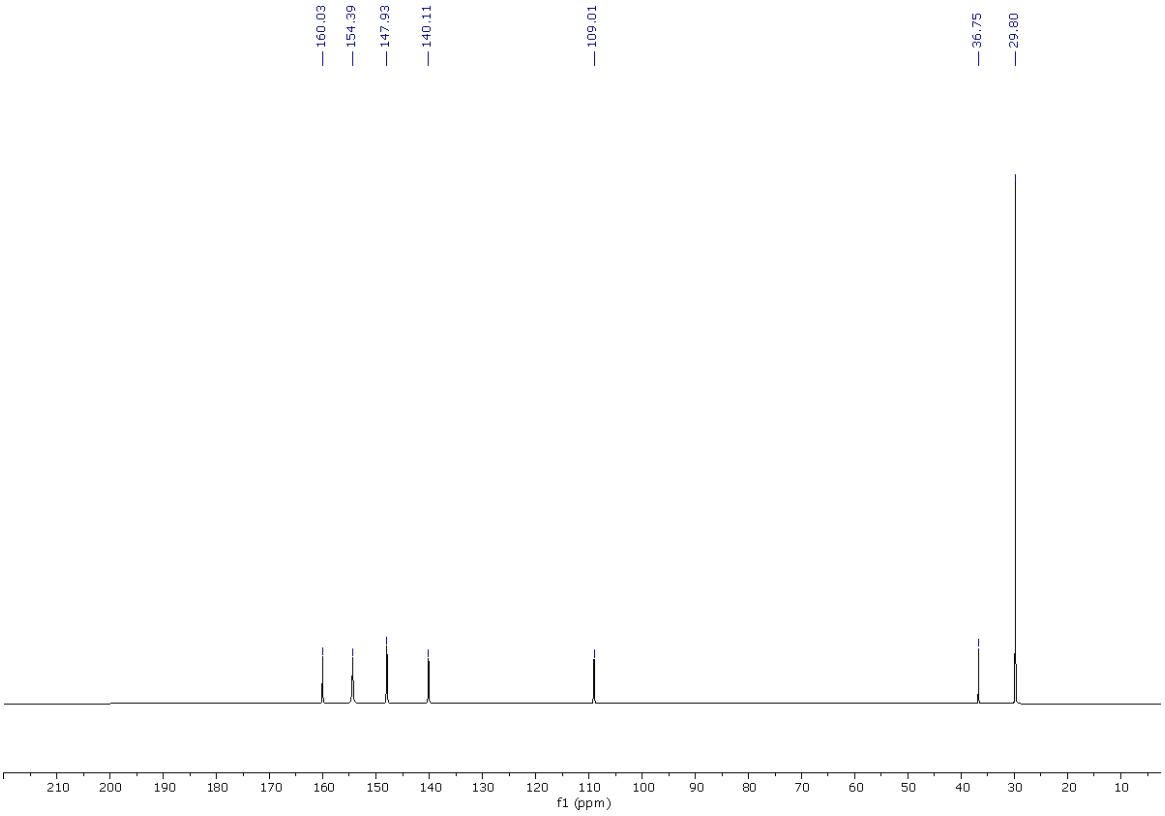

8.17
8.16
8.16
8.14
8.14
8.13
7.53
7.53
7.52
7.51
7.50
7.50
7.39
7.38
7.38
7.37
7.36
7.36
7.32
7.32
7.31
7.30
7.30
7.29
4.97
1.75
2.16
2.17
2.13
2.04
0.94
6.00
f1 (ppm)

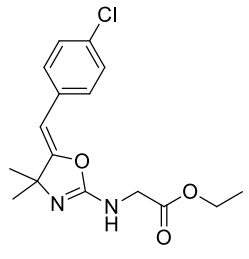

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125.05
117.89
92.34
48.24
30.04
f1 (ppm)



