

Electronic Supplementary Information

Preparation of Cyclic Imides from Alkene-tethered Amides: an Application of Homogeneous Cu(II) Catalytical System

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Abstract: A Cu-based homogeneous catalytic system was put forward for the preparation of imides from alkene-tethered amides. O₂ acted as a cheap and easily available oxygen source and a terminal oxidant. Cleavage of C=C bonds and formation of C-N bond were catalyzed by Cu(II) salts with proper nitrogen-containing ligand under 100 °C. The synthetic approach has potential of applying onto pharmaceutical synthesis. Moreover, scaled-up experiment confirmed the practical application ability.

Table of Contents

1.	Reaction condition screening of catalytic system	S3-S15
	Table S1 Ligand information	S3
	Table S2 FeCl ₃ ·6H ₂ O	S4
	Table S3 Co(BF ₄) ₂ ·6H ₂ O	S5
	Table S4 Ni(acac) ₂	S6
	Table S5 Cu(acac) ₂	S7
	Table S6 ZnCl ₂	S8
	Table S7 MnCl ₂ ·4H ₂ O	S9
	Table S8 PdCl ₂	S10
	Table S9 RuCl ₃	S11
	Table S10 RhCl ₃ ·3H ₂ O	S12
	Table S11 IrCl ₃	S13
	Table S12 Screening of Cu salts	S14
	Table S13 Screening of Cu salts	S14
2.	Studies on the catalytic system	S15-S16
	Table S14 Control experiments	S15
	Table S15 Quantitative data for the kinetic studies	S16
	Scheme S1 Process between intermediate B and C	S16
3.	NMR spectra of substrates, products and intermediate	S17-S57
4.	ESI-MS measurement: instrument parameters and results	S58
	Figure S1 ESI-MS spectra of mechanism research	S59

1. Reaction condition screening of catalytic system

Table S1 Ligand information

Entry	Code	Name
1	A	2,9-dimethyl-1,10-phenanthroline
2	B	2,2'-bipyridine
3	C	2,2'-bipyrimidine
4	D	5H-cyclopenta[2,1- <i>b</i> :3,4- <i>b'</i>]dipyridin-5-one
5	E	4-methyl-2-(pyridin-2-yl)-4,5-dihydrooxazole
6	F	(S)-4-isopropyl-2-(pyridin-2-yl)-4,5-dihydrooxazole
7	G	2,2'-(propane-2,2-diyl)bis(4,5-dihydrooxazole)
8	H	2,2'-(propane-2,2-diyl)bis(4-isopropyl-4,5-dihydrooxazole)
9	I	<i>N</i> ¹ , <i>N</i> ¹ , <i>N</i> ² , <i>N</i> ² -tetramethylethane-1,2-diamine
10	J	<i>N</i> ¹ , <i>N</i> ¹ , <i>N</i> ³ , <i>N</i> ³ -tetramethylpropane-1,3-diamine
11	K	<i>N</i> ¹ , <i>N</i> ¹ , <i>N</i> ⁴ , <i>N</i> ⁴ -tetramethylbutane-1,4-diamine
12	L	<i>N</i> ¹ , <i>N</i> ¹ , <i>N</i> ⁵ , <i>N</i> ⁵ -tetramethylpentane-1,5-diamine
13	M	triethylamine
14	N	1 <i>H</i> -imidazole
15	O	(1 <i>E</i> ,2 <i>E</i>)- <i>N</i> ¹ , <i>N</i> ² -dicyclohexylethane-1,2-diimine
16	P	7-(tert-butyl)-2,5-dimethyl-3,4-dihydro-2 <i>H</i> -pyrano[2,3- <i>b</i>]quinoline
17	Q	1,2-bis(phenylsulfinyl)ethane
18	R	ethyl(4-methoxyphenyl)sulfane
19	S	triphenylphosphane
20	T	1,2-bis(diphenylphosphanyl)ethane
21	U	2,2'-bis(diphenylphosphanyl)-1,1'-binaphthalene
22	V	(oxybis(2,1-phenylene))bis(diphenylphosphane)

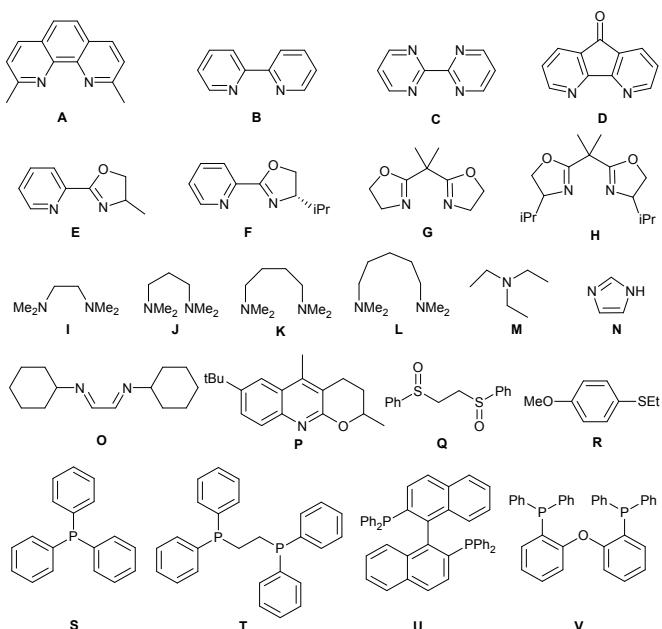
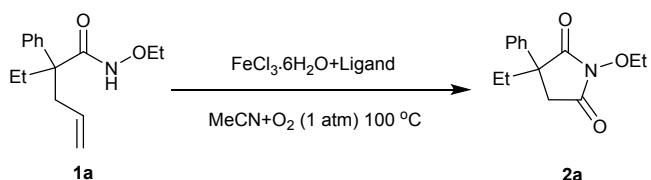


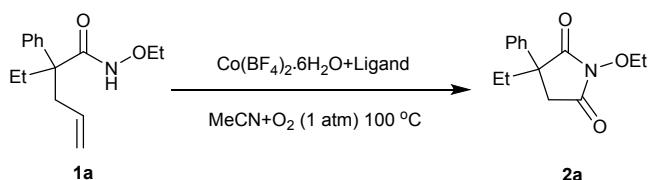
Table S2 Catalytic performance of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$: effect of ligands^a

Entry	Ligand Code	Yield/% ^b
1	A	0.5
2	B	Not detected
3	C	Not detected
4	D	0.2
5	E	0.5
6	F	Not detected
7	G	Not detected
8	H	Not detected
9	I	Not detected
10	J	Not detected
11	K	Not detected
12	L	Not detected
13	M	Not detected
14	N	Not detected
15	O	Not detected
16	P	0.8
17	Q	Not detected
18	R	Not detected
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O_2 (1 atm); 100 °C; 24 h.

^b Determined by ^1H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S3 Catalytic performance of $\text{Co}(\text{BF}_4)_2 \cdot 6\text{H}_2\text{O}$: effect of ligands^a

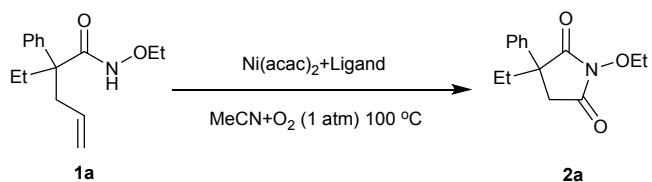


Entry	Ligand Code	Yield/% ^b
1	A	Not detected
2	B	Not detected
3	C	4.8
4	D	3.2
5	E	Not detected
6	F	Not detected
7	G	Not detected
8	H	Not detected
9	I	0.5
10	J	1
11	K	Not detected
12	L	Not detected
13	M	Not detected
14	N	Not detected
15	O	0.8
16	P	1.2
17	Q	Not detected
18	R	Not detected
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; $\text{Co}(\text{BF}_4)_2 \cdot 6\text{H}_2\text{O}$, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O_2 (1 atm); 100 °C; 24 h.

^b Determined by ^1H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S4 Catalytic performance of Ni(acac)₂: effect of ligands^a

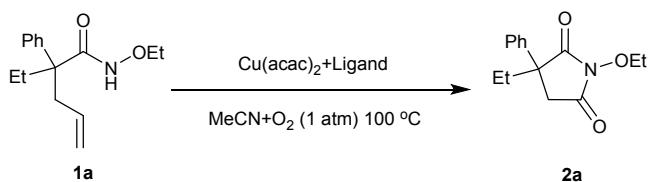


Entry	Ligand Code	Yield/% ^b
1	A	0.5
2	B	0.4
3	C	0.6
4	D	Not detected
5	E	Not detected
6	F	Not detected
7	G	Not detected
8	H	Not detected
9	I	Not detected
10	J	Not detected
11	K	Not detected
12	L	0.5
13	M	0.8
14	N	0.6
15	O	Not detected
16	P	0.5
17	Q	Not detected
18	R	Not detected
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; Ni(acac)₂, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S5 Catalytic performance of Cu(acac)₂: effect of ligands^a

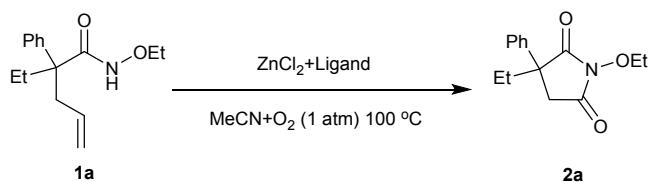


Entry	Ligand Code	Yield/% ^b
1	A	85
2	B	63
3	C	72
4	D	28
5	E	51
6	F	44
7	G	42
8	H	25
9	I	8
10	J	6
11	K	5
12	L	3
13	M	5
14	N	33
15	O	8
16	P	18
17	Q	2
18	R	1
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; Cu(acac)₂, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S6 Catalytic performance of ZnCl₂: effect of ligands^a

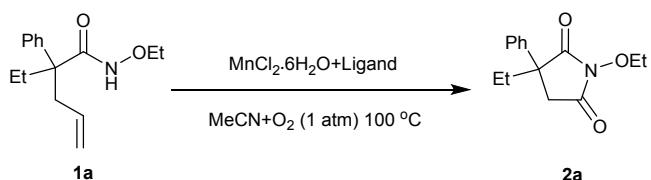


Entry	Ligand Code	Yield/% ^b
1	A	0.5
2	B	0.5
3	C	0.3
4	D	Not detected
5	E	Not detected
6	F	Not detected
7	G	Not detected
8	H	Not detected
9	I	Not detected
10	J	Not detected
11	K	Not detected
12	L	0.8
13	M	0.4
14	N	0.3
15	O	Not detected
16	P	0.5
17	Q	0.6
18	R	Not detected
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; ZnCl₂, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S7 Catalytic performance of MnCl₂·4H₂O: effect of ligands^a

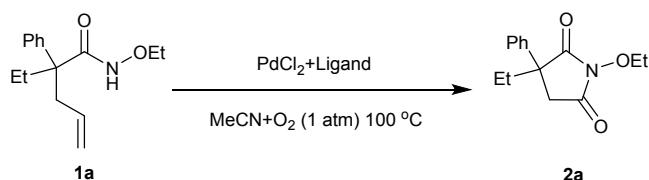


Entry	Ligand Code	Yield/% ^b
1	A	Not detected
2	B	Not detected
3	C	Not detected
4	D	Not detected
5	E	Not detected
6	F	0.4
7	G	0.5
8	H	0.6
9	I	0.3
10	J	Not detected
11	K	Not detected
12	L	Not detected
13	M	Not detected
14	N	Not detected
15	O	Not detected
16	P	Not detected
17	Q	0.3
18	R	0.5
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; MnCl₂·4H₂O, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S8 Catalytic performance of PdCl₂: effect of ligands^a

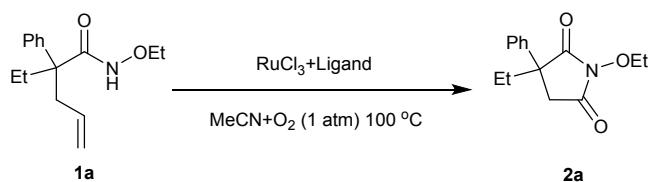


Entry	Ligand Code	Yield/% ^b
1	A	0.5
2	B	0.6
3	C	5.6
4	D	3.2
5	E	Not detected
6	F	Not detected
7	G	Not detected
8	H	Not detected
9	I	Not detected
10	J	Not detected
11	K	Not detected
12	L	1.2
13	M	Not detected
14	N	1
15	O	Not detected
16	P	0.8
17	Q	0.5
18	R	Not detected
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; PdCl₂, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S9 Catalytic performance of RuCl₃: effect of ligands^a

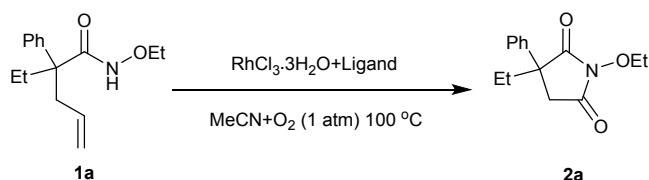


Entry	Ligand Code	Yield/% ^b
1	A	Not detected
2	B	Not detected
3	C	Not detected
4	D	Not detected
5	E	Not detected
6	F	Not detected
7	G	Not detected
8	H	4.8
9	I	Not detected
10	J	1
11	K	Not detected
12	L	Not detected
13	M	Not detected
14	N	0.5
15	O	1.5
16	P	2
17	Q	0.5
18	R	Not detected
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; RuCl₃, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S10 Catalytic performance of RhCl₃·3H₂O: effect of ligands^a

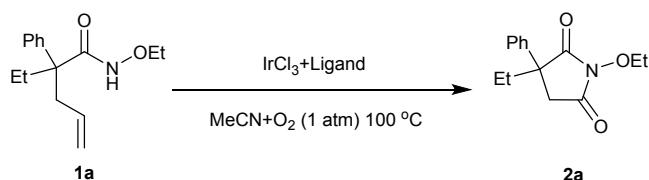


Entry	Ligand Code	Yield/% ^b
1	A	Not detected
2	B	Not detected
3	C	1
4	D	0.5
5	E	Not detected
6	F	0.6
7	G	5.5
8	H	Not detected
9	I	Not detected
10	J	Not detected
11	K	Not detected
12	L	Not detected
13	M	Not detected
14	N	Not detected
15	O	Not detected
16	P	0.5
17	Q	Not detected
18	R	0.2
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; RhCl₃·3H₂O, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

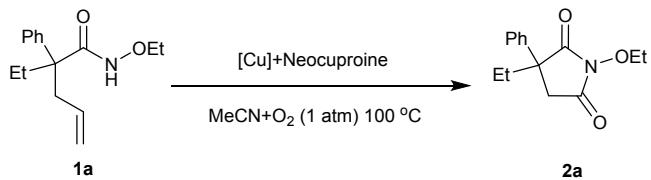
Table S11 Catalytic performance of IrCl₃: effect of ligands^a



Entry	Ligand Code	Yield/% ^b
1	A	3.2
2	B	0.5
3	C	0.2
4	D	Not detected
5	E	Not detected
6	F	Not detected
7	G	Not detected
8	H	Not detected
9	I	0.5
10	J	Not detected
11	K	1
12	L	Not detected
13	M	0.5
14	N	Not detected
15	O	Not detected
16	P	Not detected
17	Q	Not detected
18	R	Not detected
19	S	Not detected
20	T	Not detected
21	U	Not detected
22	V	Not detected

^a Reaction conditions: **1a**, 0.1 mmol; IrCl₃, 0.01 mmol (10 mol%); Ligand, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

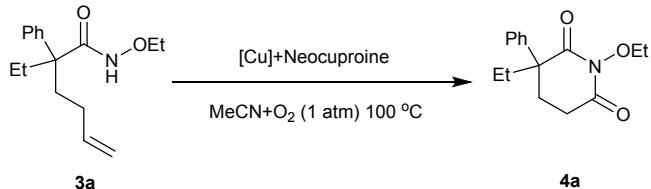
^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S12 Screening of Cu salts^a

Entry	Cu Salt	Yield/% ^b
1	CuSO ₄ ·5H ₂ O	75
2	Cu(NO ₃) ₂ ·2.5H ₂ O	78
3	Cu(BF ₄) ₂ ·2H ₂ O	82
4	CuF ₂	81
5	CuCl ₂ ·2H ₂ O	80
6	CuBr ₂	77
7	Cu(acac) ₂	85
8	CuAc ₂ ·H ₂ O	78

^a Reaction conditions: **1a**, 0.1 mmol; Cu salts, 0.01 mmol (10 mol%); Neocuproine, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

Table S13 Screening of Cu salts^a

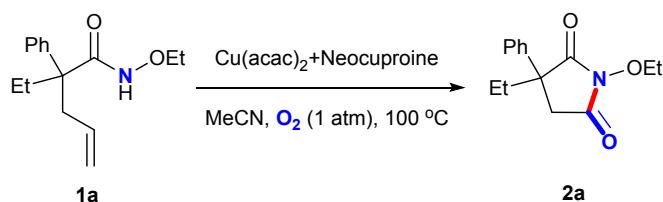
Entry	Cu Salt	Yield/% ^b
1	CuSO ₄ ·5H ₂ O	44
2	Cu(NO ₃) ₂ ·2.5H ₂ O	47
3	Cu(BF ₄) ₂ ·2H ₂ O	50
4	CuF ₂	58
5	CuCl ₂ ·2H ₂ O	49
6	CuBr ₂	45
7	Cu(acac) ₂	52
8	CuAc ₂ ·H ₂ O	48

^a Reaction conditions: **3a**, 0.1 mmol; Cu salts, 0.01 mmol (10 mol%); Neocuproine, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h.

^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

2. Studies on the catalytic system

Table S14 Control experiments^a

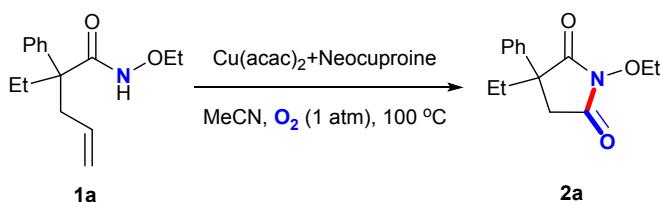


Entry	Deviation from standard conditions	2a yield / % ^b
1	No Cu(acac) ₂	0
2	No neocuproine	0
3	No O ₂	0
4	No neocuproine or O ₂	0

^a Standard conditions: **1a**, 0.1 mmol; Cu(acac)₂, 0.01 mmol (10 mol%); ligand neocuproine, 0.015 mmol (15 mol%);

MeCN, 1.5 mL; O₂ (1 atm); 100 °C; 24 h. ^b Determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

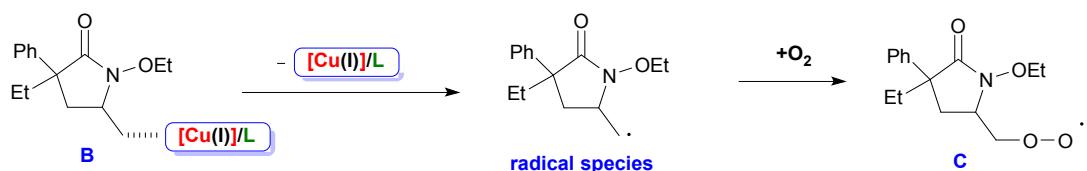
Table S15 Quantitative data for the kinetic studies^{a,b}



Entry	Time / h	1a / %	E / %	2a / %
1	0	100	0	0
2	0.5	60	30	8
3	1	40	27	18
4	1.5	35	21	25
5	2	25	18	38
6	3	19	16	49
7	6	17	13	58
8	9	16	10	63
9	12	15	8	68
10	15	12	5	72
11	18	10	3	79
12	21	9	2	82
13	24	8	2	85
14	27	8	2	85

^a Reaction conditions: 1a, 0.1 mmol; Cu(acac)₂, 0.01 mmol (10 mol%); ligand neocuproine, 0.015 mmol (15 mol%); MeCN, 1.5 mL; O₂ (1 atm); 100 °C. ^b Yields were determined by ¹H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard.

