Supporting Information

Iron-Mediated Synthesis of Isoxazoles from Alkynes: Using Iron(Ⅲ) Nitrate as Nitration and Cyclization Reagent

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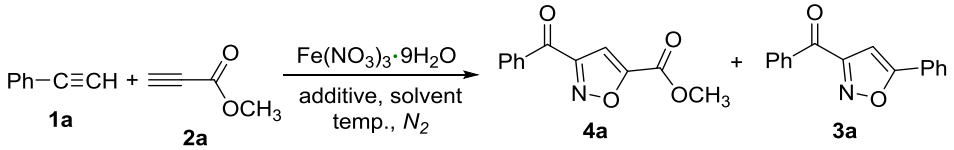
Table of contents

Table S1. Optimization of conditions on $Fe(NO_3)_3$-mediated synthesis of methyl-3-benzoylisoxazole-5-carboxylate (**4a**) from phenylacetylene (**1a**) and methyl propiolate (**2a**)[a]



Entry	Nitrogent reagent (mmol)	Solvent	Additive (mmol)	Temp (^oC)	Yield of **4a** (%)[b]
1	$Pd(OAc)_2/NaNO_3$ (0.06/1.2)	THF	KI(0.3)	60	Trace
2	$AuCl_3/NaNO_3$ (0.06/1.2)	THF	KI(0.3)	60	Trace
3	$Zn(NO_3)_2$ (1.2)	THF	KI(0.3)	60	Trace
4	$Mn(NO_3)_2$ (1.2)	THF	KI(0.3)	60	Trace
5	$Ni(NO_3)_2{\cdot}6H_2O$ (1.2)	THF	KI(0.3)	60	Trace
6	$Cu(NO_3)_2{\cdot}3H_2O$ (1.2)	THF	KI(0.3)	60	36
7	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	THF	KI (0.3)	60	43
8	--	THF	KI (0.3)	60	Trace
9	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	PhCN	KI (0.3)	70	57
10	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	EtOAc	KI (0.3)	70	58
11	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	MeCN	KI (0.3)	70	50
12	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	DMF	KI (0.3)	70	Trace
13	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	DMSO	KI (0.3)	70	Trace
14	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	CH_3OH	KI (0.3)	60	Trace
15	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	Dioxane	KI (0.3)	70	47
16	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	EtOAc/PhCN(1/1)	KI (0.3)	70	67
17	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	EtOAc/PhCN (1/1)	KI (0.06)	70	50
18	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	EtOAc/PhCN (1/1)	KI (0.15)	70	54
19	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	EtOAc/PhCN (1/1)	KI (0.45)	70	69
20	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	EtOAc/PhCN (1/1)	--	70	49
21	$Fe(NO_3)_3{\cdot}9H_2O$ (1.2)	EtOAc/PhCN (1/1)	L-Proline (0.3)	70	47

22	$Fe(NO_3)_3 \cdot 9H_2O$ (1.2)	EtOAc/PhCN (1/1)	Ph_3P (0.3)	70	47
23	$Fe(NO_3)_3 \cdot 9H_2O$ (1.2)	EtOAc/PhCN (1/1)	K_3PO_4 (0.3)	70	51
24	$Fe(NO_3)_3 \cdot 9H_2O$ (1.2)	EtOAc/PhCN (1/1)	*t*-BuCN (0.3)	70	58
25	$Fe(NO_3)_3 \cdot 9H_2O$ (1.2)	EtOAc/PhCN (1/1)	*t*-BuCN/I_2 (0.6/0.3)	70	61
26	**$Fe(NO_3)_3 \cdot 9H_2O$ (1.2)**	**EtOAc/PhCN** (1/1)	***t*-BuCN/KI (0.6/0.3)**	**70**	**76**
27	$Fe(NO_3)_3 \cdot 9H_2O$ (1.2)	EtOAc/PhCN (1/1)	*t*-BuCN/KI (0.6/0.15)	70	62
28[b]	$Fe(NO_3)_3 \cdot 9H_2O$ (1.2)	EtOAc/PhCN (1/1)	*t*-BuCN/KI (0.6/0.3)	70	60
29[c]	$Cu(NO_3)_2 \cdot 3H_2O$ (0.6)	PhCN	--	60	61
30	$Fe(NO_3)_3 \cdot 9H_2O$ (0.9)	EtOAc/PhCN (1/1)	*t*-BuCN/KI (0.6/0.3)	70	50
31	$Fe(NO_3)_3 \cdot 9H_2O$ (0.3)	EtOAc/PhCN (1/1)	*t*-BuCN/KI (0.6/0.3)	70	40