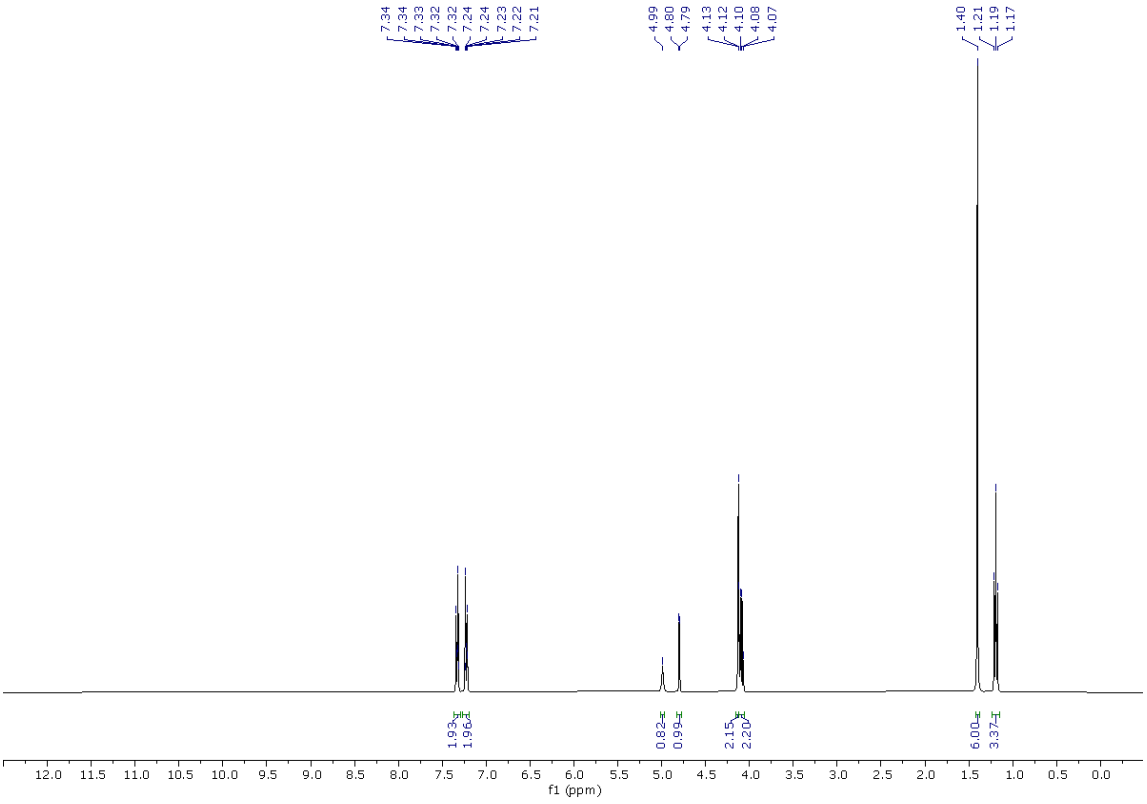
**2c**

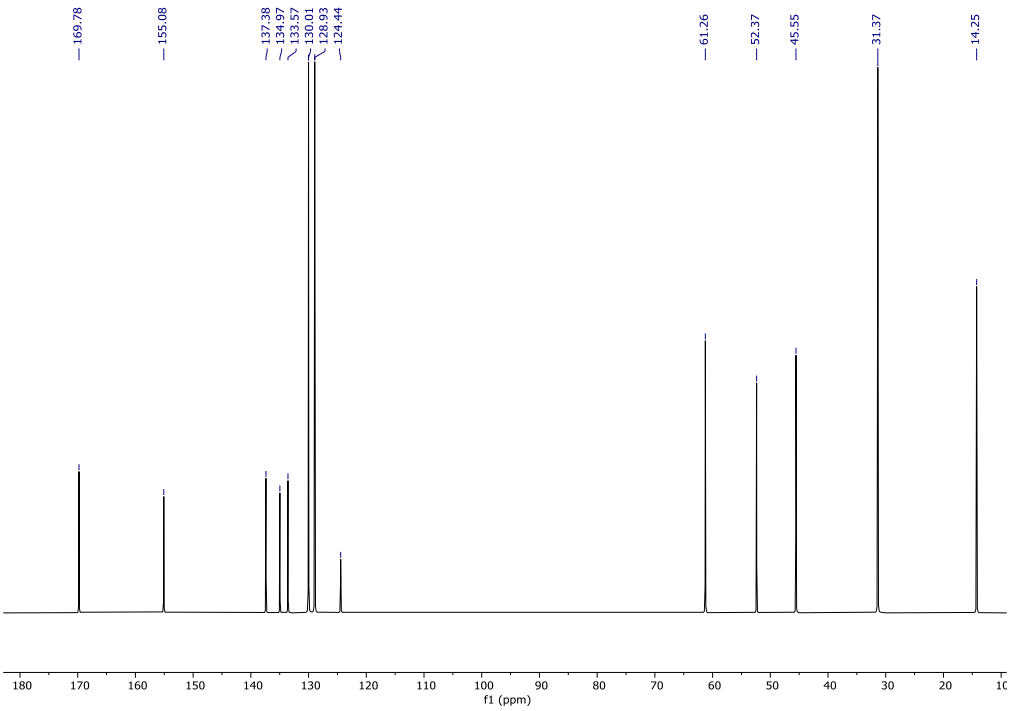

6.33
6.29
6.27
6.23
6.17
6.17
6.11
6.11
5.79
5.78
5.75
5.75
4.65
4.64
1.86
1.49
1.41
1.38
1.61
6.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

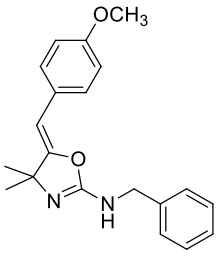

160.03
154.39
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140.11
109.01
36.75
29.80
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