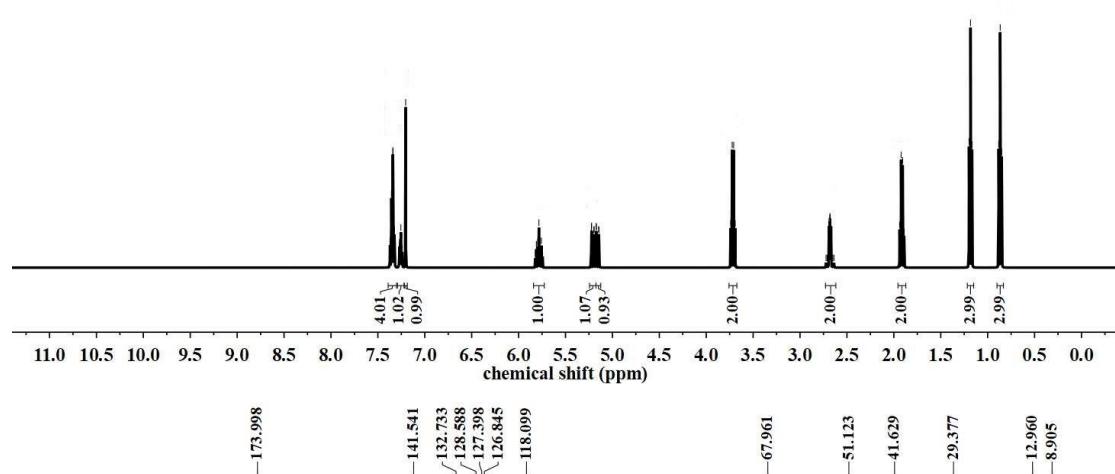
Scheme S1 Process between intermediate B and C



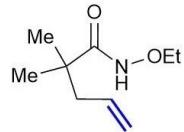
3. NMR spectra of substrates, products and intermediate



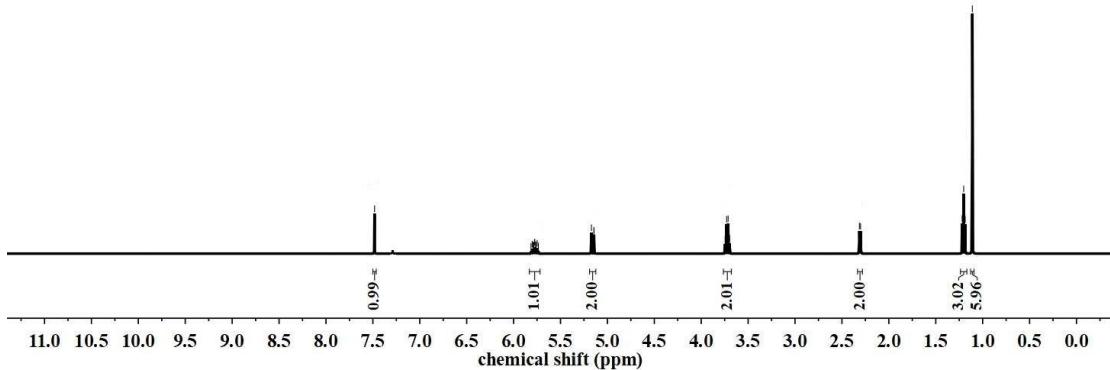
¹H NMR (400 M, CDCl₃)



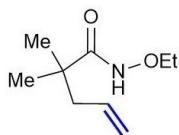
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 2.299
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 1.110



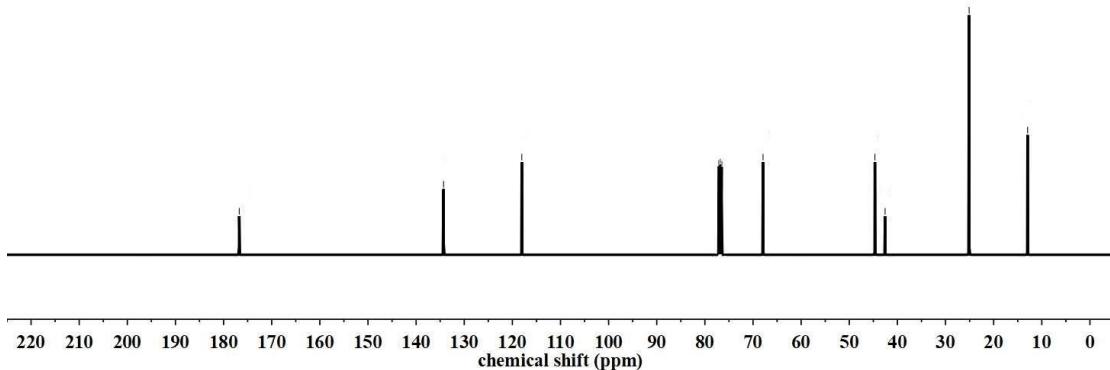
¹H NMR (400 M, CDCl₃)



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 ~42.558
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 -12.955

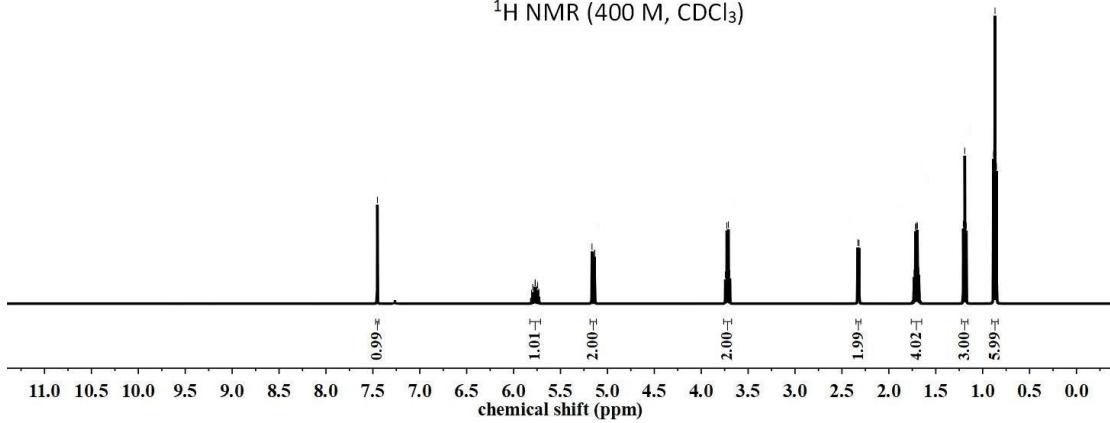


¹³C NMR (100 M, CDCl₃)



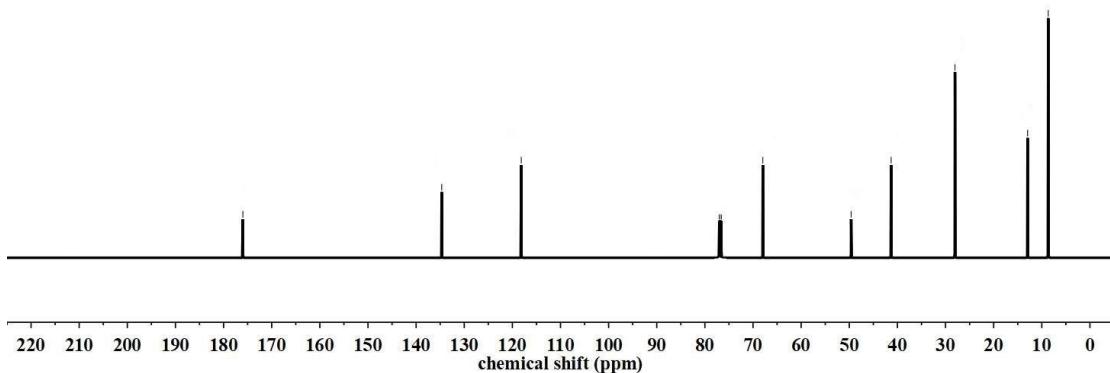
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1.715
1.713
1.699
1.696
1.683
1.680
1.297
1.207
1.191
1.175
0.884
0.868

¹H NMR (400 M, CDCl₃)

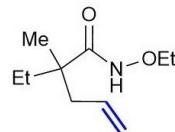


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-118.154
-67.957
-49.616
-41.297
-28.038
-12.951
-8.659

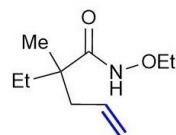
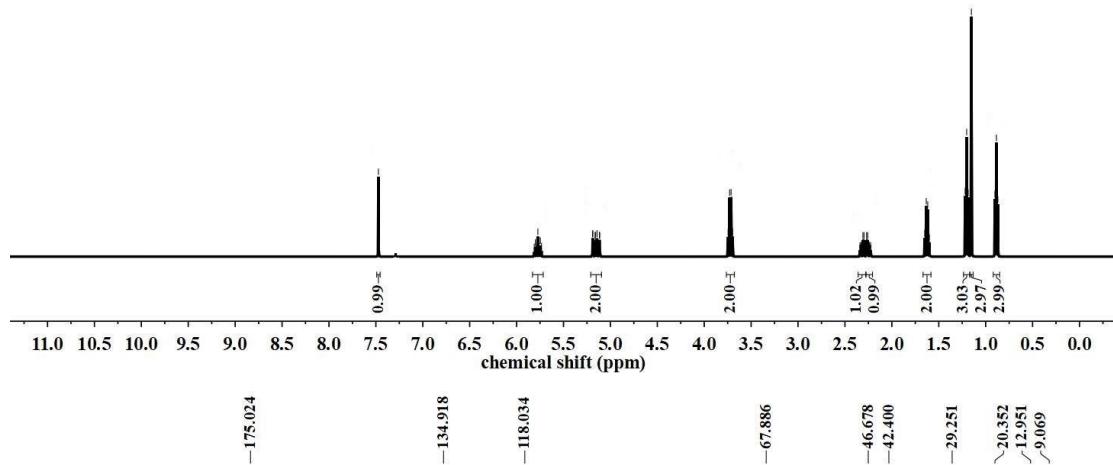
¹³C NMR (100 M, CDCl₃)



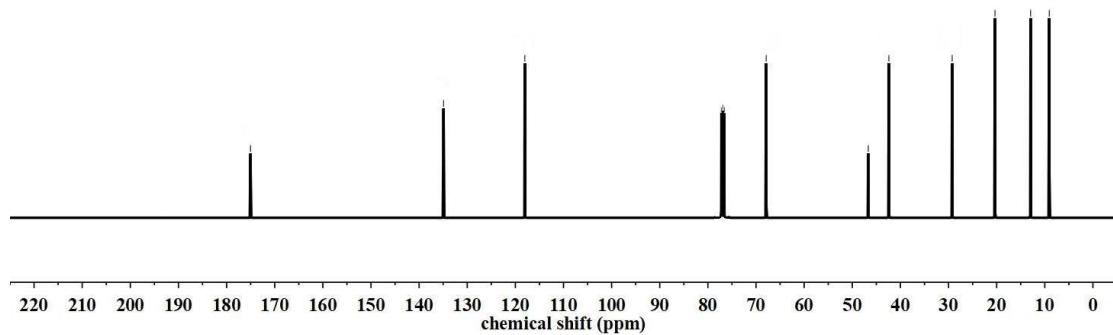
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 5.190
 5.188
 5.186
 5.165
 5.163
 5.161
 5.159
 5.145
 5.143
 5.141
 5.139
 5.119
 5.117
 5.115
 5.112
 5.110
 3.745
 3.729
 3.713
 3.697
 2.339
 2.327
 2.310
 2.308
 2.306
 2.298
 2.296
 2.294
 2.271
 2.269
 2.267
 2.259
 2.257
 2.255
 2.239
 2.237
 2.226
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 0.869

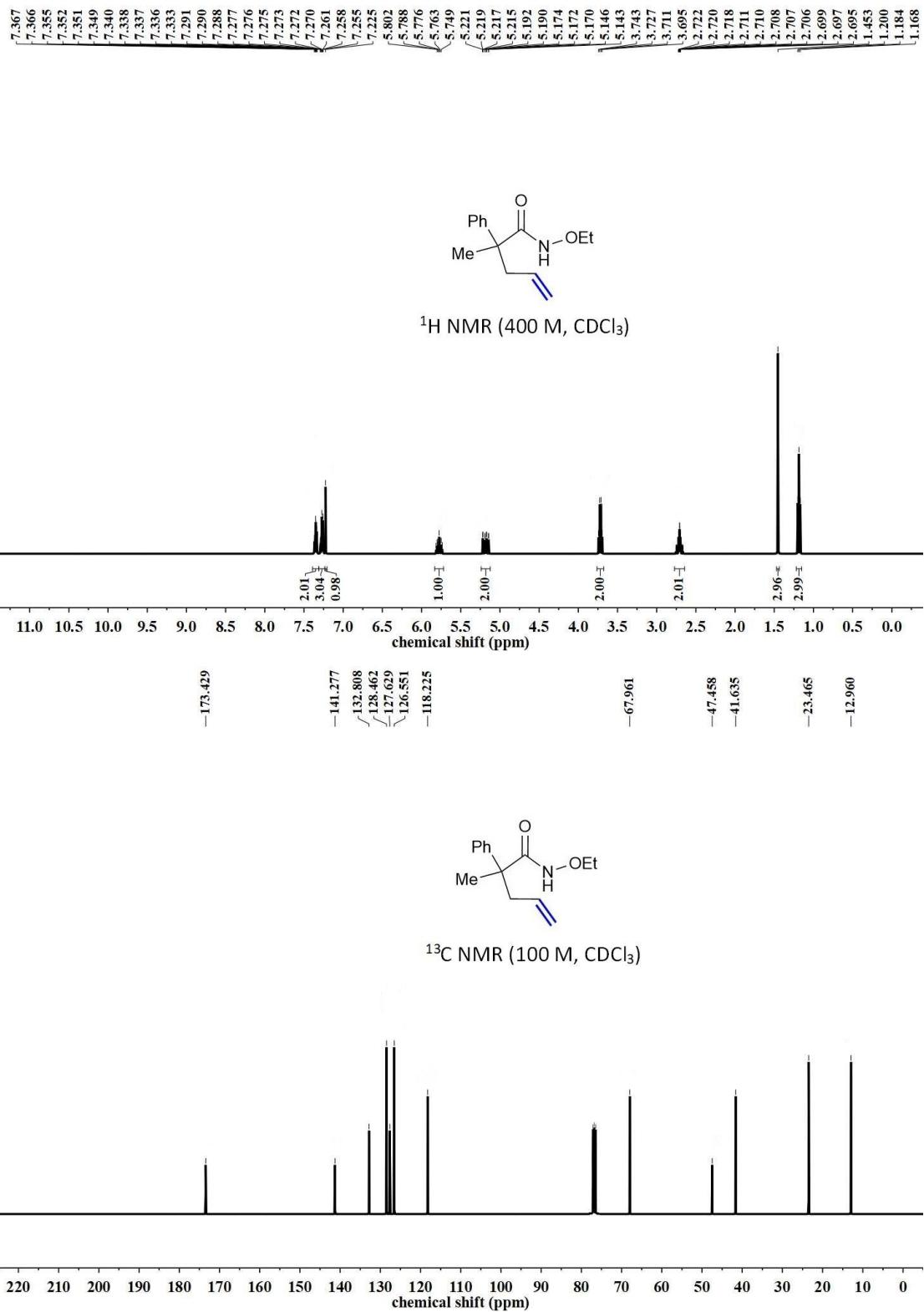


¹H NMR (400 M, CDCl₃)

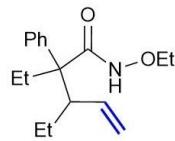


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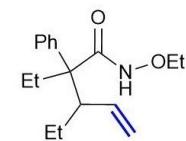
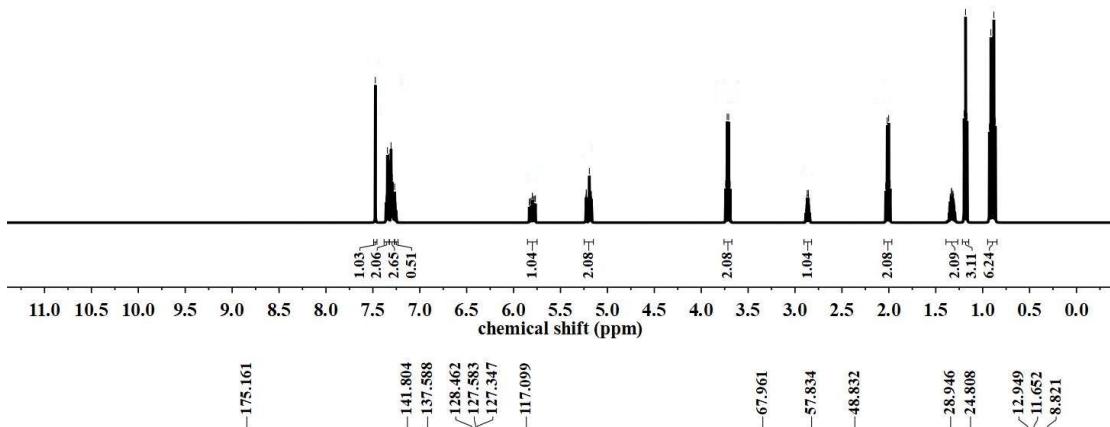




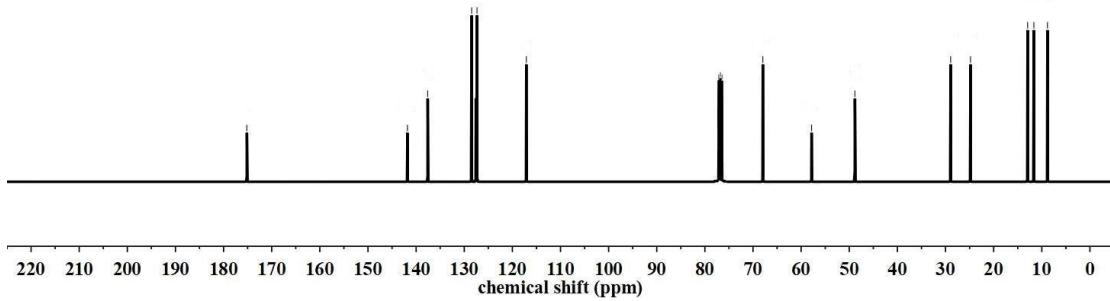
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7.333
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7.329
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7.302
7.294
7.290
7.285
7.267
5.801
5.800
5.780
5.767
5.228
5.226
5.224
5.194
5.192
5.190
5.188
5.174
5.172
3.739
3.723
3.707
3.691
2.872
2.870
2.858
2.856
2.034
2.018
2.002
1.986
1.346
1.341
1.332
1.330
1.327
1.325
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0.865

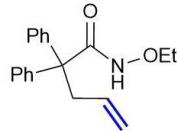
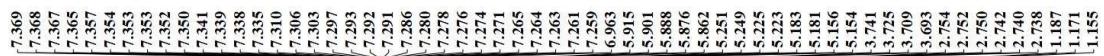


¹H NMR (400 M, CDCl₃)

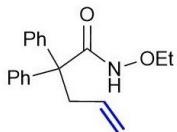
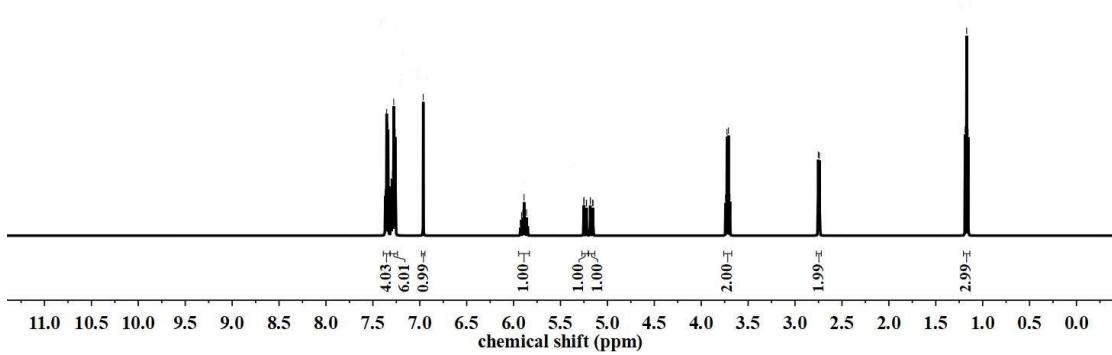


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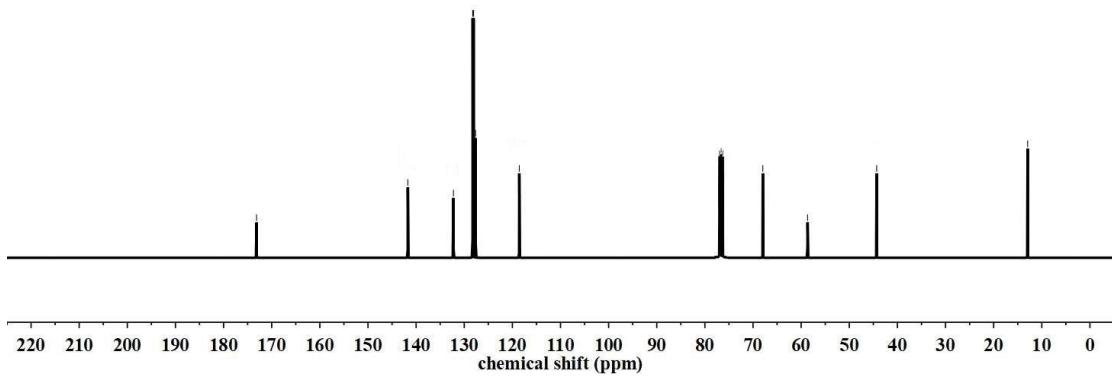




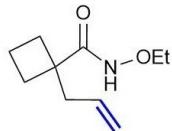
¹H NMR (400 M, CDCl₃)