[a] Reaction condition: **1a** (0.3 mmol), **2a** (0.6 mmol), $Fe(NO_3)_3 \cdot 9H_2O$ (1.2 mmol), additive, solvent (1 mL), reaction time (12 h) under nitrogen atmosphere. [b] In air. [c] **1a** (0.45 mmol), **2a** (0.3 mmol), $Cu(NO_3)_3 \cdot 3H_2O$ (0.6 mmol), PhCN (1.5 mL), reaction time (2.5 h) at 60 ^oC under nitrogen atmosphere [note: (1) condition **d** was based on the copper-catalyzed conditions in the reference (*Org. Chem. Front.* **2017**, *4*, 445), but the substrate **1a** is added only once instead of dropwise addition by using a syringe pumping for 2.5 h; (2) 29% of byproduct **3a** was obtained under condition **d**].

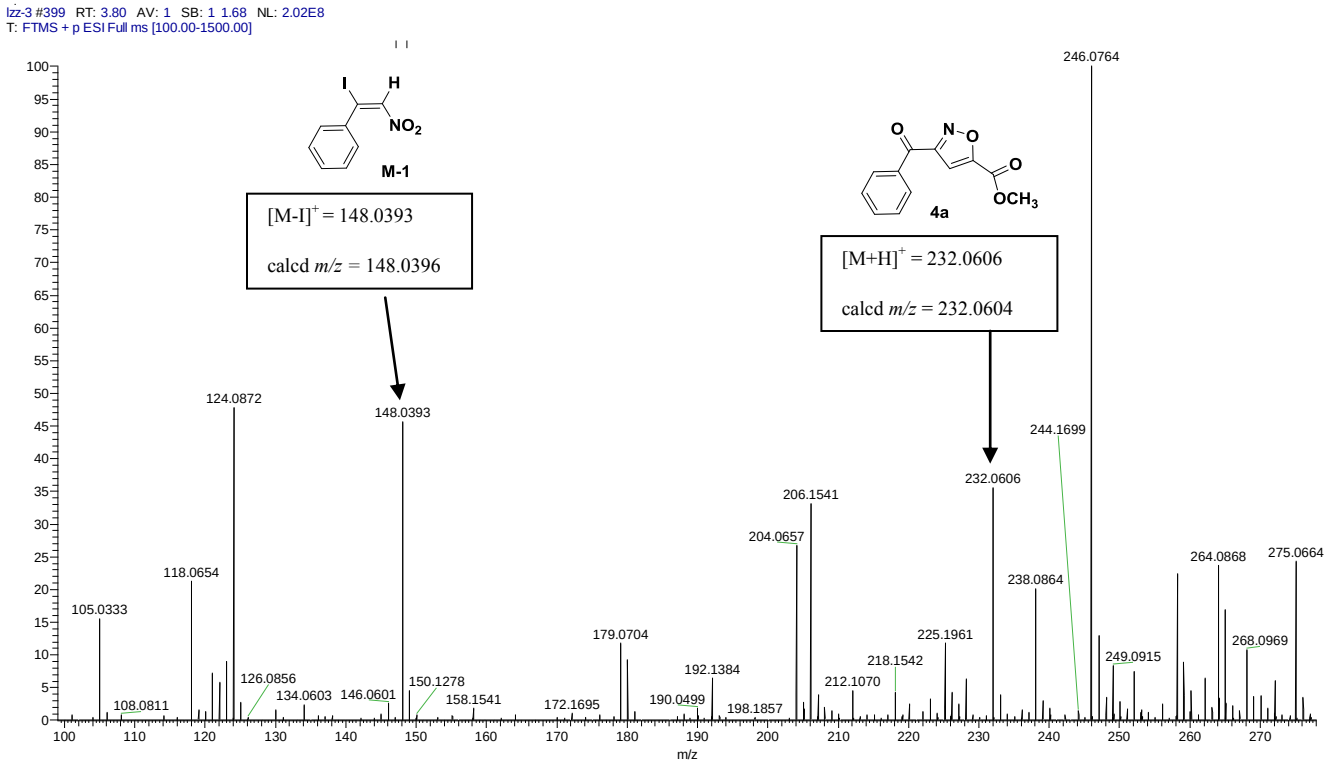


Figure S1. ESI-HRMS spectrogram of reaction mixture of **1a** and **2a** [a] **(**positive ion**)** .

[a] Reaction condition: **1a** (1.5 mmol), **2a** (3 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (6 mmol), KI (1.5 mmol), *t*-BuCN (3 mmol), EtOAc/PhCN (2.5 mL/2.5 mL), reaction time (30 min) at 70 ^{o}C under nitrogen atmosphere. The resulting solution is concentrated by a rotary evaporator, and extracted with EtOAc. The organic layer was used for the ESI-HRMS test.