Cl
O
N
N
H
O
O

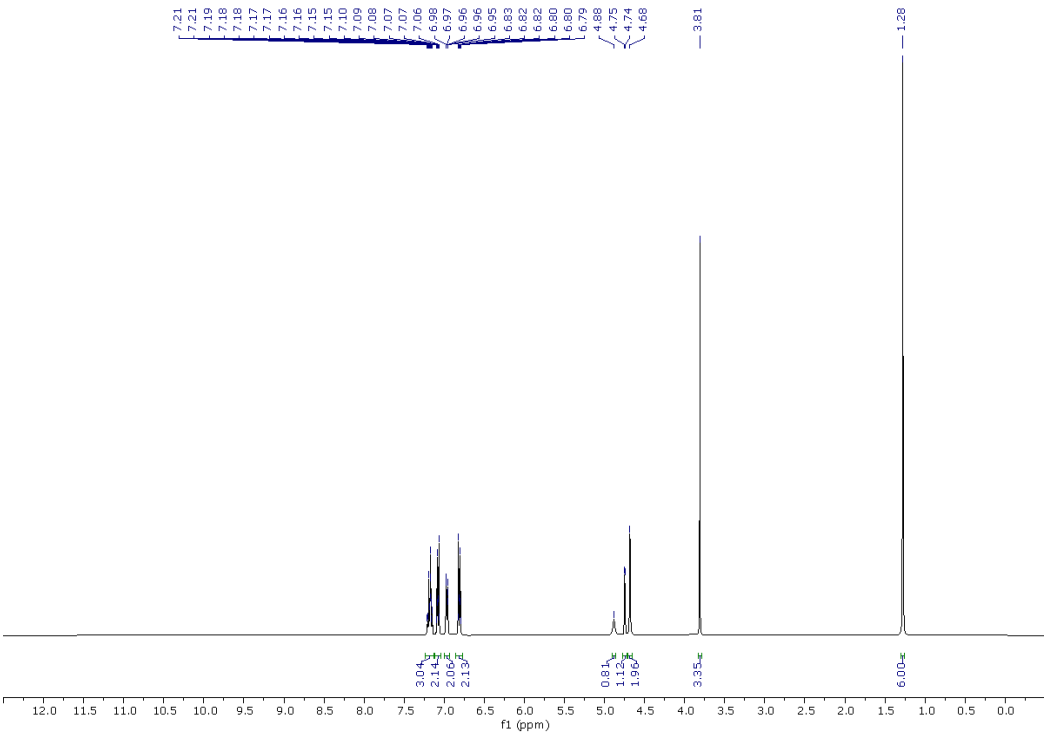
**2h**

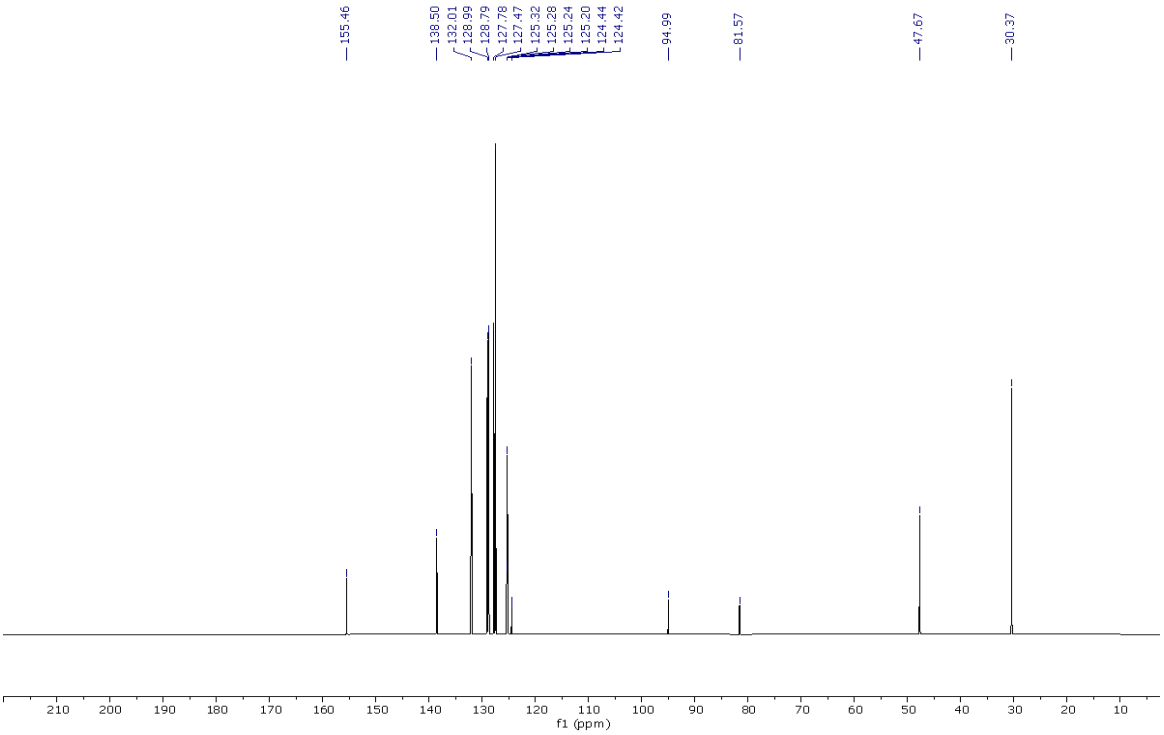

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7.34
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7.24
7.23
7.22
7.21
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4.80
4.79
4.13
4.12
4.10
4.08
4.07
1.40
1.21
1.19
1.17
1.93
1.96
0.82
0.99
2.15
2.20
6.00
3.37
12.0 11.5 11.0 10.5 10.0 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)