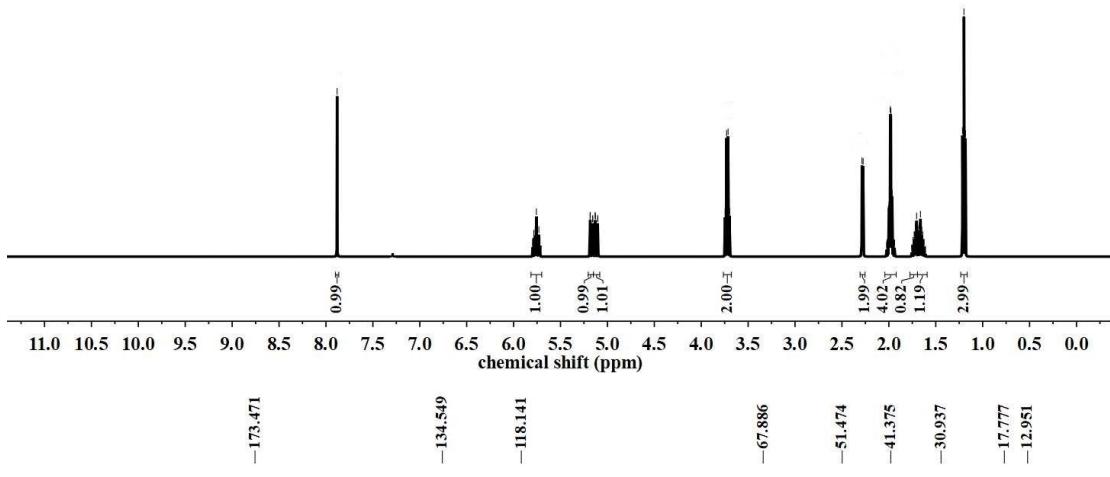
¹³C NMR (100 M, CDCl₃)



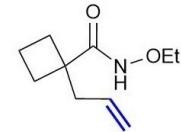
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5.154
5.132
5.130
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5.126
5.106
5.104
5.101
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3.729
3.713
3.697
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2.010
2.000
1.999
1.995
1.993
1.992
1.985
1.979
1.971
1.970
1.968
1.965
1.964
1.954
1.729
1.718
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1.199
1.183



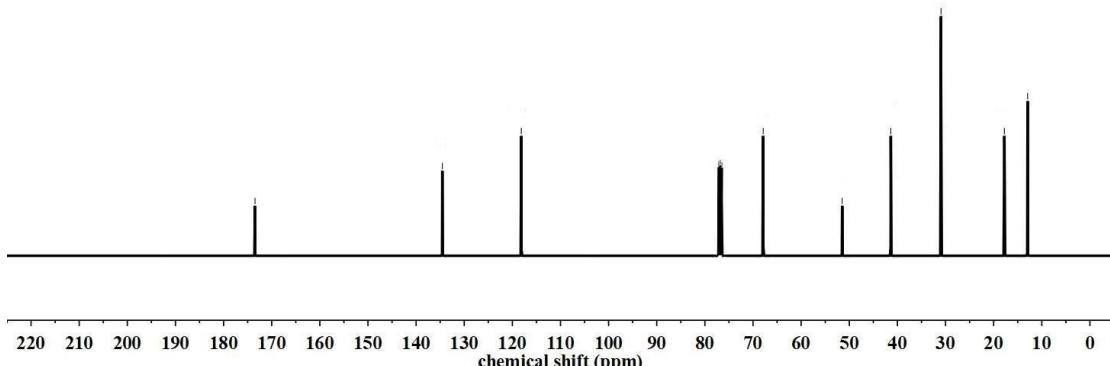
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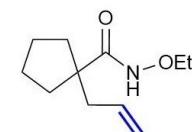
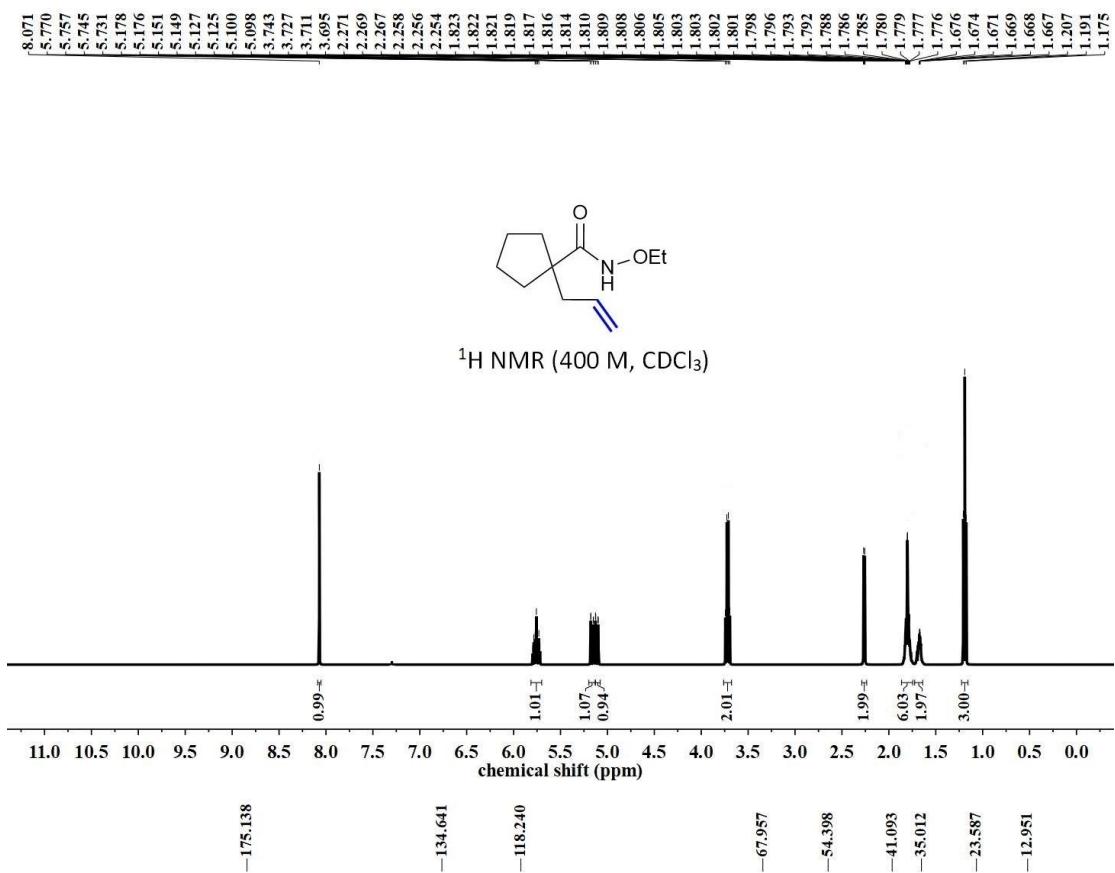


-173.471
-134.549
-118.141
-67.886
-51.474
-41.375
-30.937
-17.777
-12.951

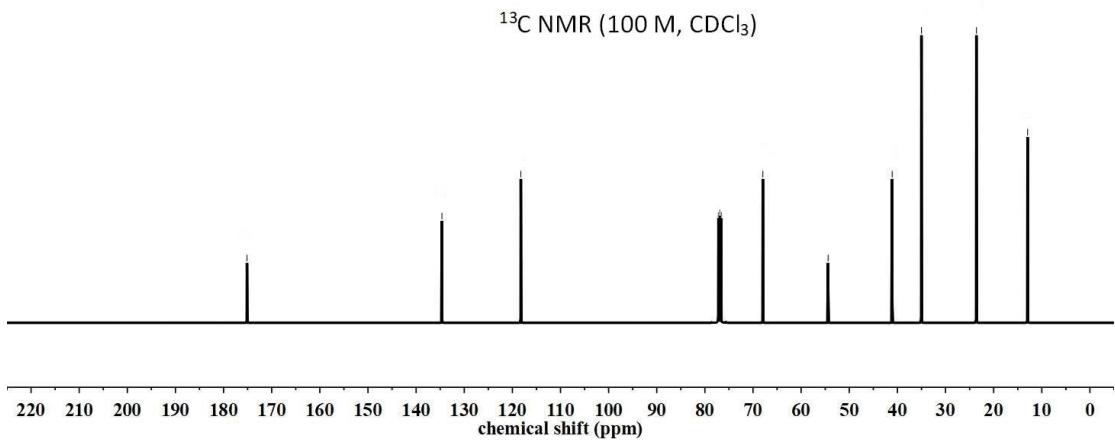


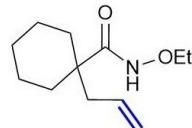
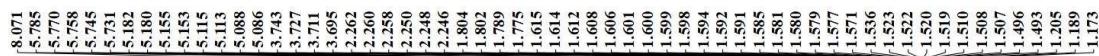
¹³C NMR (100 M, CDCl₃)



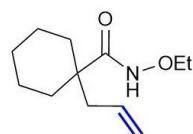
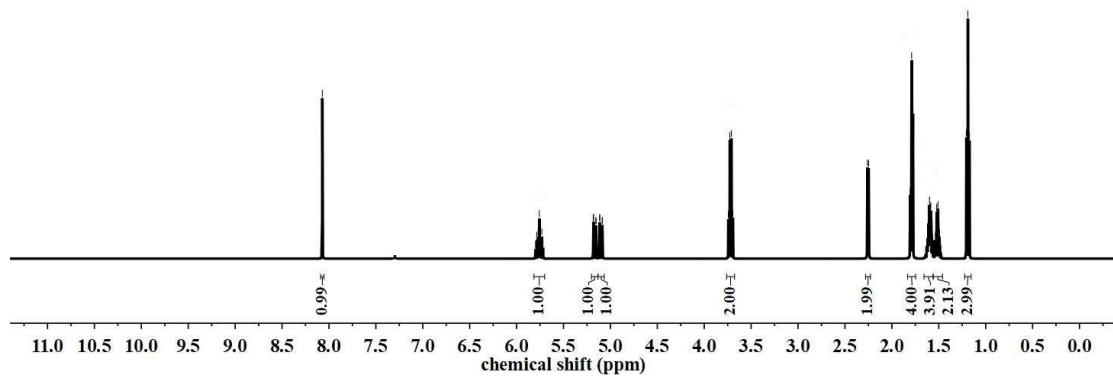


¹³C NMR (100 M, CDCl₃)

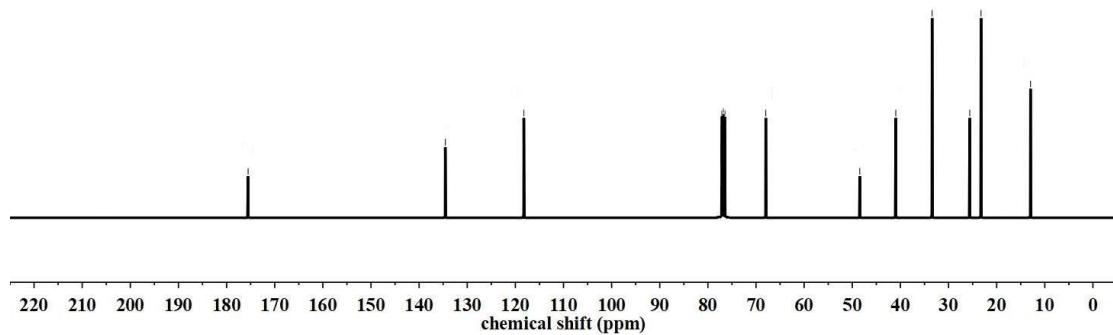




¹H NMR (400 M, CDCl₃)



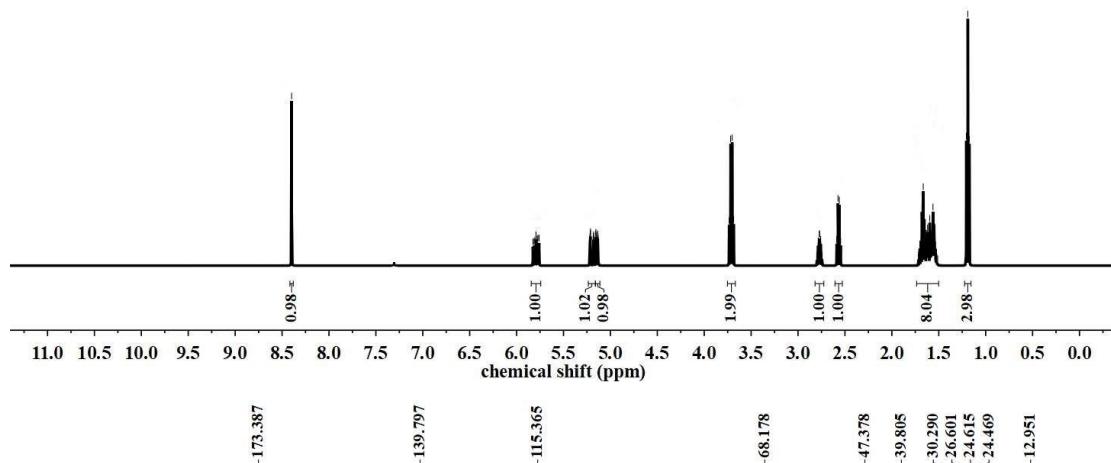
¹³C NMR (100 M, CDCl₃)



8.398
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5.792
5.216
5.213
5.211
5.180
5.178
5.156
5.154
5.152
5.136
5.134
3.734
3.718
3.702
3.686
2.773
2.771
2.574
2.560
1.680
1.679
1.678
1.677
1.668
1.666
1.665
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1.190
1.174



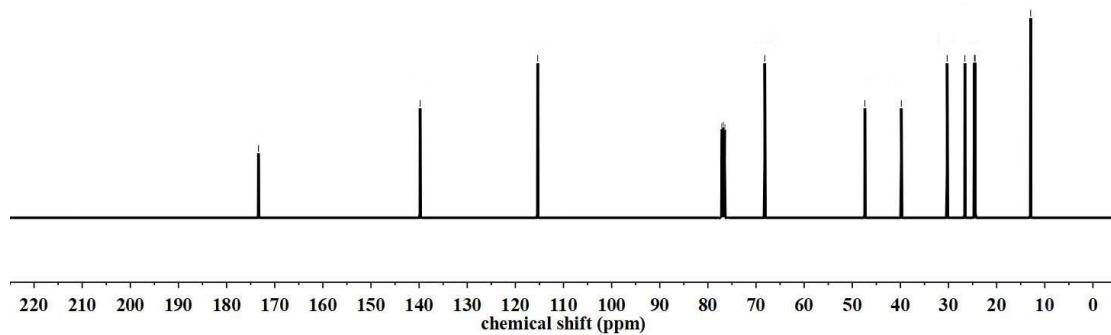
¹H NMR (400 M, CDCl₃)



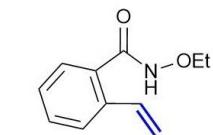
-173.387 -139.797 -115.365 -68.178 -47.378 -39.805 /30.290 26.601 <24.615 <24.469 -12.951



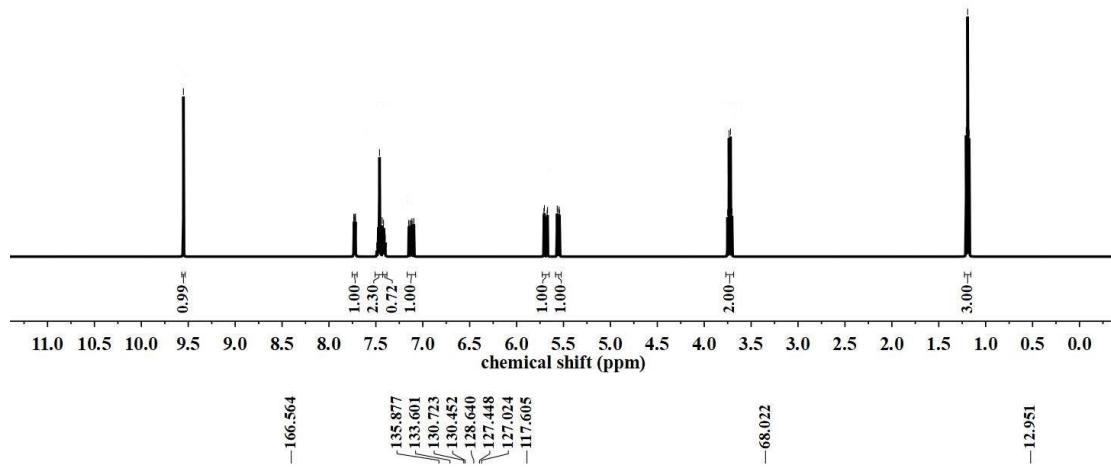
¹³C NMR (100 M, CDCl₃)



9.551	[7.735
	7.735
	7.732
	7.732
	7.721
	7.719
	7.718
	7.717
	7.491
	7.488
	7.488
	7.478
	7.476
	7.473
	7.466
	7.466
	7.465
	7.463
	7.461
	7.459
	7.452
	7.451
	7.445
	7.433
	7.427
	7.420
	7.418
	7.415
	7.412
	7.406
	7.400
	7.149
	7.148
	7.129
	7.128
	7.115
	7.114
	7.095
	7.095
	5.562
	5.547
	5.542
	5.675
	3.752
	3.736
	3.720
	3.704
	1.208
	1.192
	1.176



¹H NMR (400 M, CDCl₃)

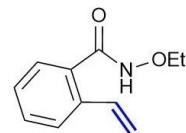


-166.564

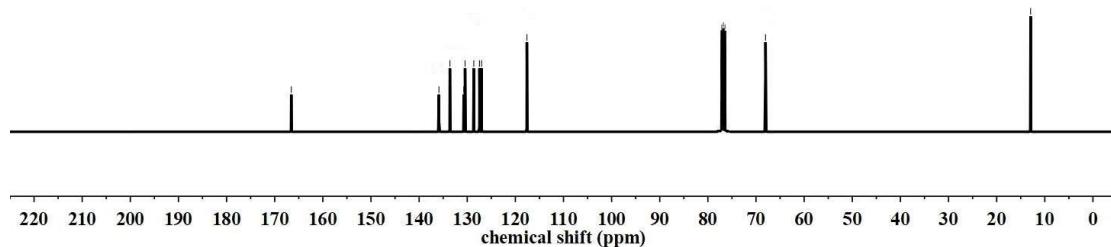
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130.452
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-68.022

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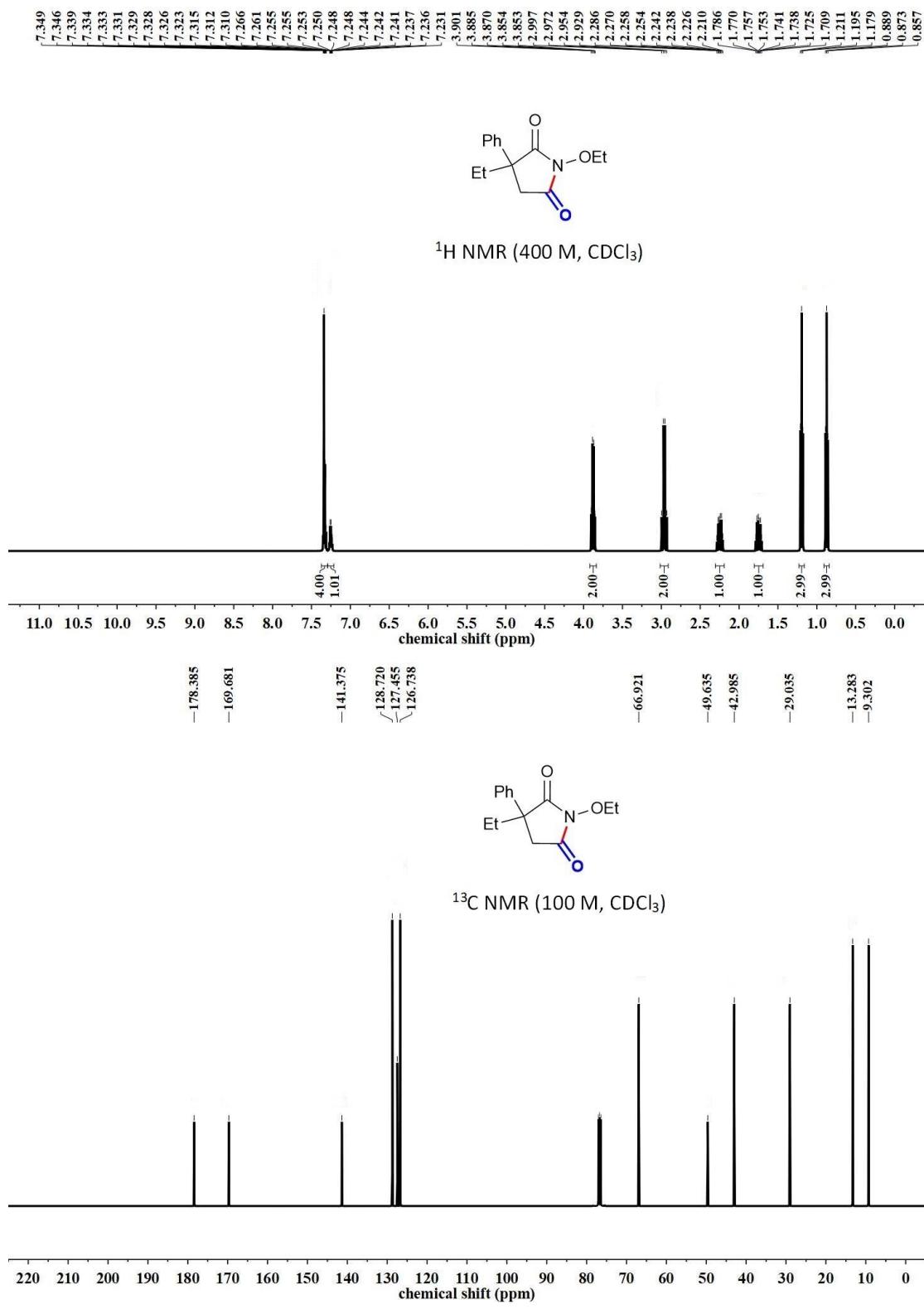