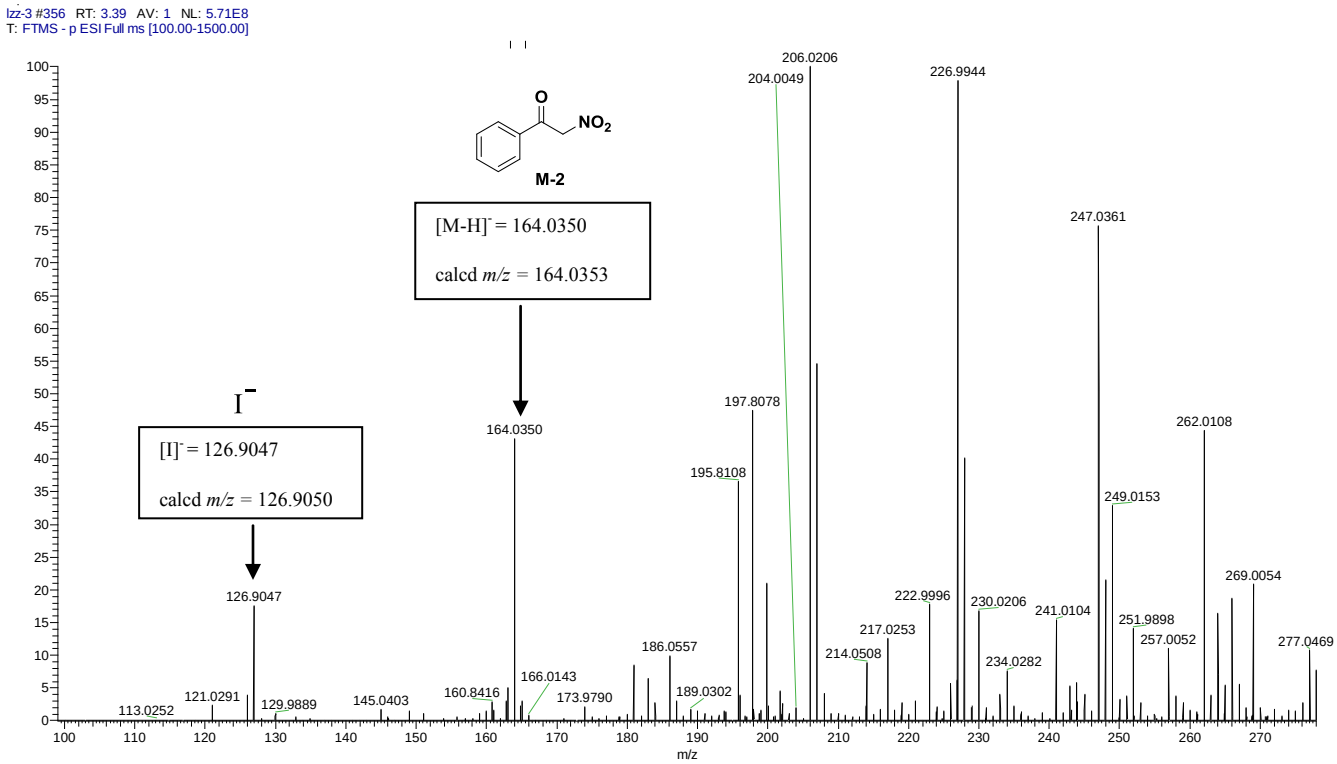
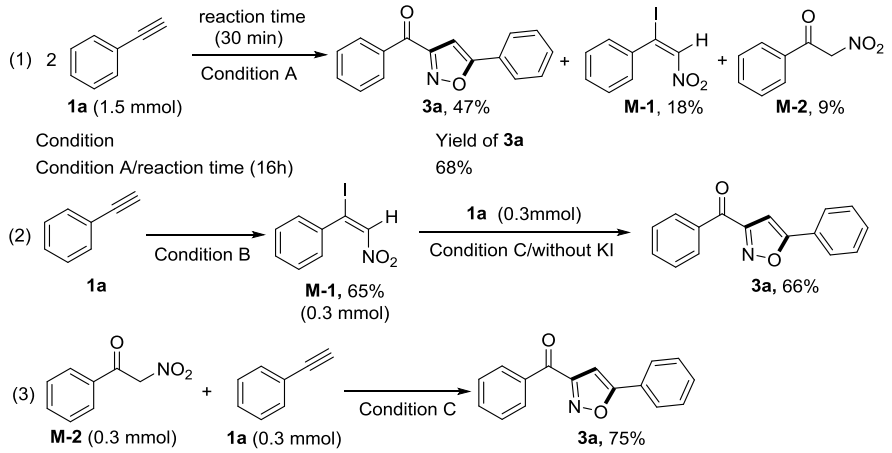


Figure S2. ESI-HRMS spectrogram of reaction mixture of **1a** and **2a** [a] (negative ion).