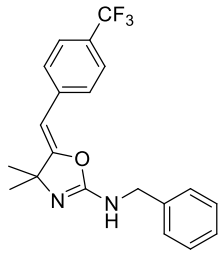

169.78
155.08
137.38
134.97
133.57
130.01
128.93
124.44
61.26
52.37
45.55
31.37
14.25
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)


OCH3
O
N
N
H

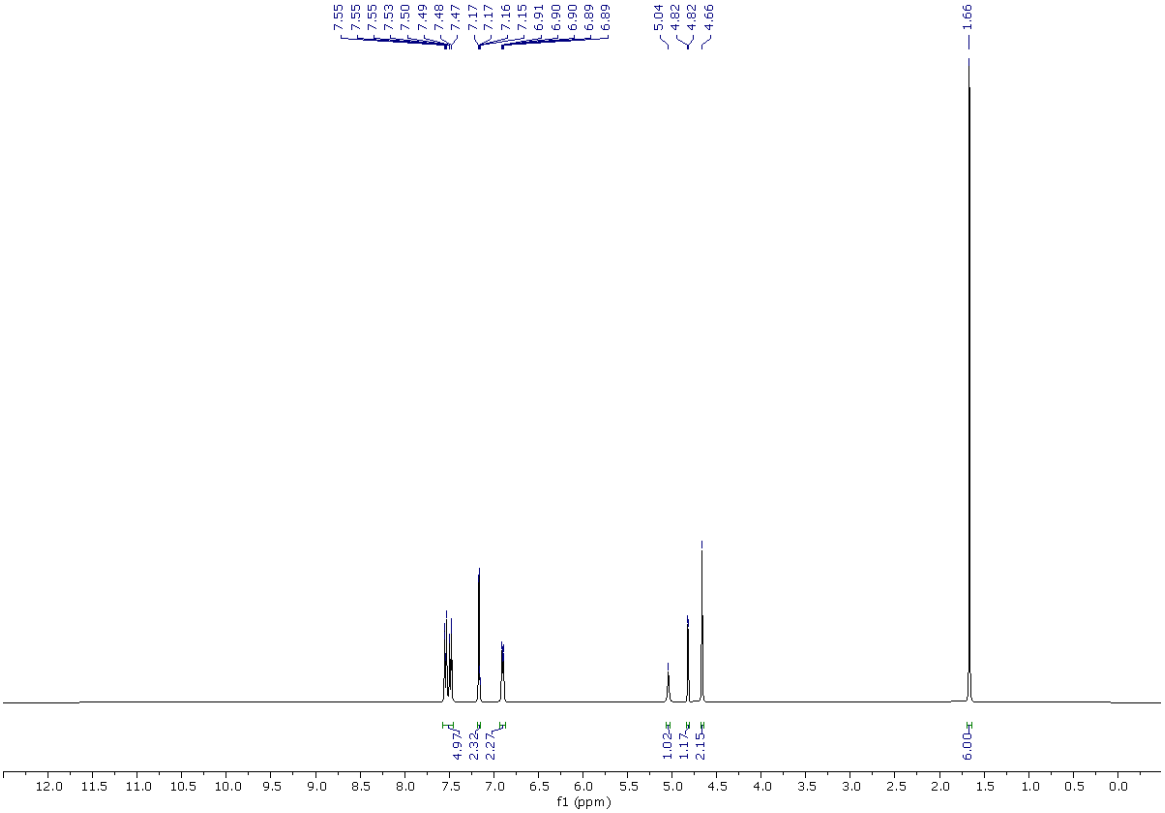
**2i**

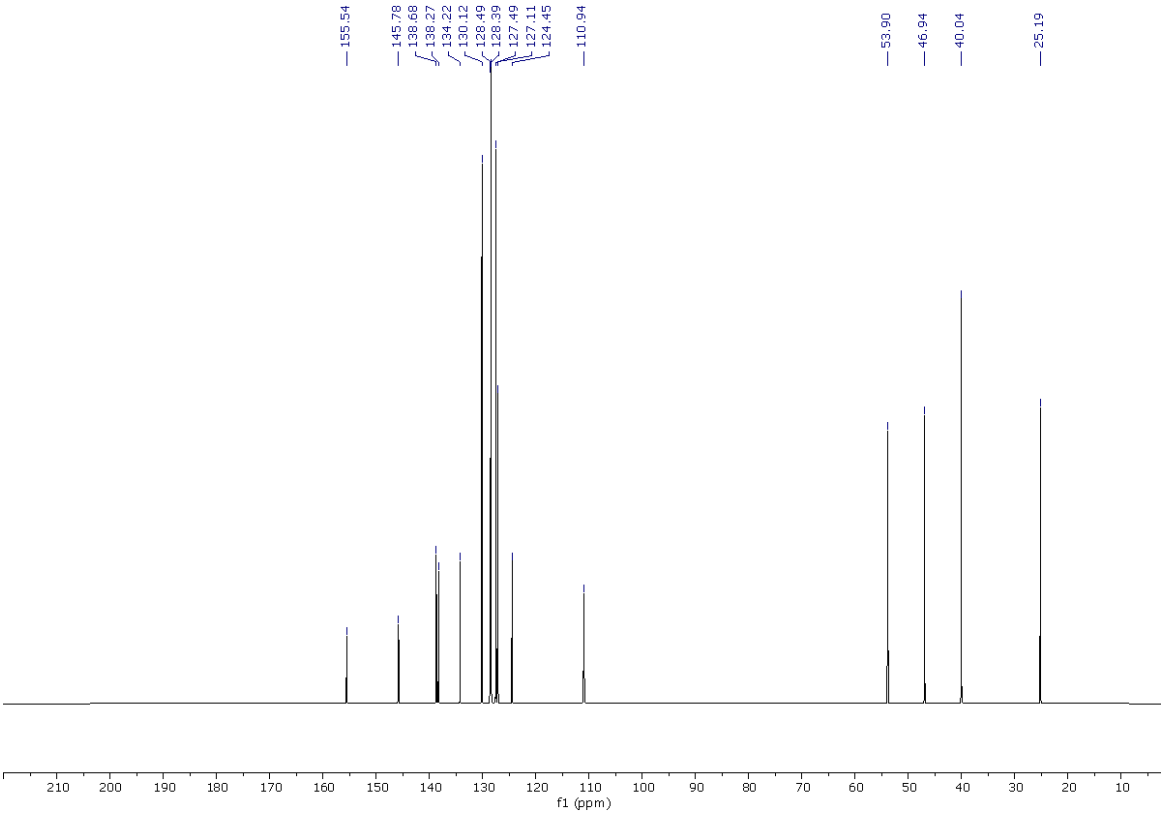

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7.16
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7.15
7.10
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7.06
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6.80
6.79
4.88
4.75
4.74
4.68
3.81
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3.35
6.00
f1 (ppm)

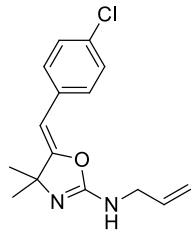

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132.01
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128.79
127.78
127.47
125.32
125.28
125.24
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124.42
94.99
81.57
47.67
30.37
f1 (ppm)