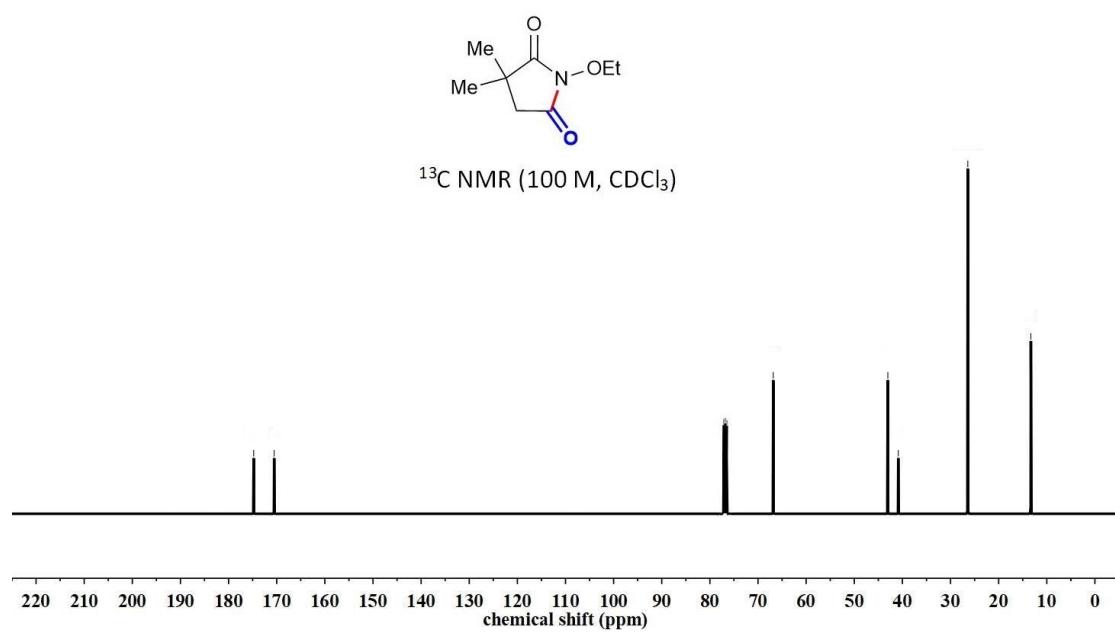
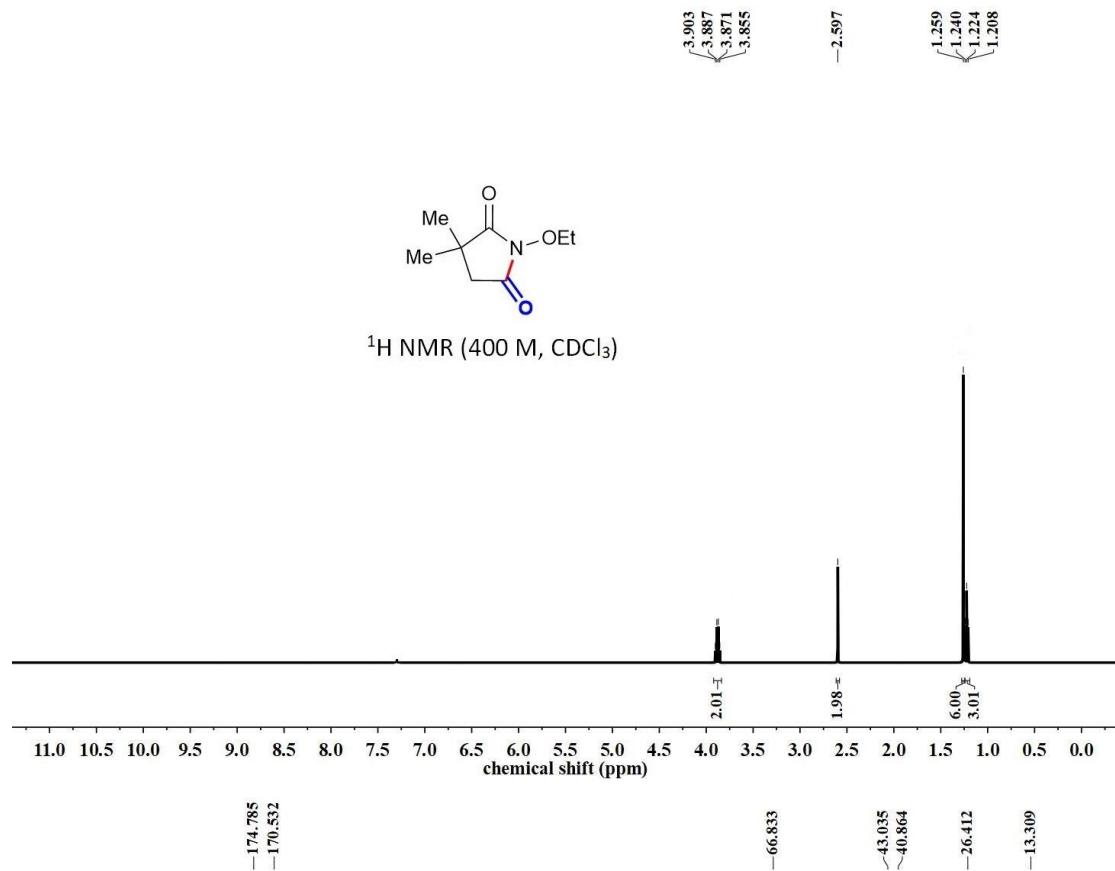
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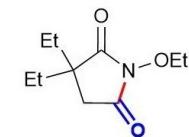
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chemical shift (ppm)

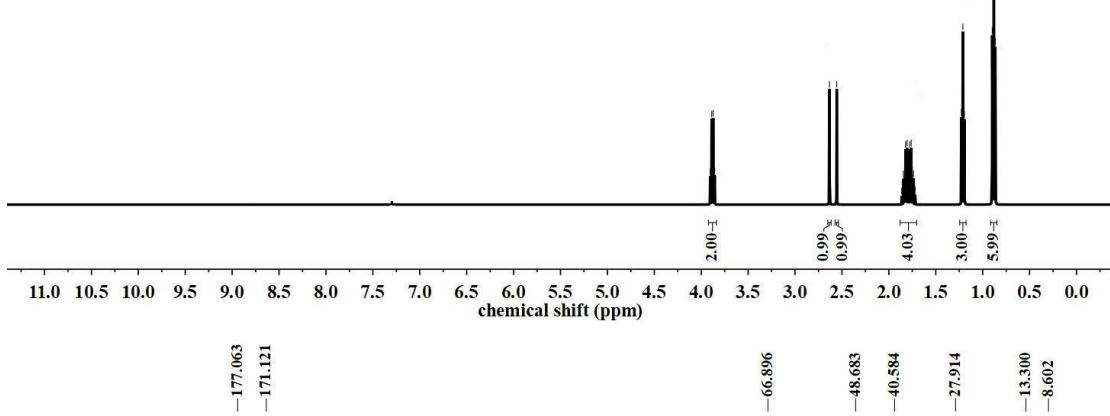




CC(C)(C)C1CC(=O)N(OCC)C(=O)C1

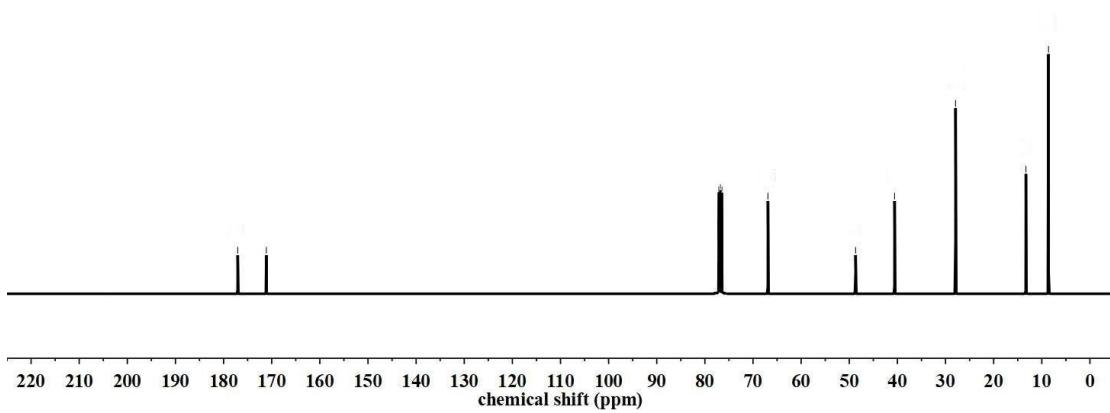


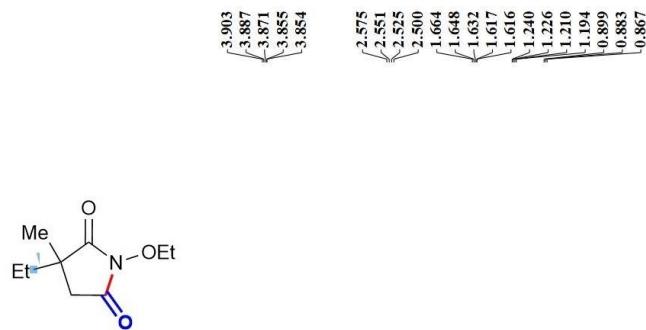
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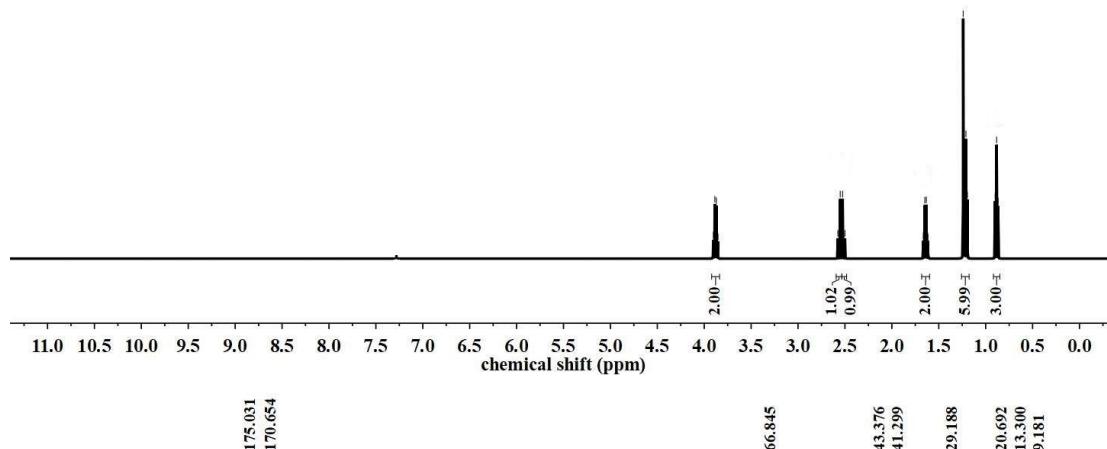
-177.063
-171.121

^{13}C NMR (100 M, CDCl_3)

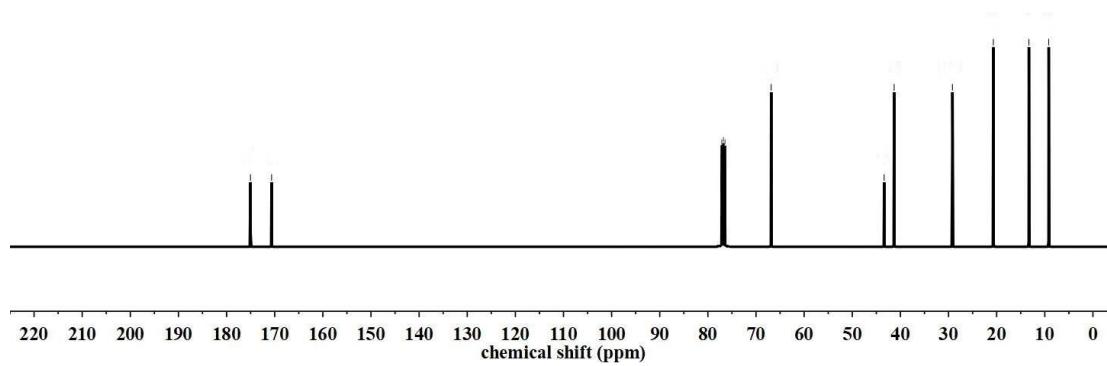


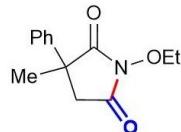


¹H NMR (400 M, CDCl₃)

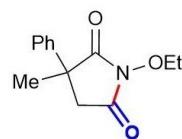
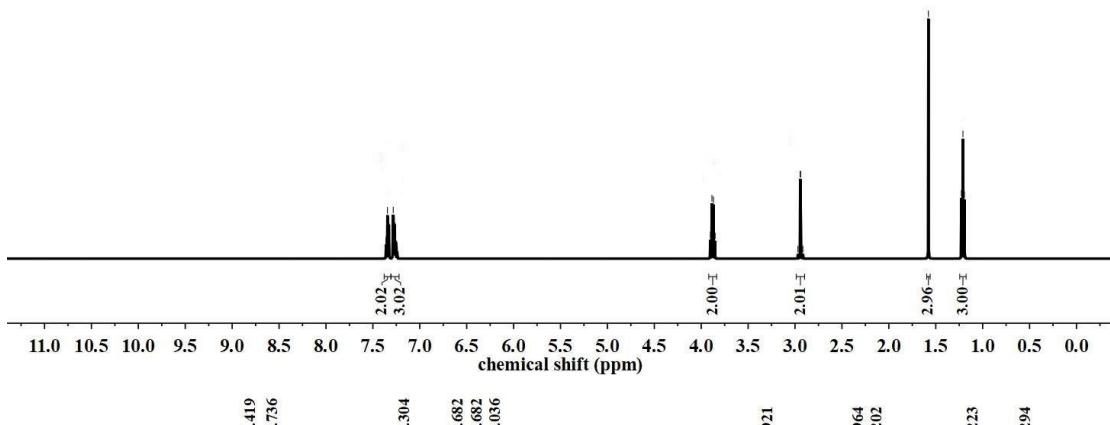


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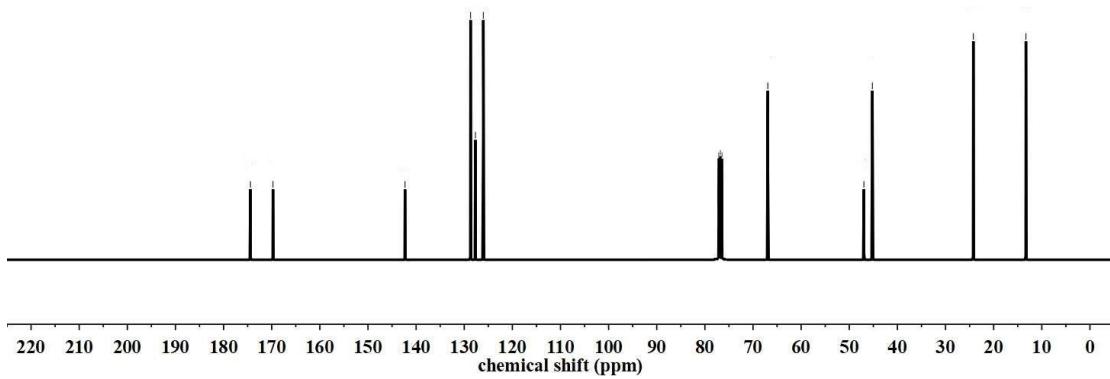


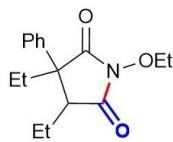
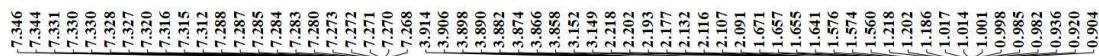


¹H NMR (400 M, CDCl₃)

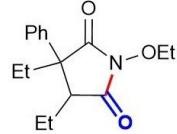
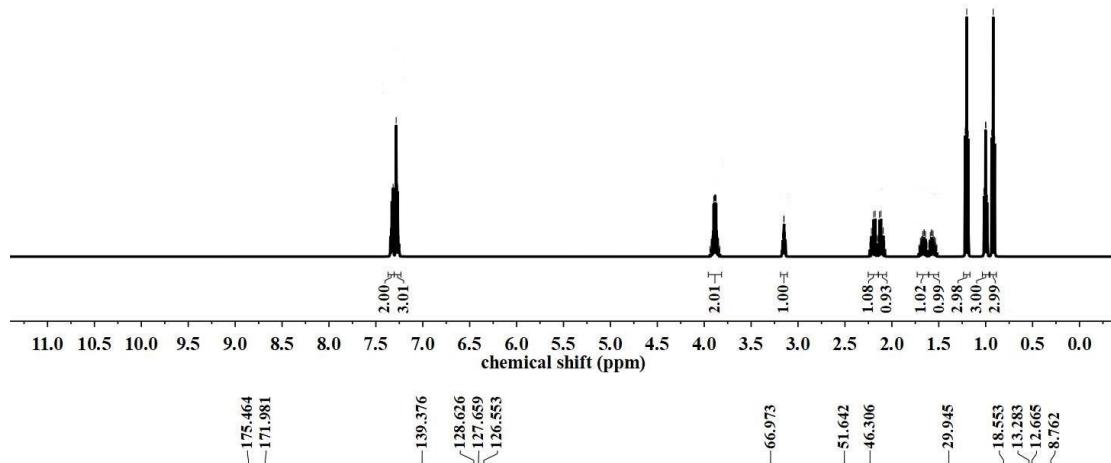


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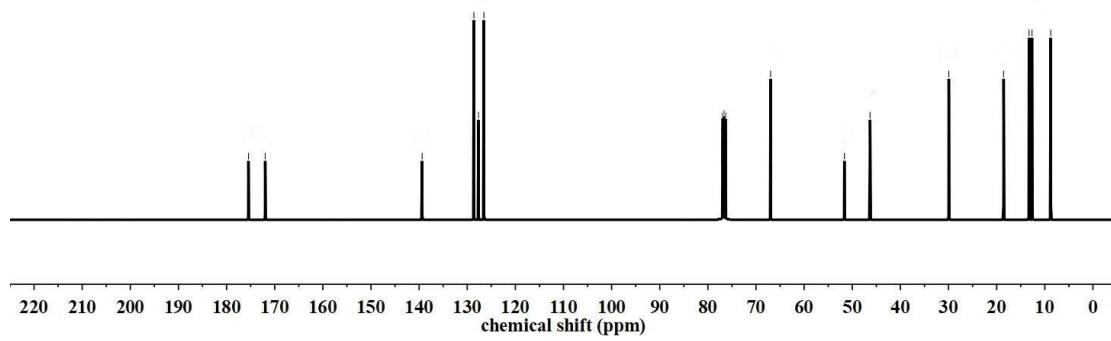


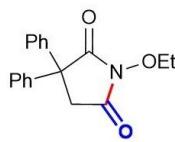


¹H NMR (400 M, CDCl₃)