[a] Reaction condition: **1a** (1.5 mmol), **2a** (3 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (6 mmol), KI (1.5 mmol), *t*-BuCN (3 mmol), EtOAc/PhCN (2.5 mL/2.5 mL), reaction time (30 min) at 70 ^{o}C under nitrogen atmosphere. The resulting solution is concentrated by a rotary evaporator, and extracted with EtOAc. The organic layer was used for the ESI-HRMS test.

Scheme S1. Control experiments about self-coupling and cyclizing of aromatic terminal alkynes



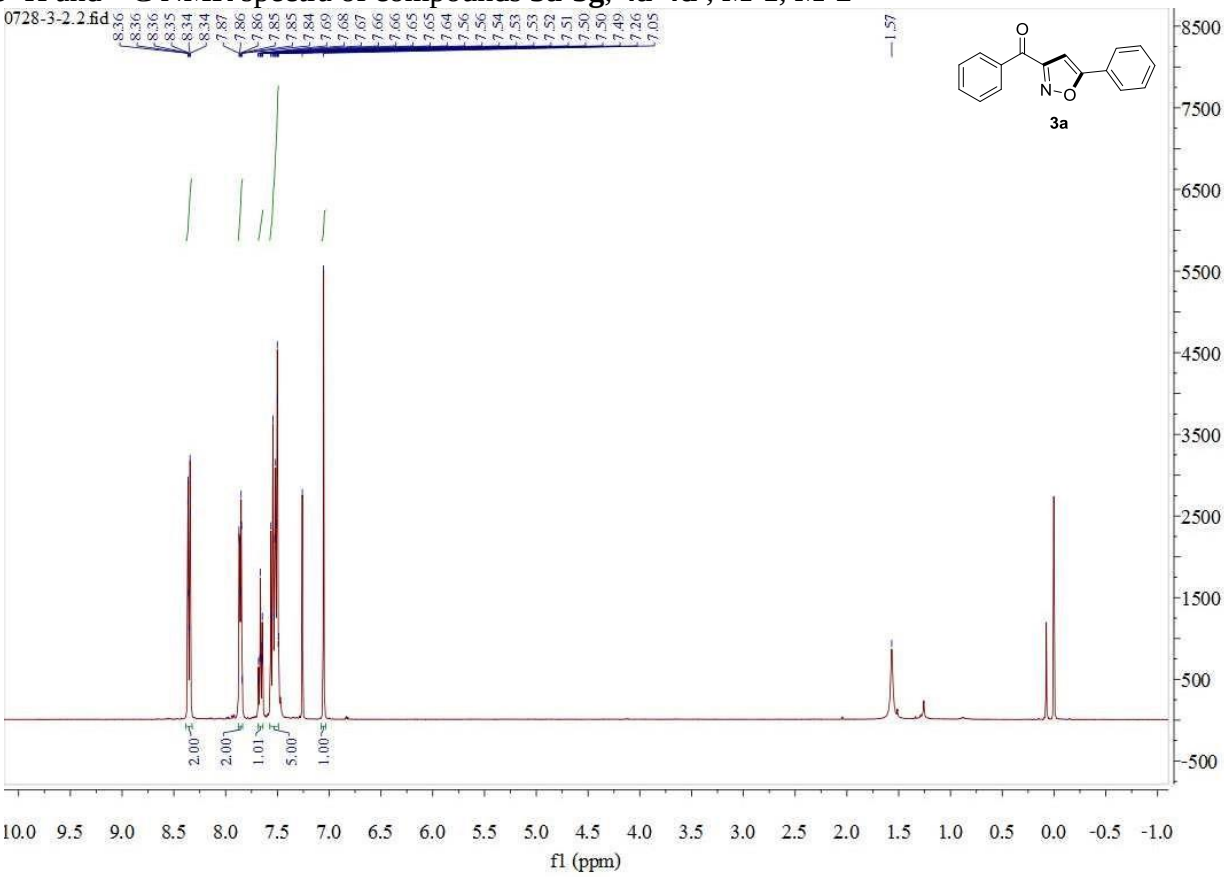
Condition A: $Fe(NO_3)_3 \cdot 9H_2O$ (2.25 mmol), KI (0.75 mmol), THF (5 mL), reaction temperature (60 ^oC), reaction time (16 h) under nitrogen atmosphere; Condition B: $NaNO_3$ (3 equiv), KI (1.5 equiv), acetic acid as solvent, reaction time (3h) at 85 ^oC in air; Condition C: $Fe(NO_3)_3 \cdot 9H_2O$ (0.9 mmol), KI (0.3 mmol), THF (2 mL), reaction temperature (60 ^oC), reaction time (16 h) under nitrogen atmosphere.