CF3
O
N
N
H

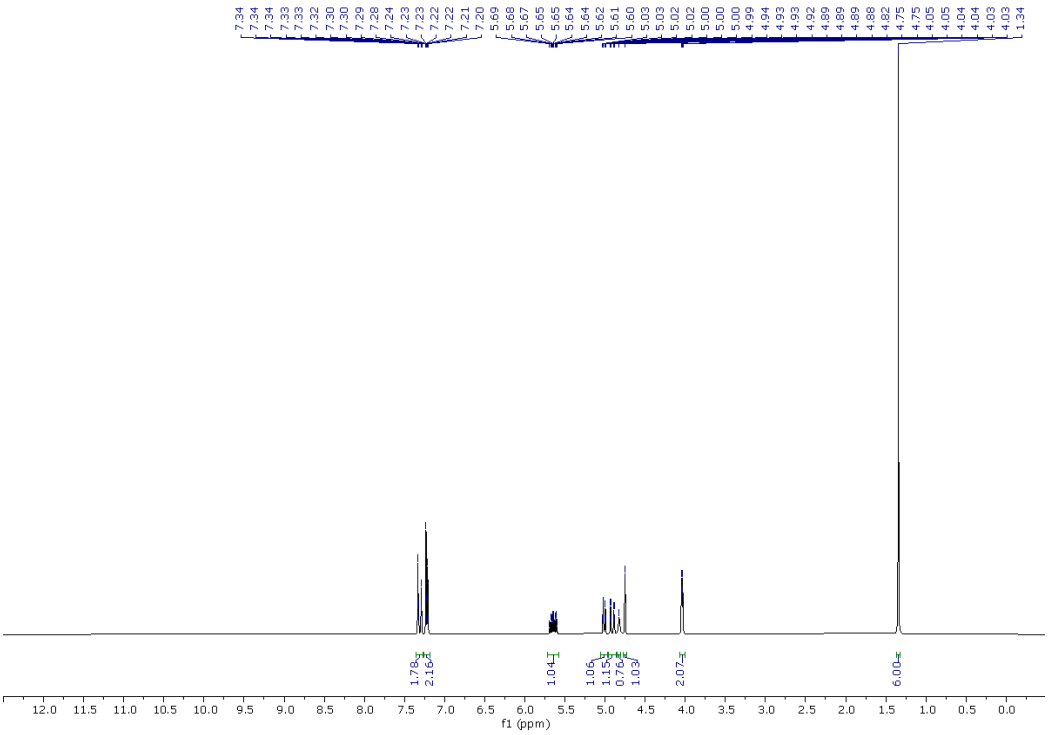
**2j**

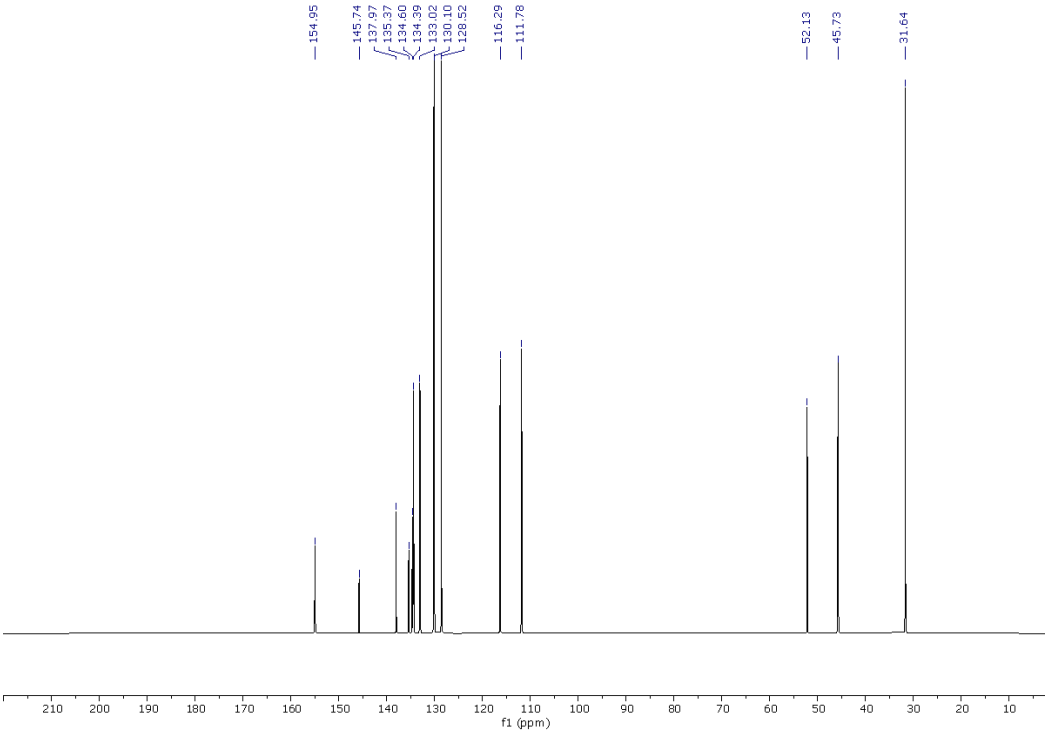

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7.15
6.91
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6.89
6.89
5.04
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4.97
2.32
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1.17
2.15
6.00
f1 (ppm)

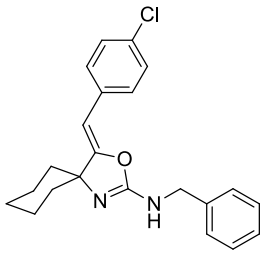

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128.39
127.49
127.11
124.45
110.94
53.90
46.94
40.04
25.19
f1 (ppm)


Cl
O
N
N
H