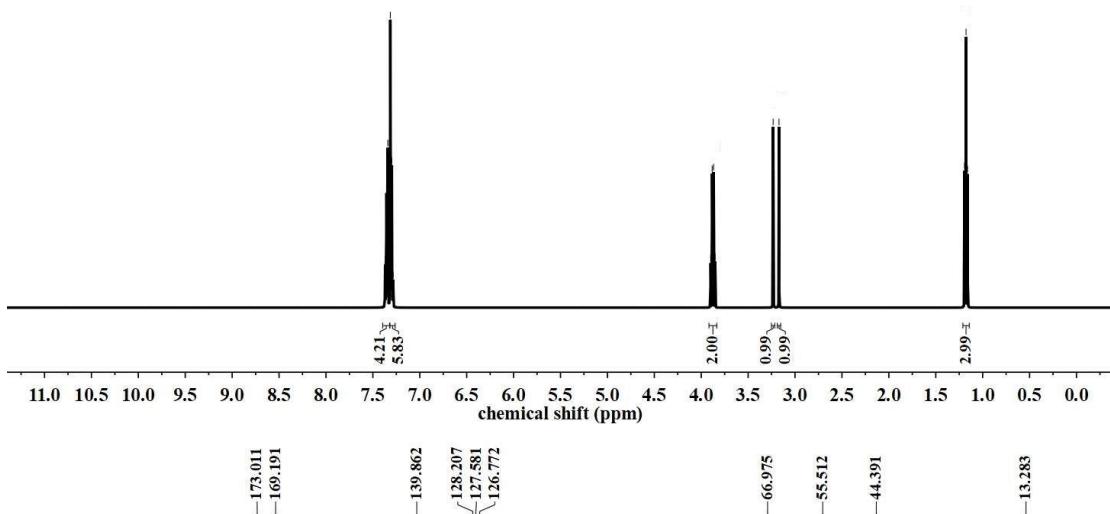


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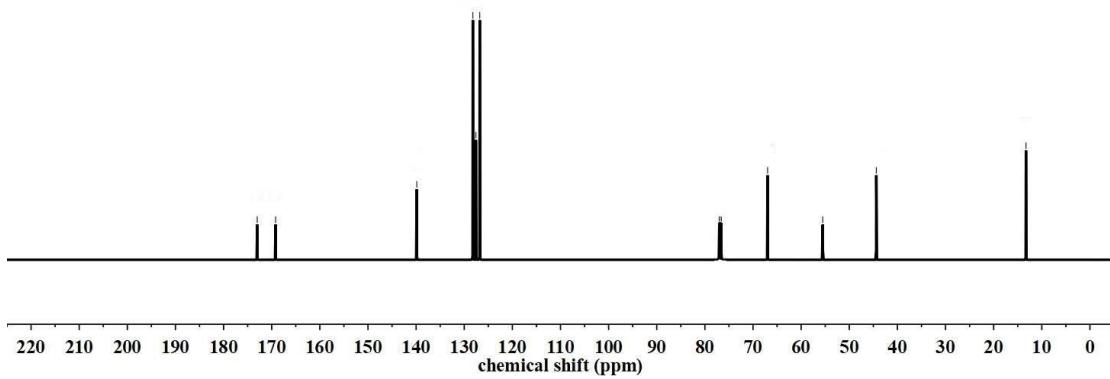


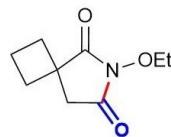
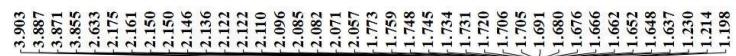


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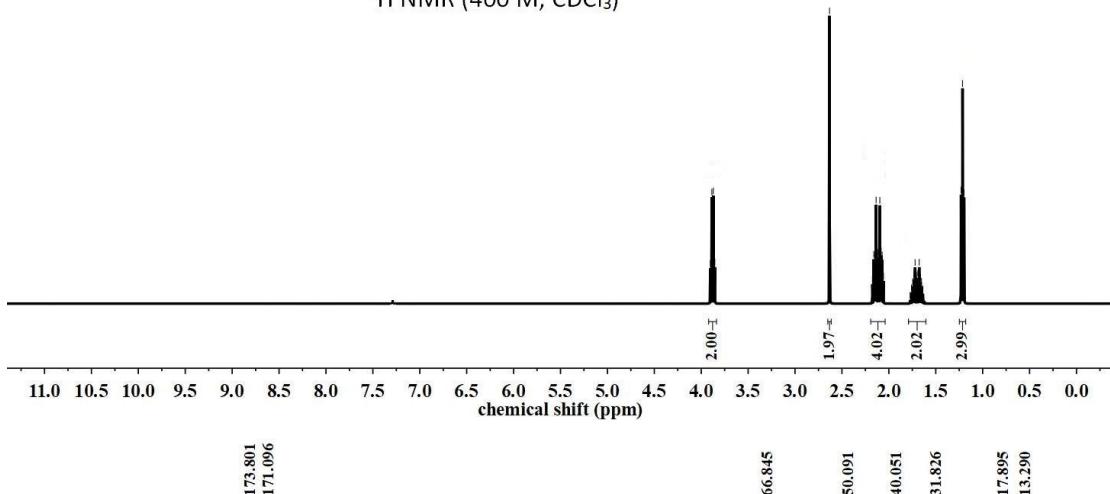


¹³C NMR (100 MHz, CDCl₃)

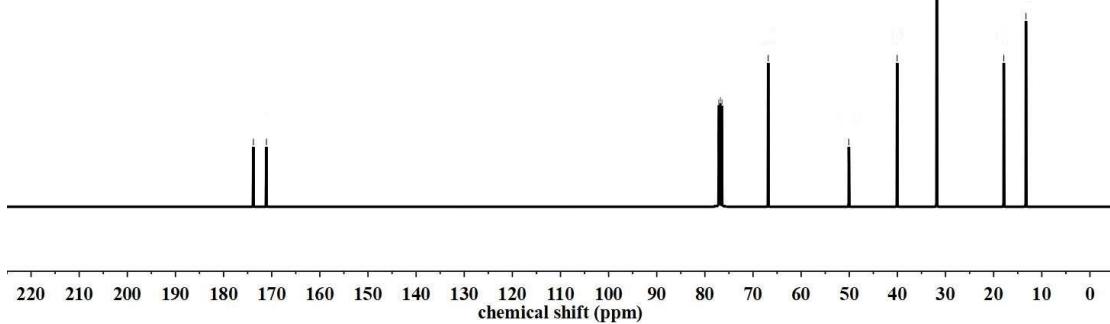


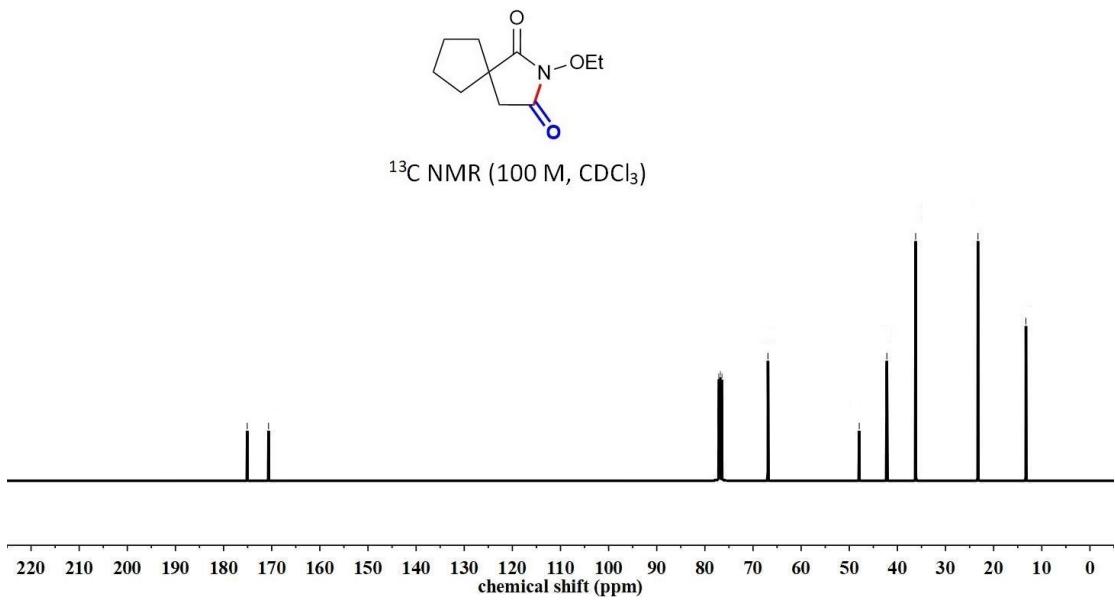
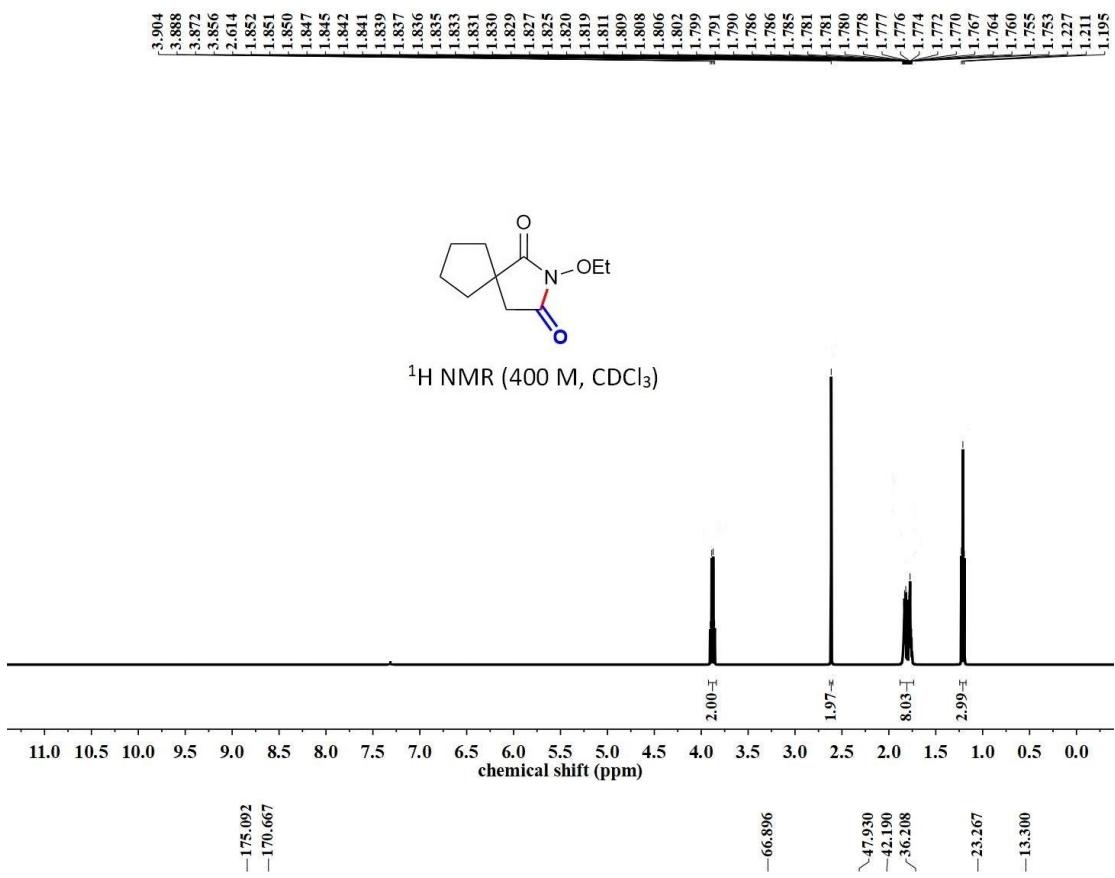


¹H NMR (400 M, CDCl₃)



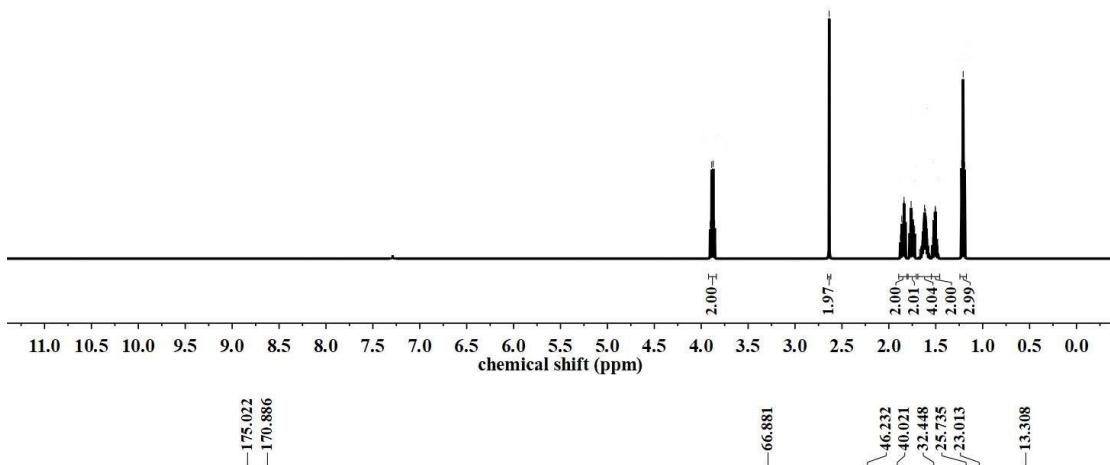
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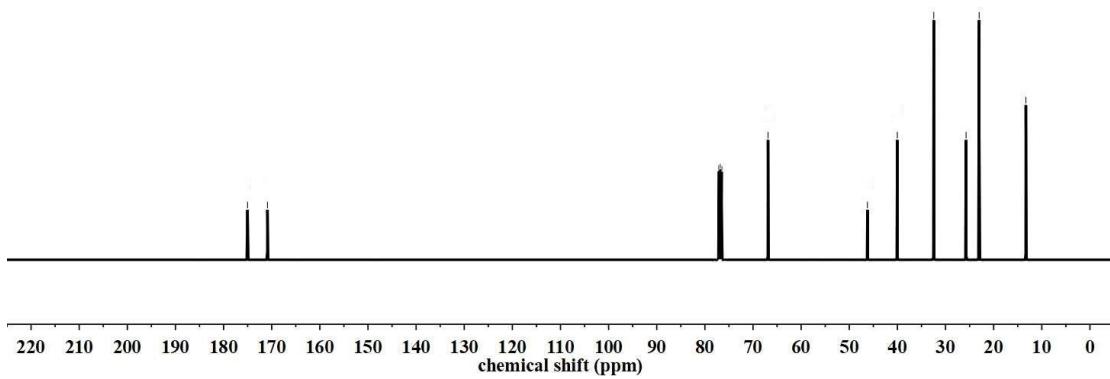


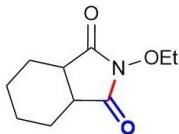


¹H NMR (400 M, CDCl₃)

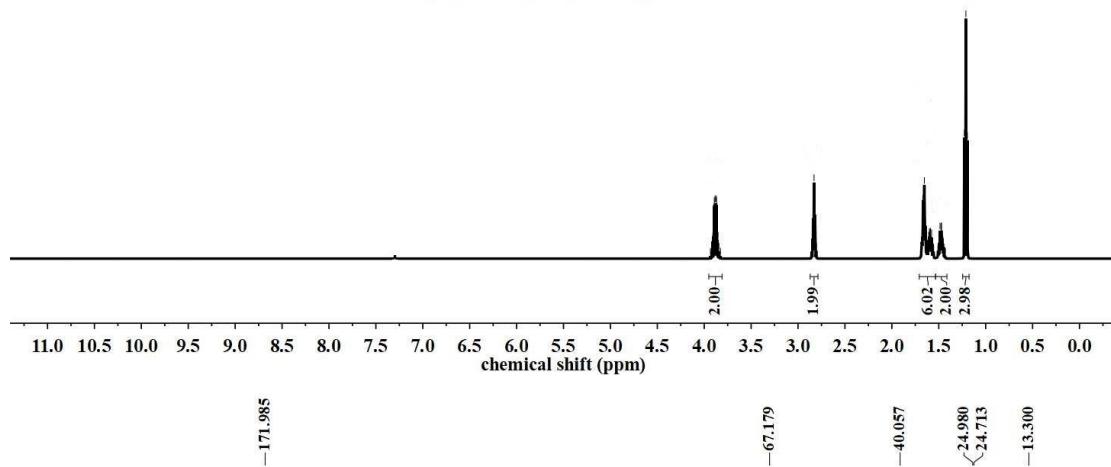


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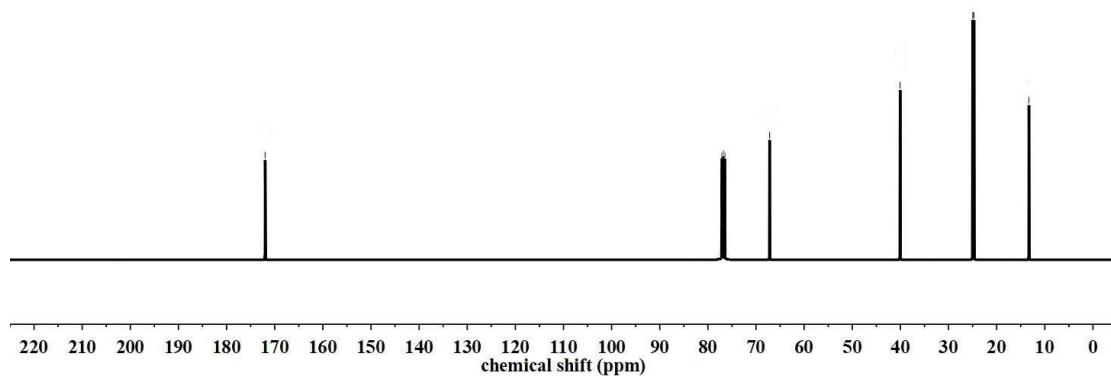


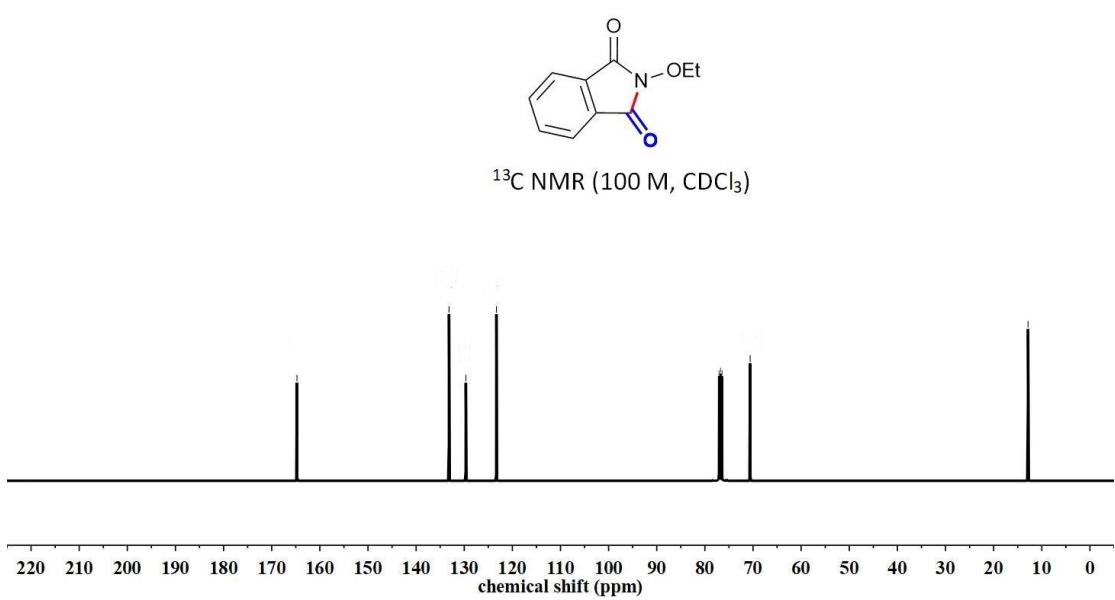
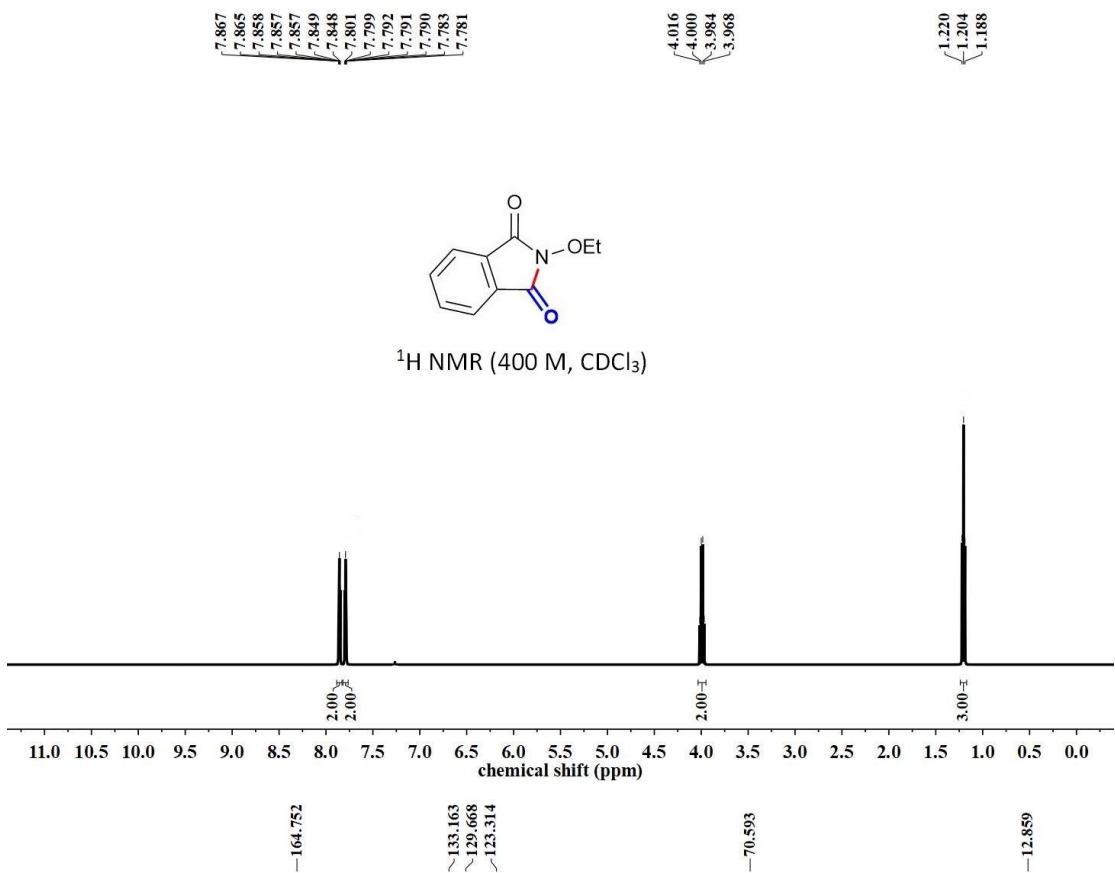