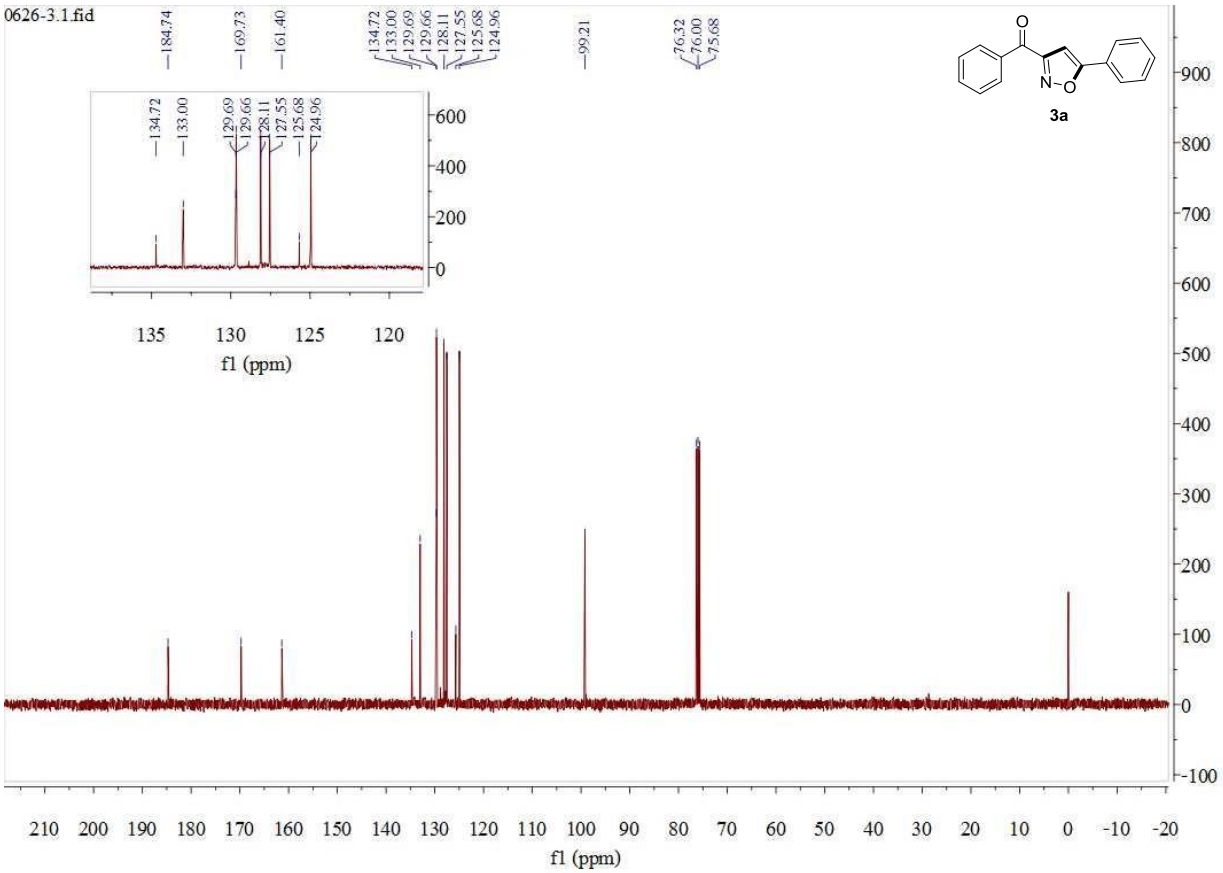
Reference

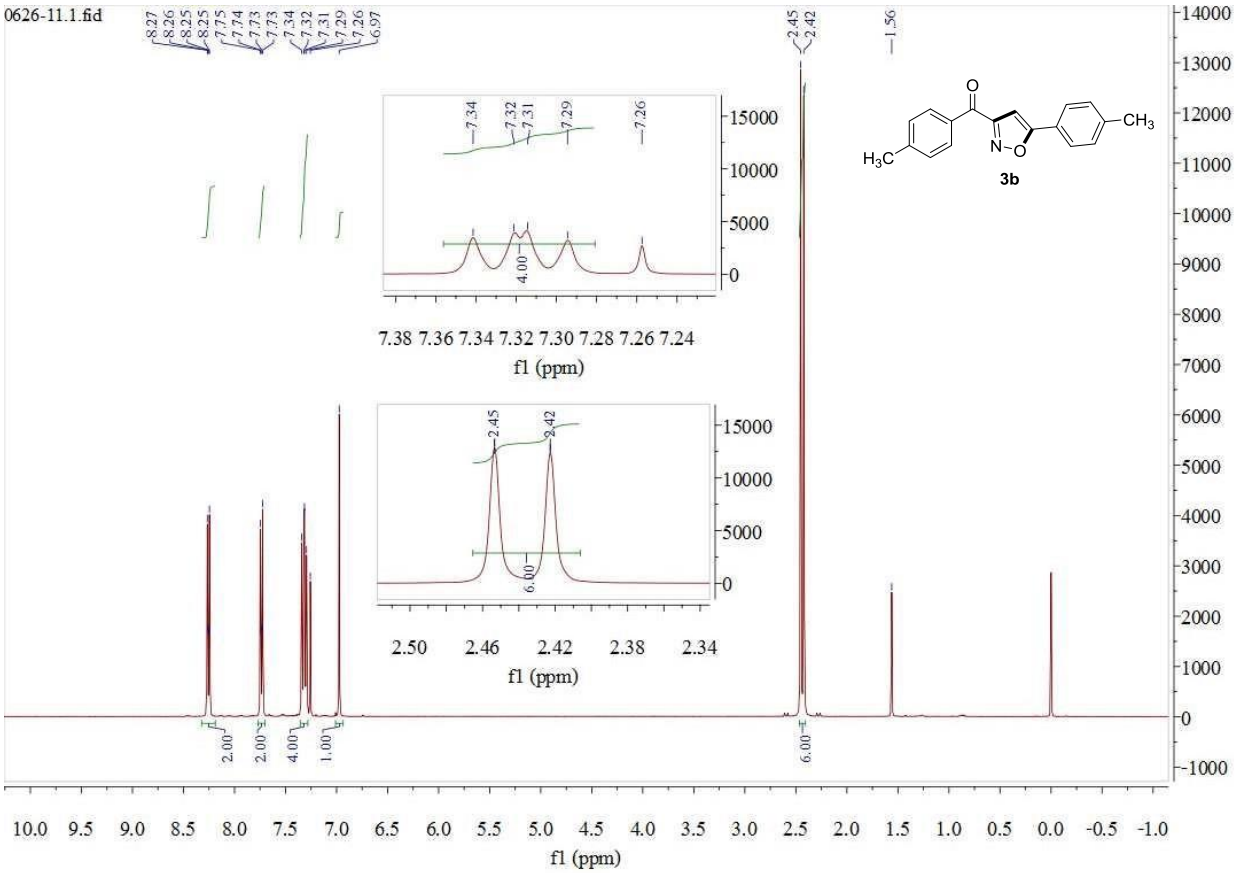
(1) Yusubov, M. S.; Perederina, I. A.; Filimonov, V. D.; Park, T. -H.; Chi, K. -W. *Synth. Commun.* **1998**, *28*, 833.

(2) Sarkar, R.; Mukherjee, S. *Org. Lett.* **2016**, *18*, 6160.

The ^{1}H and ^{13}C NMR spectra of compounds **3a-3g**, **4a-4d'**, **M-1**, **M-2**