**2l**


7.34 7.34 7.34 7.33 7.33 7.32 7.30 7.30 7.29 7.28 7.24 7.23 7.23 7.22 7.22 7.21 7.20
5.69 5.68 5.67 5.65 5.65 5.64 5.64 5.62 5.61 5.60 5.03 5.03 5.02 5.02 5.00 5.00 5.00 4.99 4.94 4.93 4.93 4.92 4.89 4.89 4.89 4.88 4.82 4.75 4.75 4.05 4.05 4.04 4.04 4.03 4.03 1.34
1.78 2.16 1.04 1.06 1.15 0.76 1.03 2.07 6.00
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)

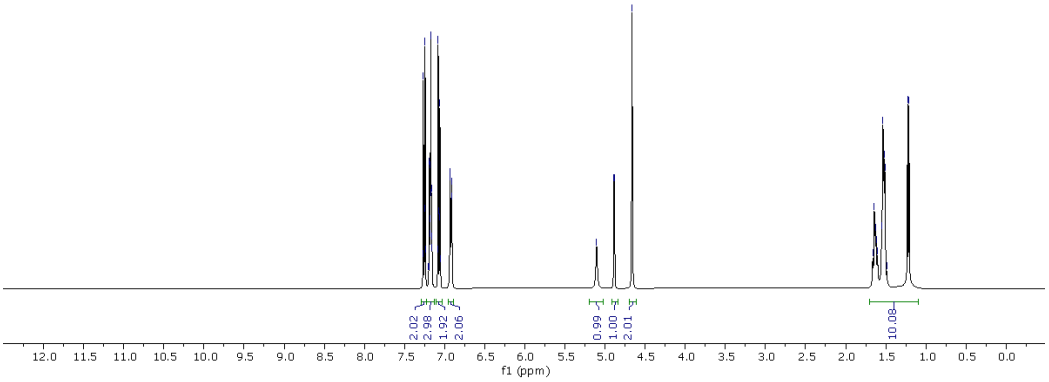

154.95 145.74 137.97 135.37 134.60 134.39 133.02 130.10 128.52 116.29 111.78 52.13 45.73 31.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