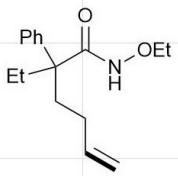
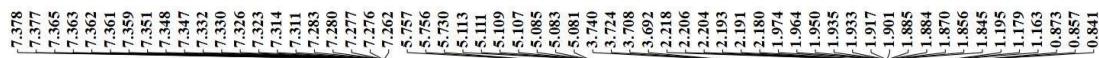
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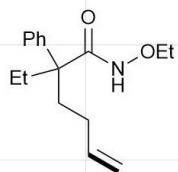
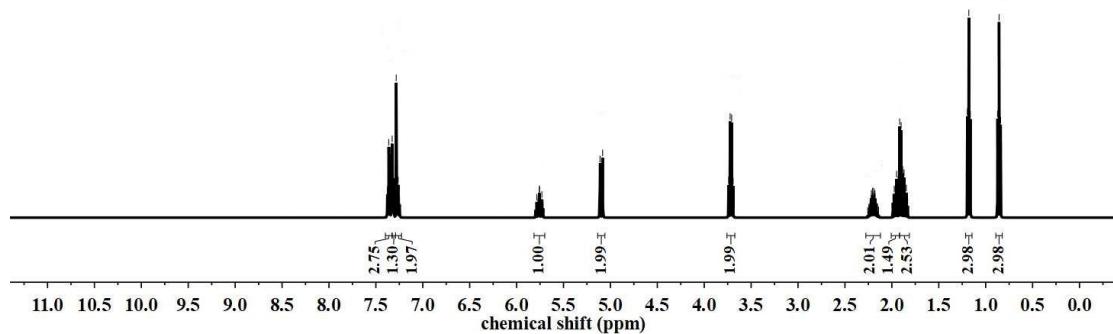
¹³C NMR (100 M, CDCl₃)



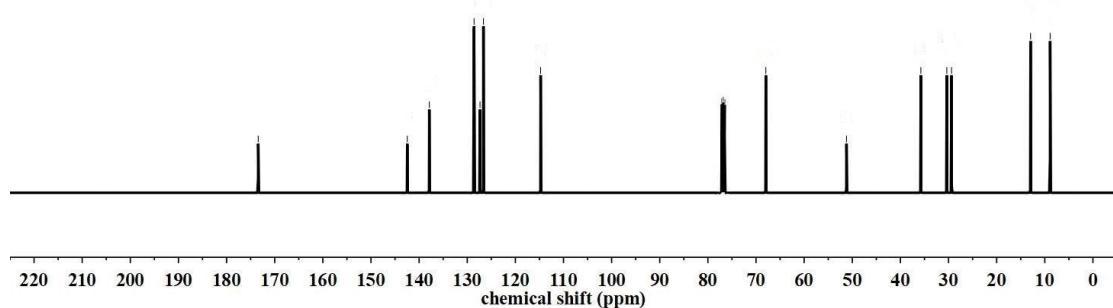




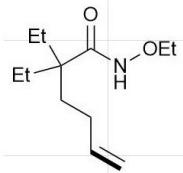
¹H NMR (400 M, CDCl₃)



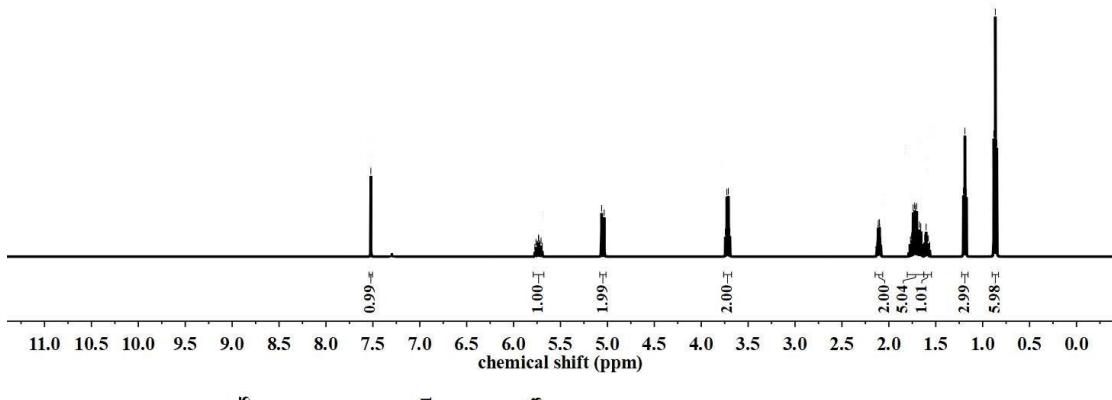
¹³C NMR (100 M, CDCl₃)



7.522
 5.761
 5.747
 5.734
 5.733
 5.722
 5.721
 5.707
 5.063
 5.061
 5.059
 5.037
 5.035
 5.032
 5.032
 3.743
 3.727
 3.711
 3.695
 2.127
 2.116
 2.114
 2.112
 2.110
 2.102
 2.100
 2.098
 2.096
 2.086
 1.770
 1.757
 1.754
 1.741
 1.738
 1.735
 1.725
 1.719
 1.709
 1.706
 1.703
 1.698
 1.690
 1.687
 1.684
 1.674
 1.670
 1.659
 1.658
 1.645
 1.617
 1.603
 1.593
 1.589
 1.578
 1.205
 1.189
 1.173
 0.881
 0.865
 0.849



¹H NMR (400 M, CDCl₃)



-175.545

-137.731

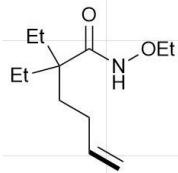
-114.743

-67.970

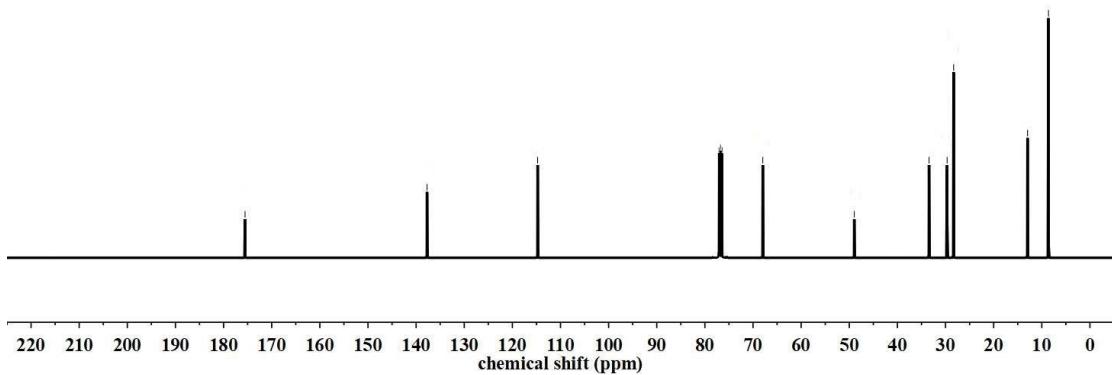
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-12.964

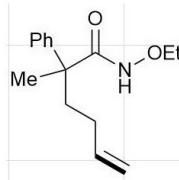
-8.609



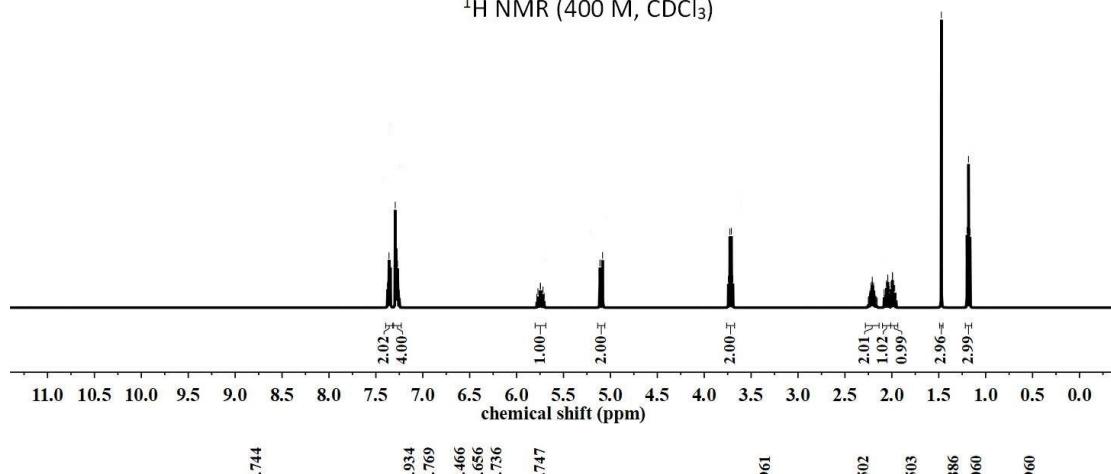
¹³C NMR (100 M, CDCl₃)



7.376
7.375
7.361
7.360
7.359
7.358
7.349
7.346
7.345
7.293
7.290
7.289
7.287
7.285
7.282
7.280
7.275
7.273
7.269
7.267
7.265
5.746
5.745
5.719
5.113
5.112
5.111
5.110
5.109
5.108
5.086
5.084
5.082
3.743
3.727
3.711
3.695
2.223
2.221
2.211
2.209
2.207
2.196
2.195
2.193
2.069
2.057
2.044
2.043
2.029
2.008
1.994
1.993
1.979
1.969
1.967
1.972
1.199
1.183
1.167



¹H NMR (400 M, CDCl₃)



-173.744

-141.934

-137.769

-128.466

-127.656

-126.736

-114.747

-67.961

-47.502

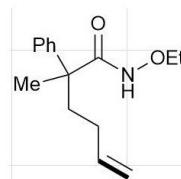
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-37.503

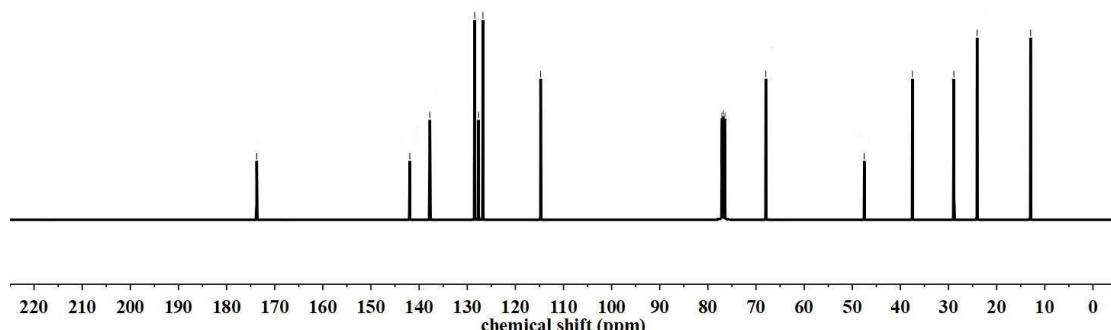
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-24.060

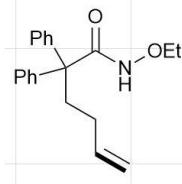
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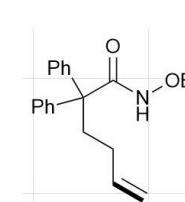
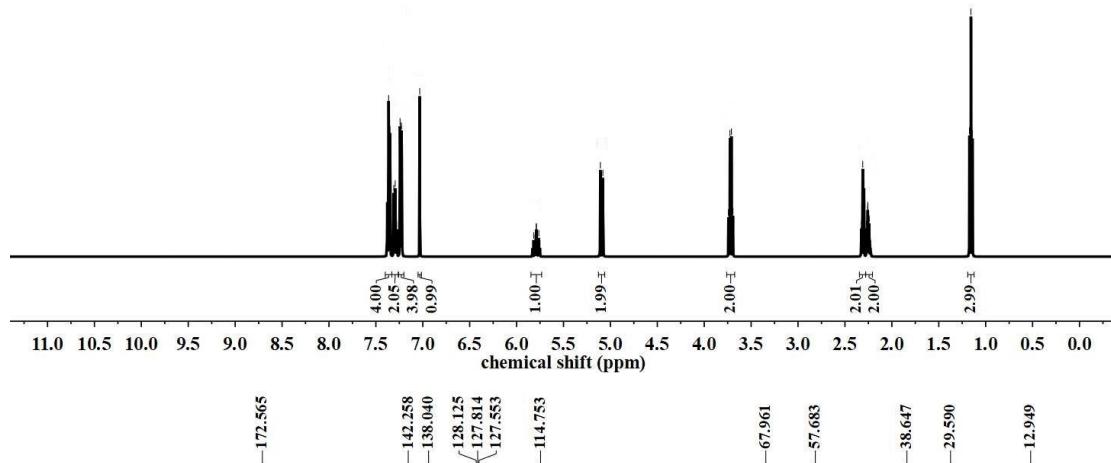
¹³C NMR (100 M, CDCl₃)



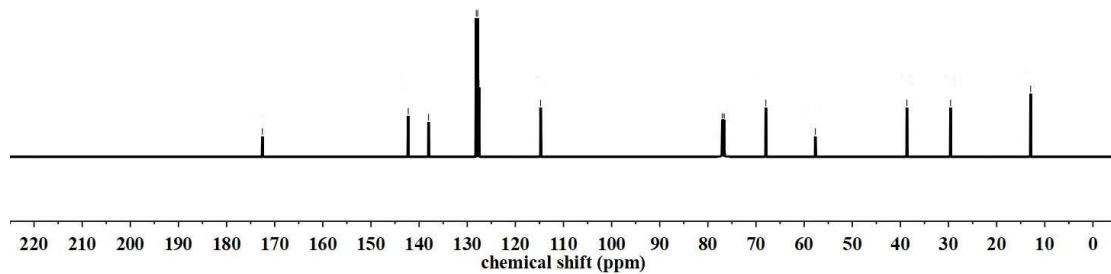
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7.379
7.378
7.376
7.368
7.367
7.365
7.364
7.362
7.361
7.360
7.351
7.349
7.348
7.312
7.309
7.306
7.296
7.294
7.293
7.279
7.244
7.242
7.239
7.236
7.229
7.227
7.225
7.224
7.032
5.791
5.109
5.107
5.105
5.082
5.080
5.078
3.742
3.726
3.710
3.694
2.324
2.314
2.312
2.310
2.299
2.297
2.269
2.267
2.258
2.257
2.256
2.255
2.255
2.244
2.242
2.242
1.173
1.157
1.141



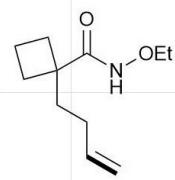
¹H NMR (400 M, CDCl₃)



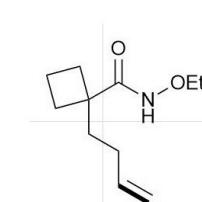
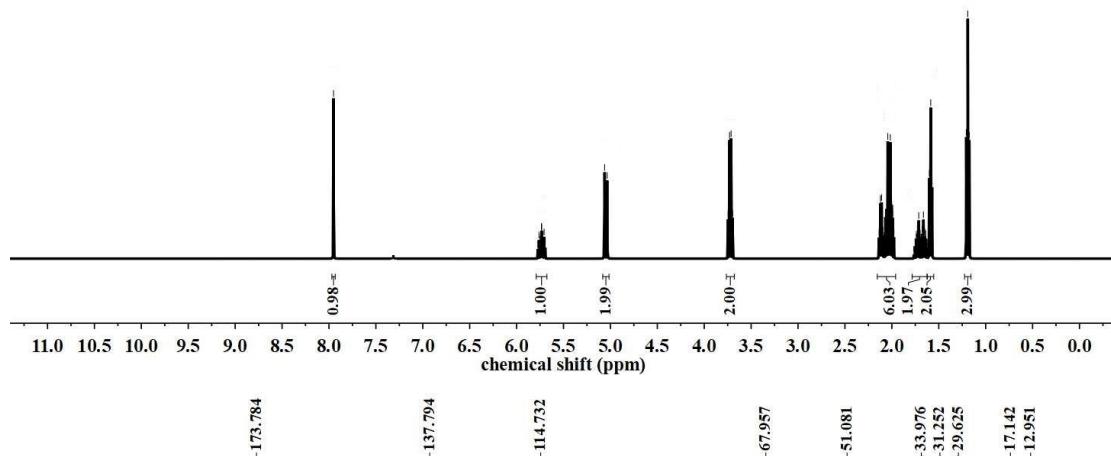
¹³C NMR (100 M, CDCl₃)



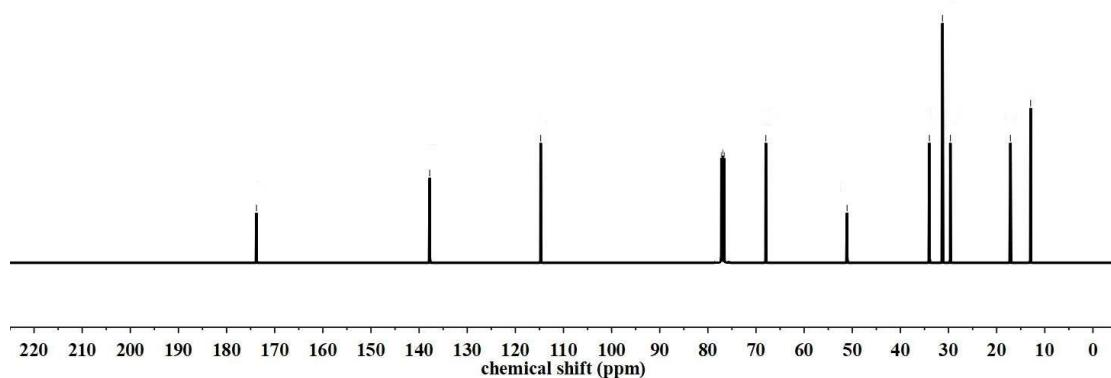
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5.761
5.747
5.734
5.733
5.722
5.721
5.707
5.064
5.062
5.060
5.037
5.035
5.033
3.745
3.729
3.713
3.697
2.136
2.125
2.124
2.122
2.120
2.111
2.109
2.107
2.105
2.095
2.082
2.068
2.058
2.057
2.054
2.043
2.031
2.030
2.029
2.029
2.016
2.006
2.003
2.002
1.992
1.978
1.738
1.727
1.726
1.713
1.699
1.678
1.663
1.649
1.638
1.600
1.586
1.572
1.207
1.191
1.175



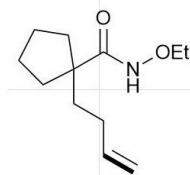
¹H NMR (400 M, CDCl₃)



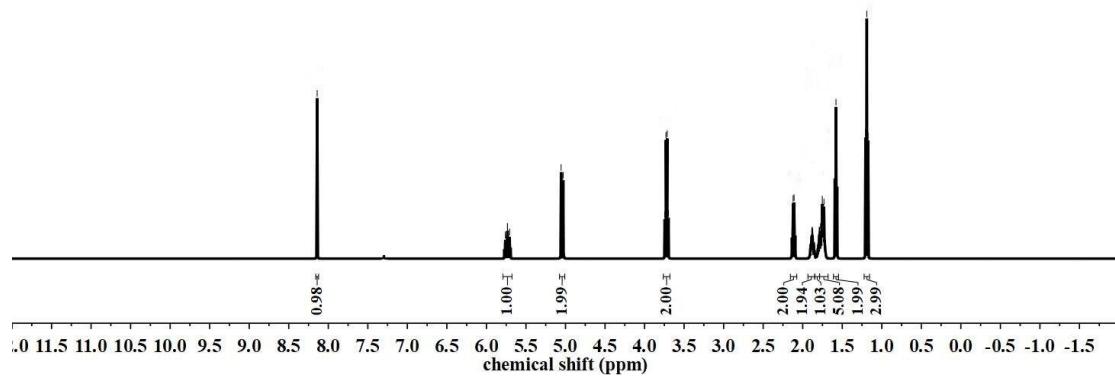
¹³C NMR (100 M, CDCl₃)