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7.26
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f1 (ppm)
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6.00
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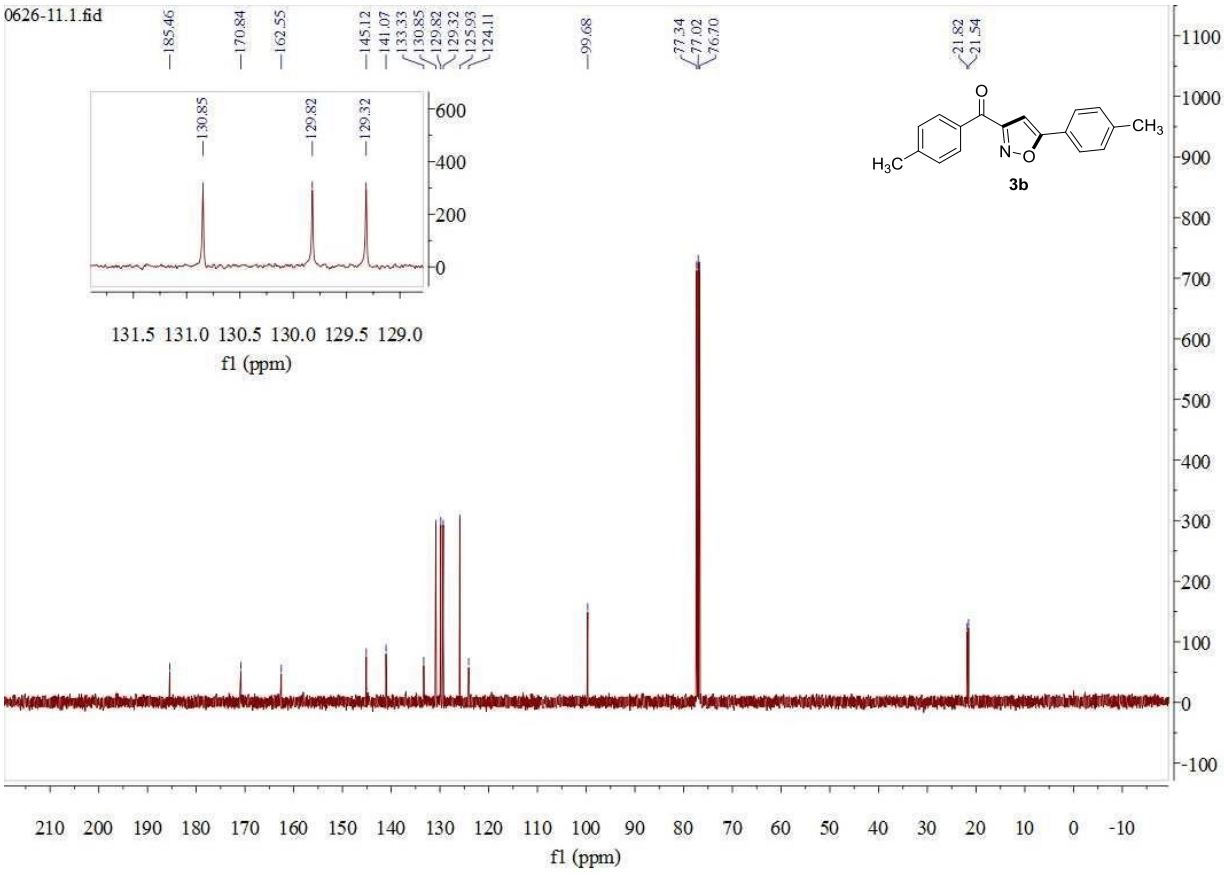
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N