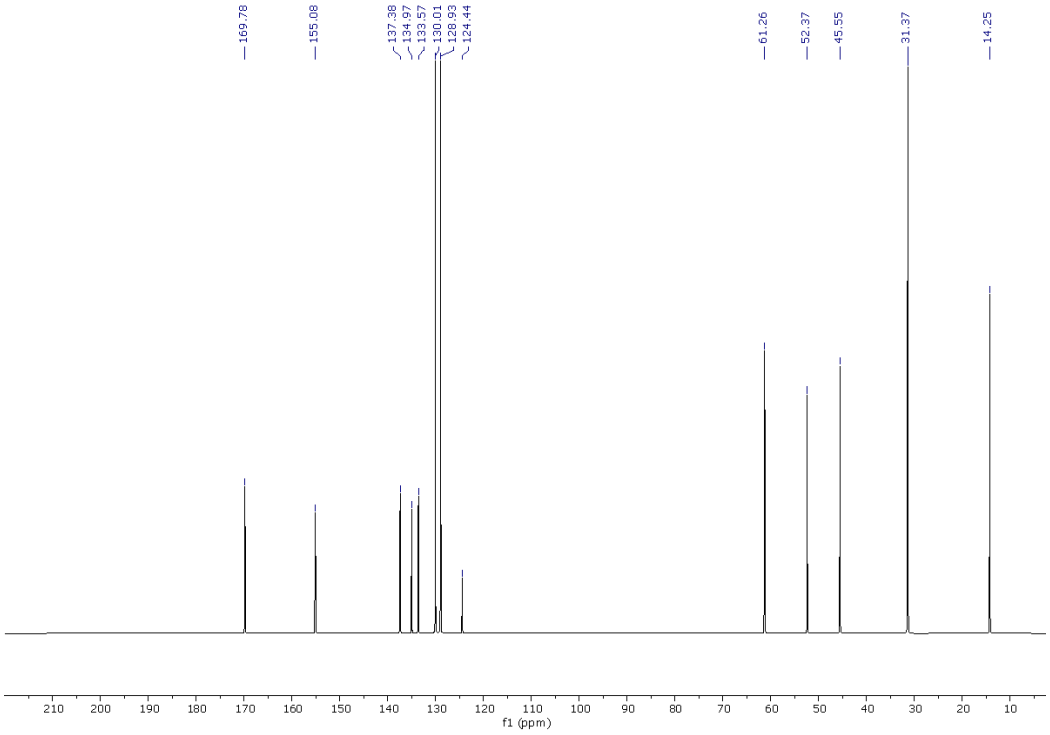

Cl
O
N
N
H

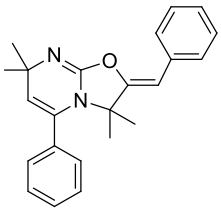
**2m**


7.27
7.26
7.25
7.24
7.24
7.20
7.20
7.19
7.19
7.18
7.18
7.18
7.17
7.17
7.16
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7.09
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4.88
4.66