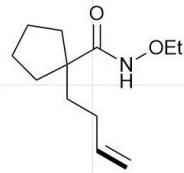
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5.734
5.733
5.707
5.058
5.056
5.054
5.031
5.029
5.027
3.745
3.729
3.713
3.697
2.123
2.121
2.119
2.111
2.109
2.107
2.095
1.885
1.883
1.881
1.880
1.876
1.790
1.789
1.788
1.775
1.766
1.764
1.763
1.762
1.760
1.759
1.757
1.756
1.755
1.752
1.751
1.750
1.748
1.746
1.742
1.741
1.738
1.737
1.736
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1.731
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1.593
1.579
1.565
1.565
1.205
1.189
1.173



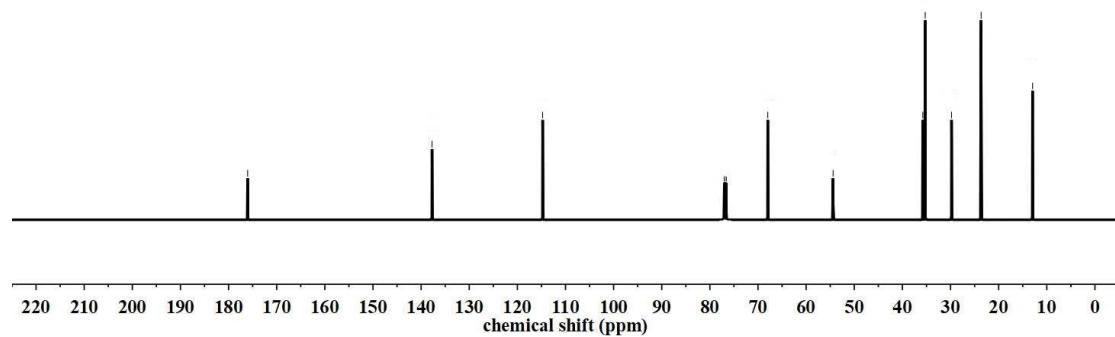
¹H NMR (400 M, CDCl₃)



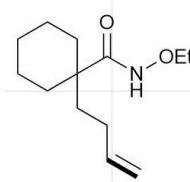
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-137.731
-114.743
-67.970
-54.400
35.771
35.265
29.793
-23.645
-12.964



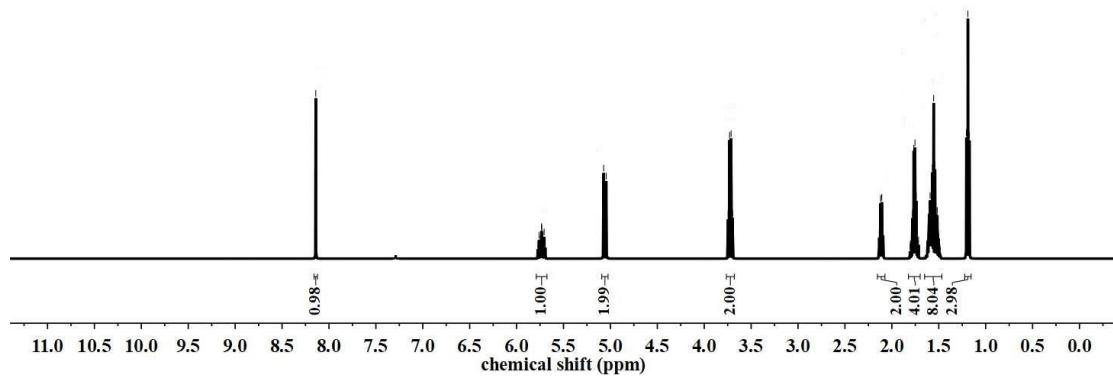
¹³C NMR (100 M, CDCl₃)



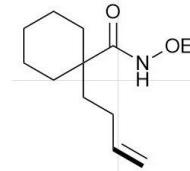
8.141
5.734
5.733
5.706
5.074
5.072
5.070
5.047
5.045
5.043
3.745
3.729
3.713
3.697
2.122
2.120
2.118
2.109
2.107
2.105
2.093
1.781
1.767
1.766
1.754
1.754
1.752
1.740
1.608
1.607
1.605
1.603
1.601
1.594
1.592
1.589
1.587
1.586
1.578
1.576
1.575
1.574
1.572
1.569
1.565
1.554
1.540
1.531
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1.528
1.527
1.518
1.516
1.515
1.514
1.513
1.205
1.189
1.173



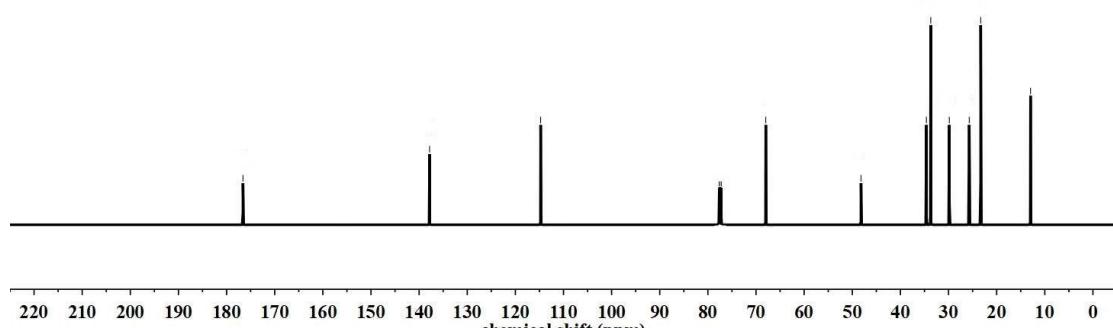
¹H NMR (400 M, CDCl₃)



-176.598, -137.819, -114.743, -67.970, -48.187, 34.600, 33.693, ~29.835, ~25.703, ~23.315, -12.964

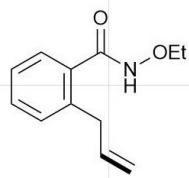


¹³C NMR (100 M, CDCl₃)

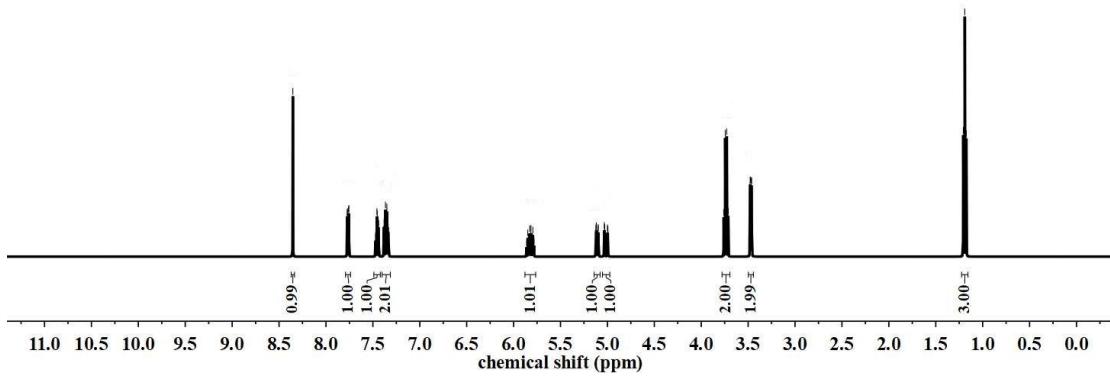


220, 210, 200, 190, 180, 170, 160, 150, 140, 130, 120, 110, 100, 90, 80, 70, 60, 50, 40, 30, 20, 10, 0

8.353
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7.771
7.759
7.756
7.472
7.469
7.457
7.454
7.442
7.439
7.384
7.381
7.369
7.366
7.354
7.352
7.351
7.349
7.347
7.337
7.336
7.335
7.334
5.849
5.828
5.827
5.816
5.815
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5.120
5.118
5.116
5.102
5.100
5.098
5.035
5.033
5.030
5.028
4.999
4.997
4.995
3.760
3.744
3.728
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3.480
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3.467
3.465
3.463
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1.190
1.174

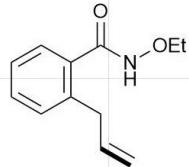


¹H NMR (400 M, CDCl₃)

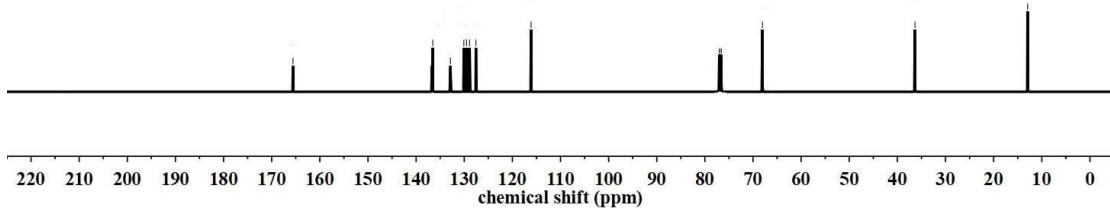


-165.580 -165.580
 -116.107 -116.107
 127.572 127.572
 129.513 129.513
 130.080 130.080
 132.859 132.859
 136.695 136.695
 128.894 128.894

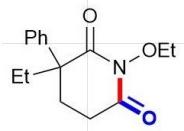
-68.047 -68.047
 -36.356 -36.356
 -12.951 -12.951



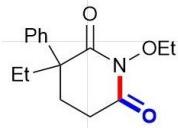
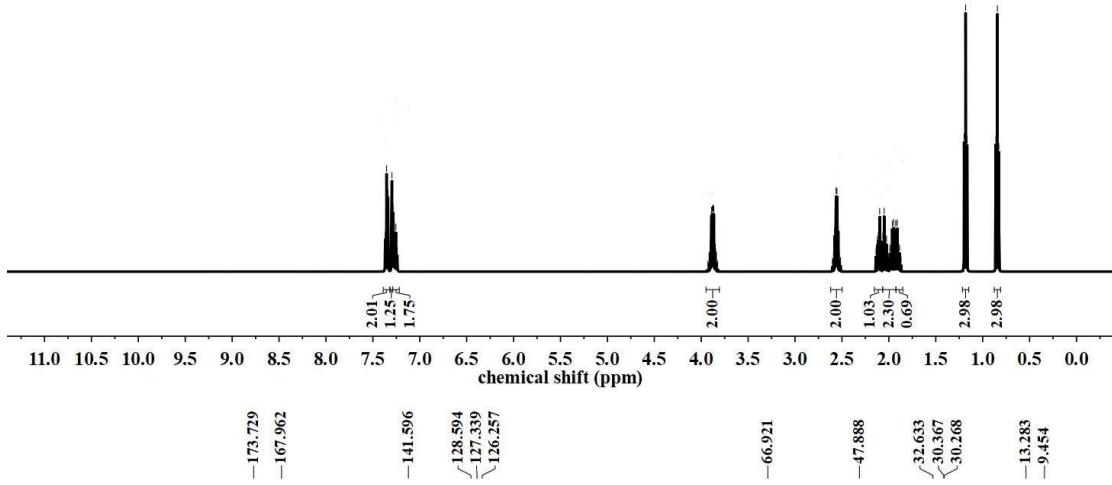
¹³C NMR (100 M, CDCl₃)



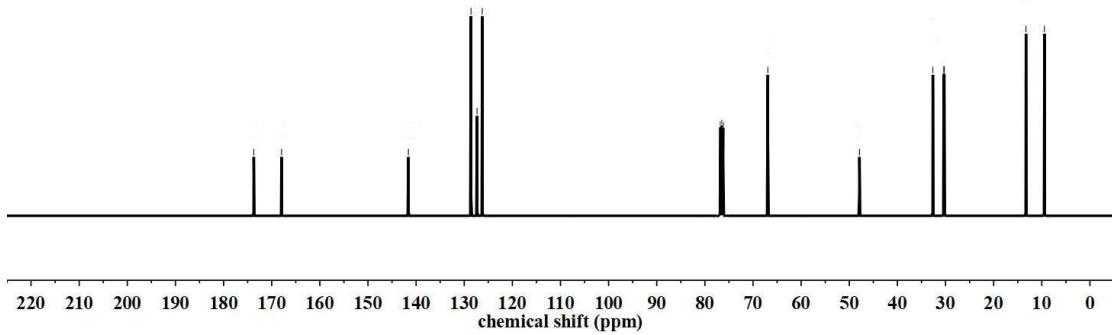
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 7.342
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 7.338
 7.302
 7.300
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 7.293
 7.270
 7.287
 7.285
 7.282
 7.281
 7.273
 7.270
 7.269
 7.257
 7.255
 3.906
 3.897
 3.890
 3.881
 3.874
 3.865
 3.857
 3.849
 2.577
 2.568
 2.562
 2.554
 2.548
 2.540
 2.112
 2.112
 2.098
 2.084
 2.064
 2.050
 2.035
 2.025
 1.988
 1.972
 1.964
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 0.859
 0.843
 0.827



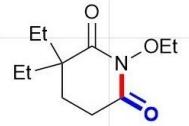
¹H NMR (400 M, CDCl₃)



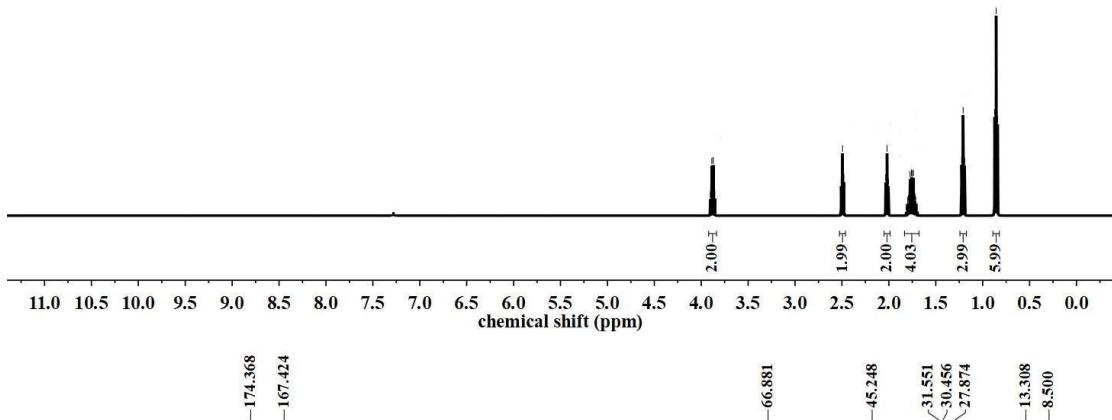
¹³C NMR (100 M, CDCl₃)



3.903
3.887
3.871
3.855
2.508
2.494
2.480
2.034
2.019
2.005
1.815
1.799
1.779
1.773
1.774
1.769
1.767
1.758
1.753
1.745
1.742
1.738
1.731
1.729
1.722
1.713
1.697
1.624
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1.308
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0.872
0.856
0.840

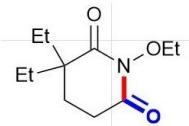


¹H NMR (400 M, CDCl₃)

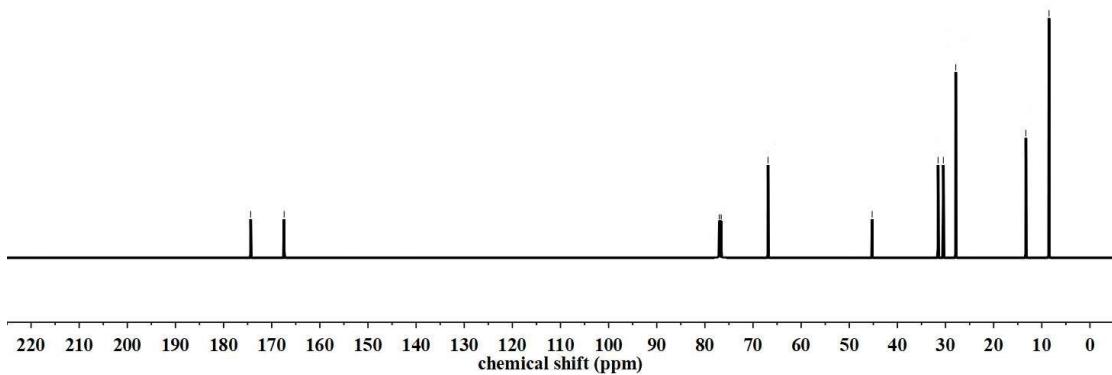


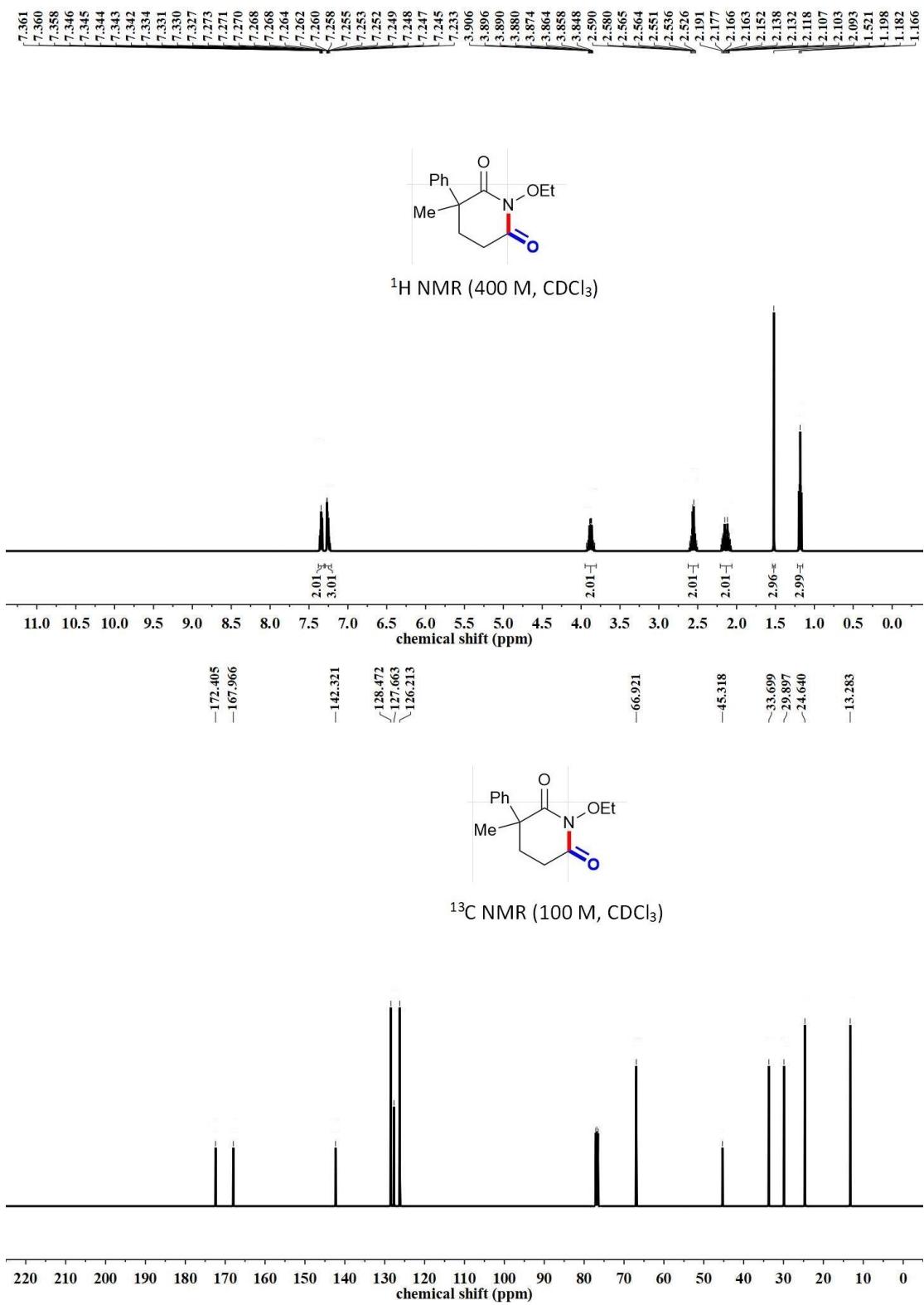
-174.368
-167.424

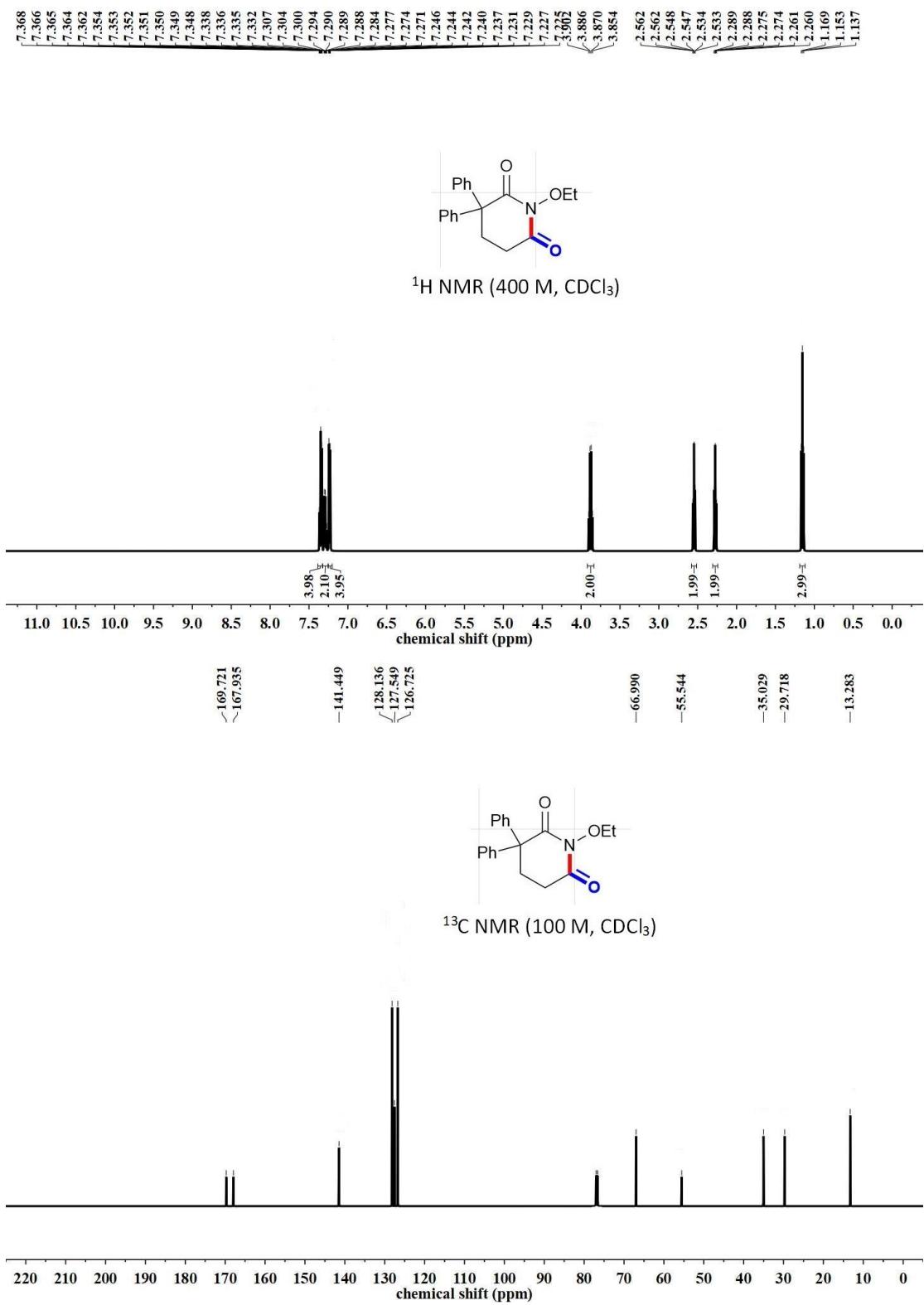
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-45.448
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30.456
27.874
-13.308
-8.500