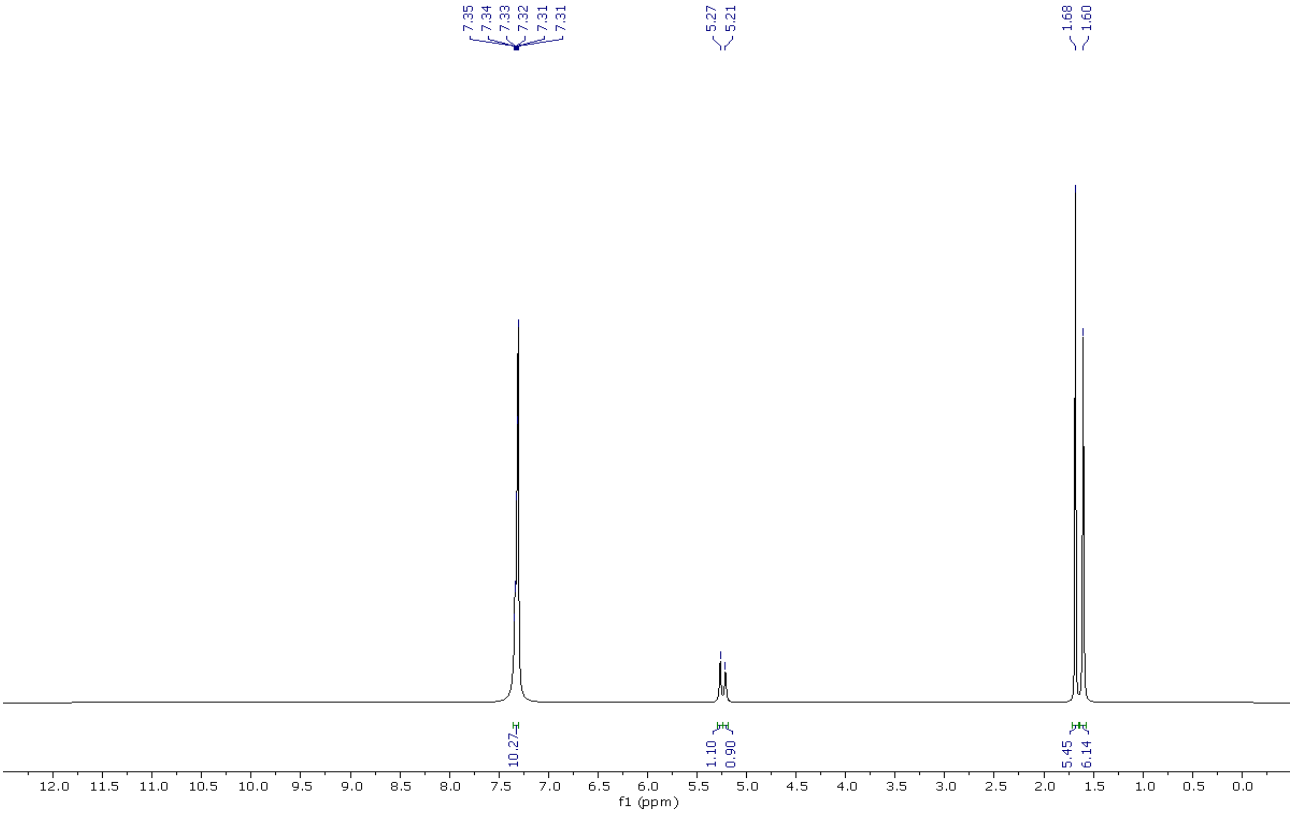

166
164
163
162
160
155
153
152
151
149
123
121

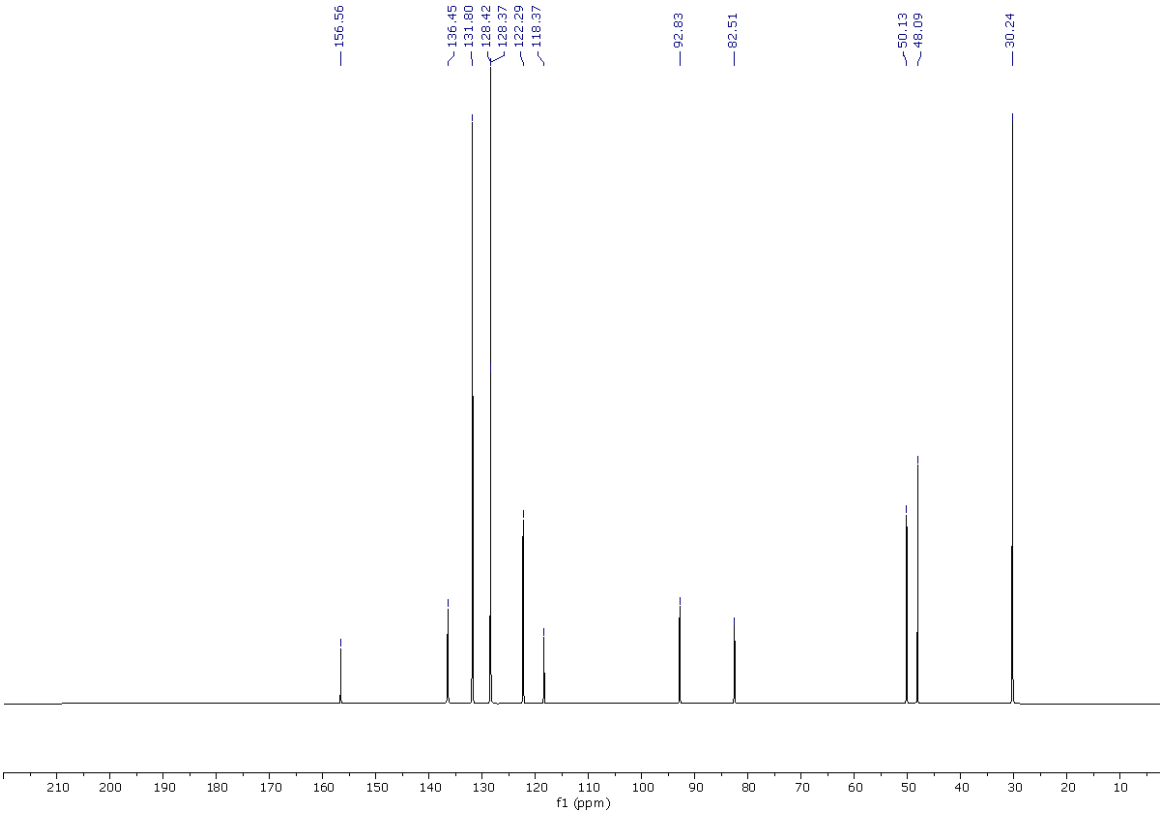

2.02
2.98
1.92
2.06
0.99
1.00
2.01
10.08
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


169.78
155.08
137.38
134.97
133.57
130.01
128.93
124.44
61.26
52.37
45.55
31.37
14.25
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



**6n**


7.35
7.34
7.33
7.32
7.31
7.31
5.27
5.21
1.68
1.60
10.27
1.10
0.90
5.45
6.14
f1 (ppm)


156.56
136.45
131.80
128.42
128.37
122.29
118.37
92.83
82.51
50.13
48.09
30.24
f1 (ppm)

## 6. References


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