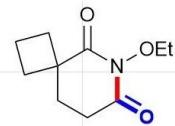
¹³C NMR (100 M, CDCl₃)



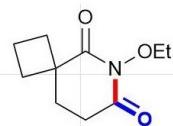
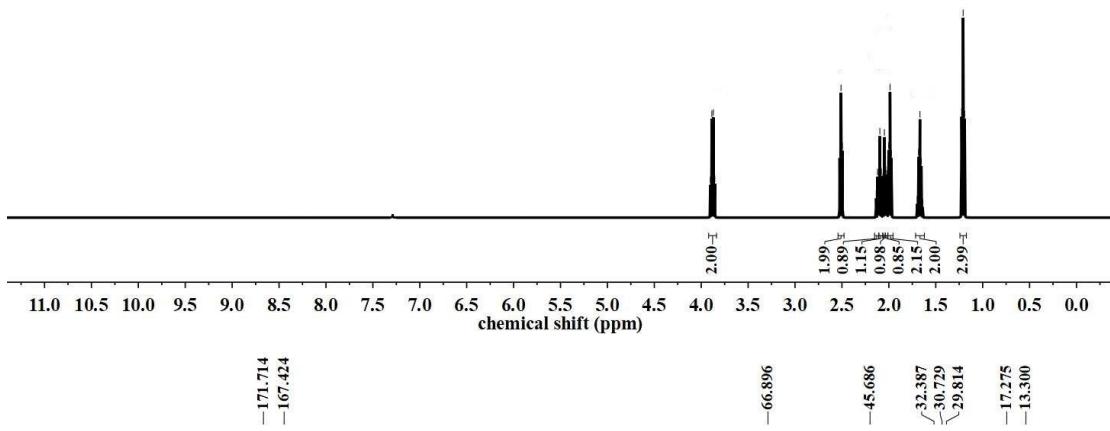




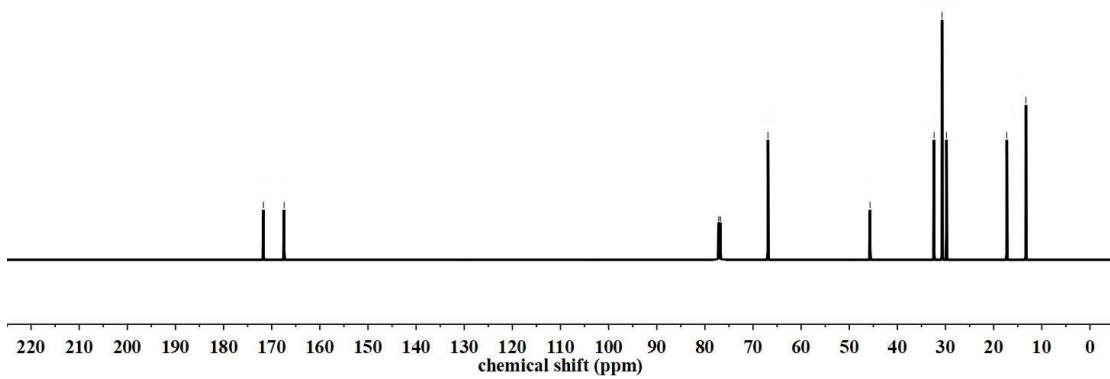
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3.887
3.871
3.855
2.524
2.510
2.495
2.135
2.121
2.110
2.110
2.106
2.096
2.082
2.081
2.062
2.061
2.047
2.037
2.033
2.023
2.008
2.002
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1.208
1.192



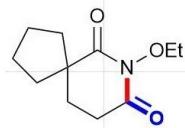
¹H NMR (400 M, CDCl₃)



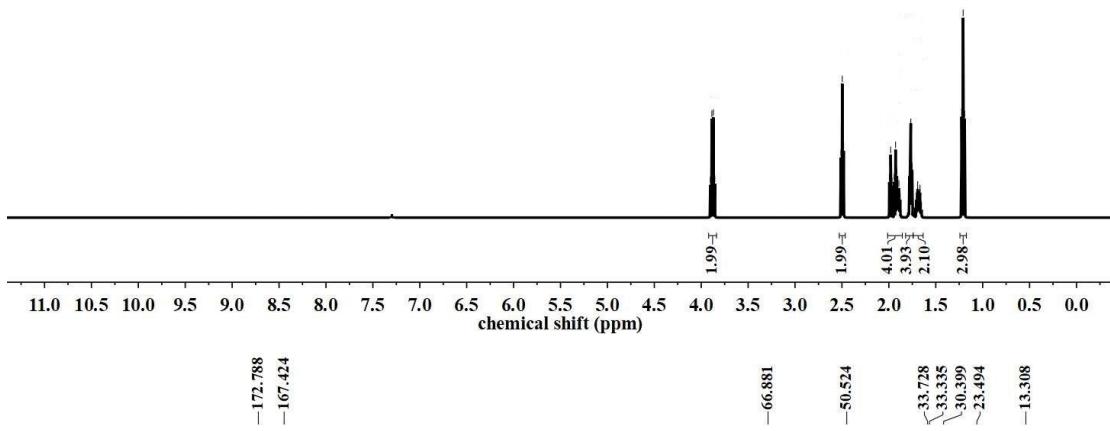
¹³C NMR (100 M, CDCl₃)



3.903
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 1.943
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 1.903
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 1.192

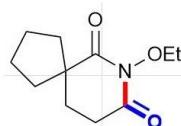


¹H NMR (400 M, CDCl₃)

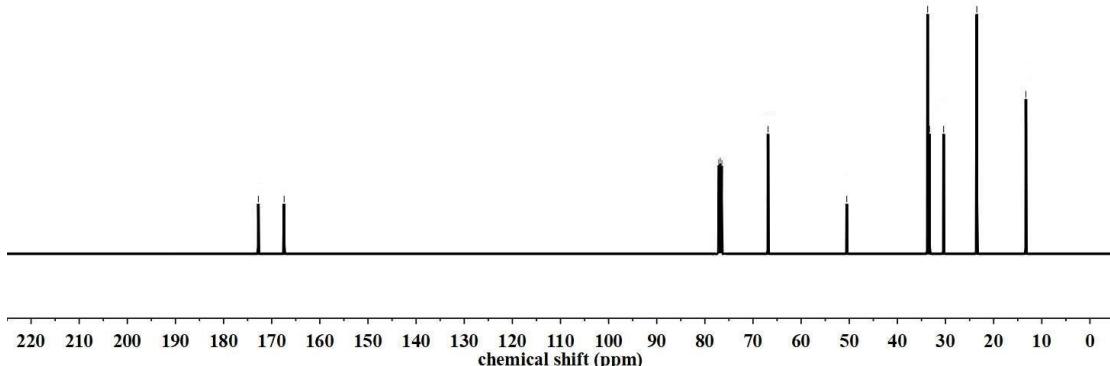


-172.788
-167.424

-66.881
-50.524
-13.308



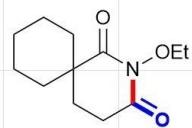
¹³C NMR (100 M, CDCl₃)



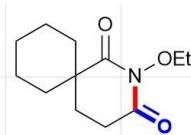
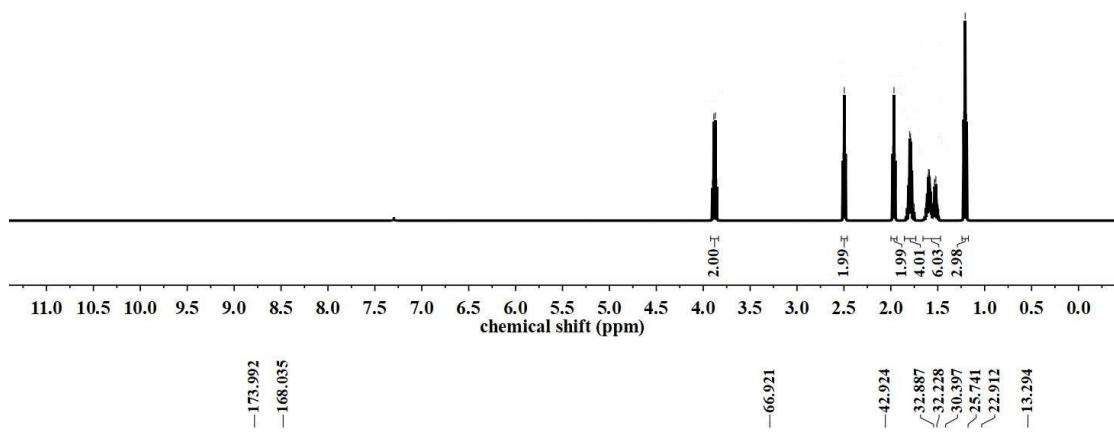
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

chemical shift (ppm)

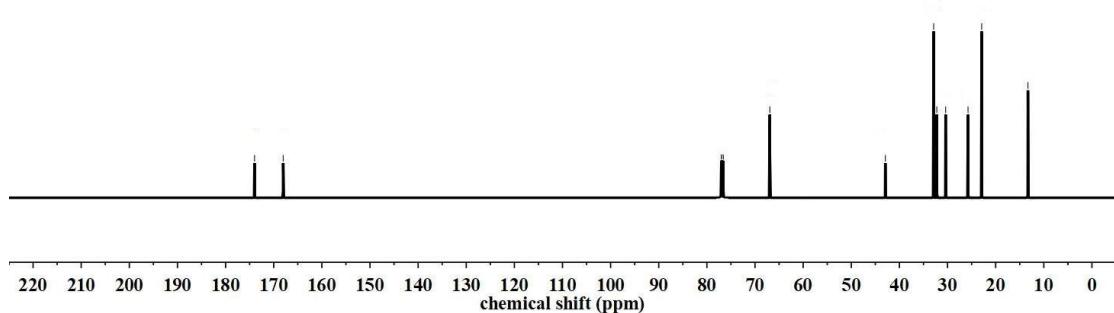
3.903
3.887
3.871
3.855
2.511
2.496
2.482
1.981
1.967
1.952
1.823
1.813
1.809
1.803
1.799
1.790
1.784
1.778
1.776
1.765
1.618
1.616
1.614
1.612
1.605
1.604
1.600
1.599
1.598
1.593
1.591
1.590
1.589
1.586
1.585
1.579
1.577
1.575
1.572
1.565
1.564
1.547
1.546
1.543
1.535
1.534
1.533
1.532
1.520
1.518
1.508
1.504
1.224
1.208
1.192

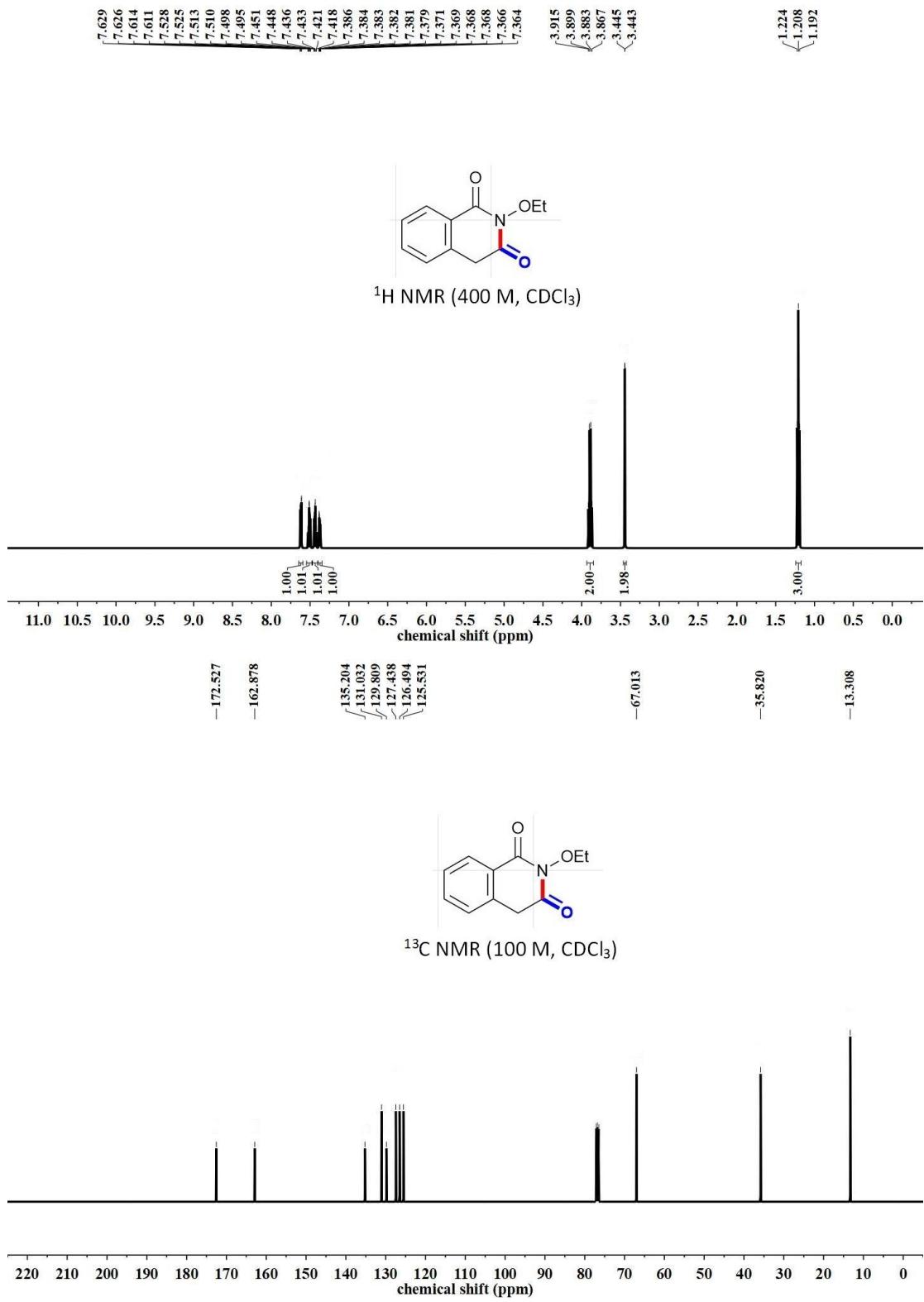


¹H NMR (400 M, CDCl₃)

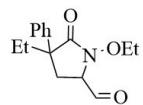


¹³C NMR (100 M, CDCl₃)

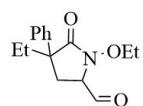
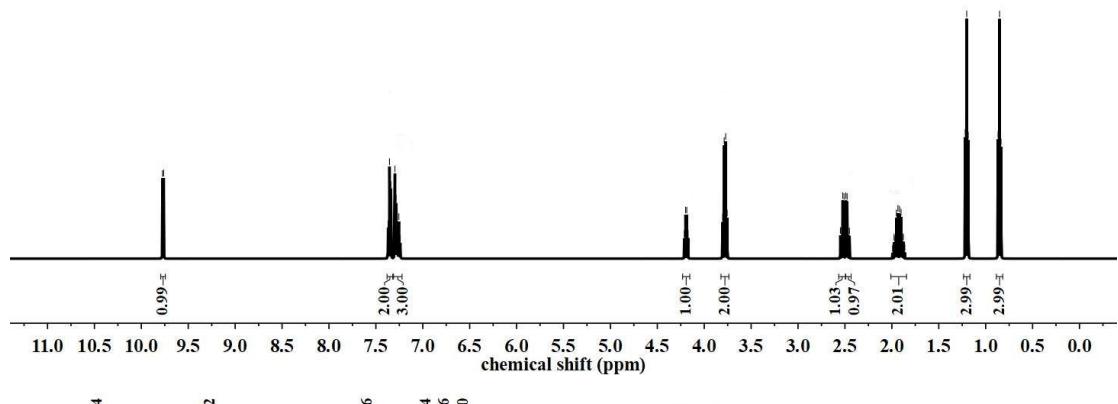




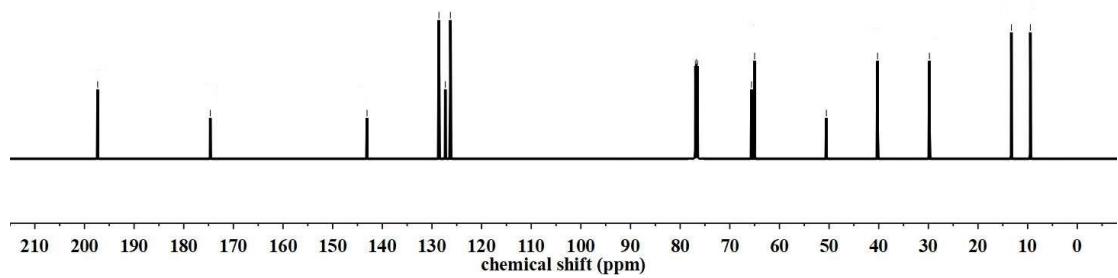
9.775
9.762
7.370
7.369
7.357
7.355
7.353
7.352
7.351
7.343
7.340
7.339
7.302
7.300
7.297
7.294
7.287
7.285
7.282
7.281
7.273
7.270
7.269
7.257
7.255
4.212
4.200
4.198
4.186
4.184
4.172
3.803
3.787
3.771
3.755
2.548
2.534
2.524
2.510
2.491
2.477
2.466
2.452
1.976
1.960
1.951
1.935
1.934
1.919
1.918
1.902
1.893
1.877
1.218
1.202
1.186
0.868
0.852
0.836



¹H NMR (CDCl₃, 400 MHz)



¹³C NMR (CDCl₃, 101 MHz)



4. ESI-MS measurement: instrument parameters and results

Acquisition Mass Control	
Detection Mode	Broad band
TD (Acquisition)	1 M
Low Mass	57.75 m/z
High Mass	2000.00 m/z
Estimated R.P.	38000
Transient Length	0.2097 sec
Data transfer mode	standard

API Source		Source Gas Tune		Source Gas Control	
Source Type	ESI	Dry Gas	4.0 L/min	Drying Gas Flow	Yes
Capillary -	2600 V	Dry Temp	100 °C	Drying Gas Heater	Yes
End Plate Offset	-500 V	Nebulizer	1.0 bar	Nebulizer Gas Flow	Yes
Corona Needle	0.0 nA	Vaporizar Temp	10.0 °C	Nebulizer Gas Heater	No

Figure S1 ESI-MS spectra of mechanism research