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H_3C
CH_3
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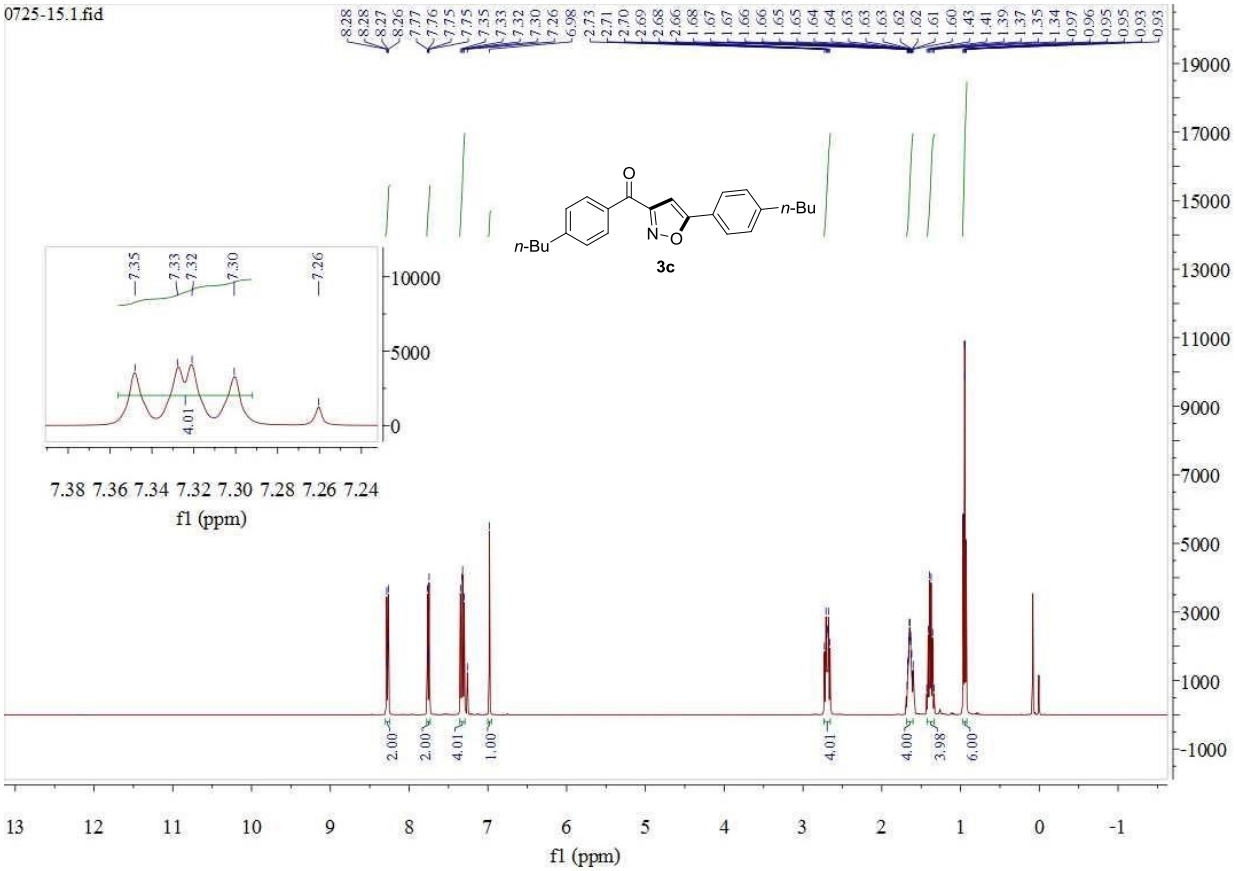
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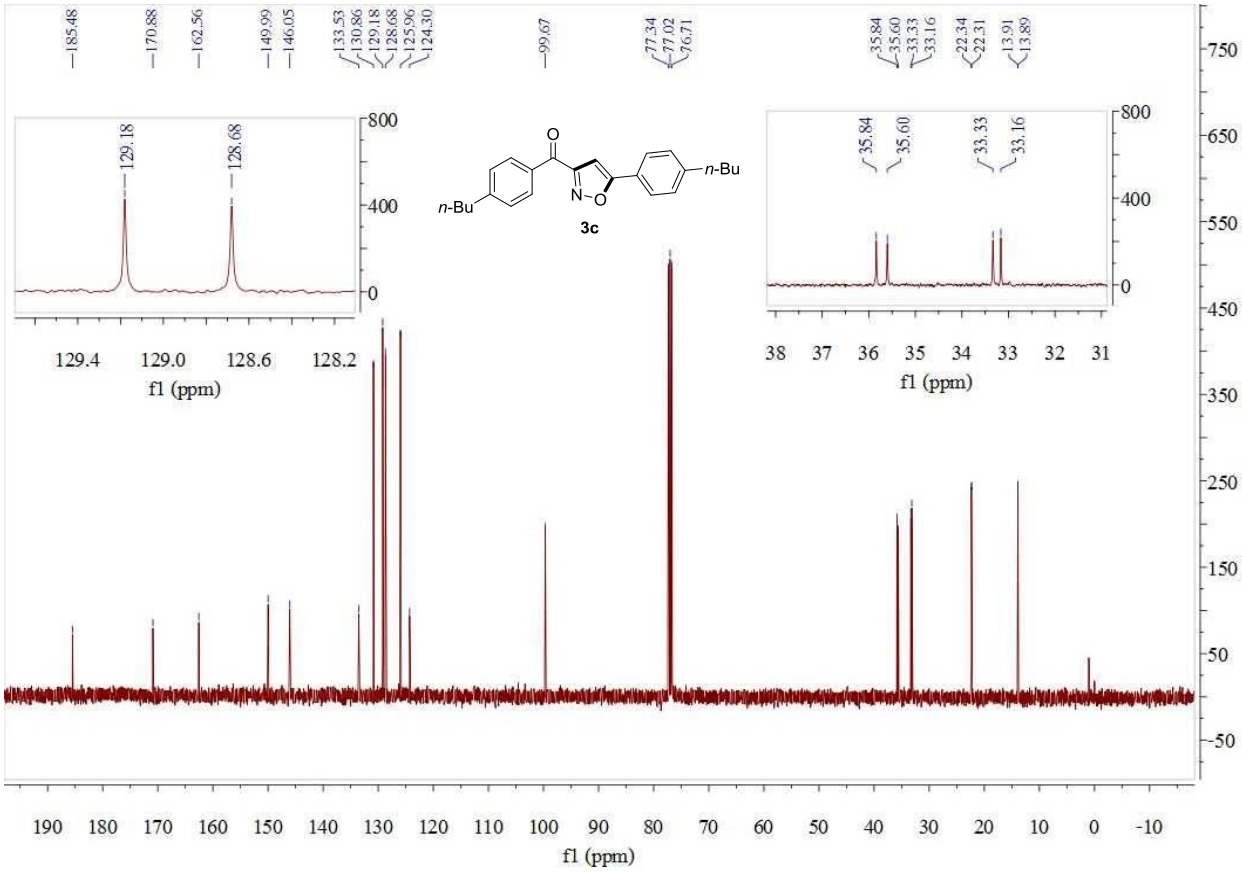
f1 (ppm)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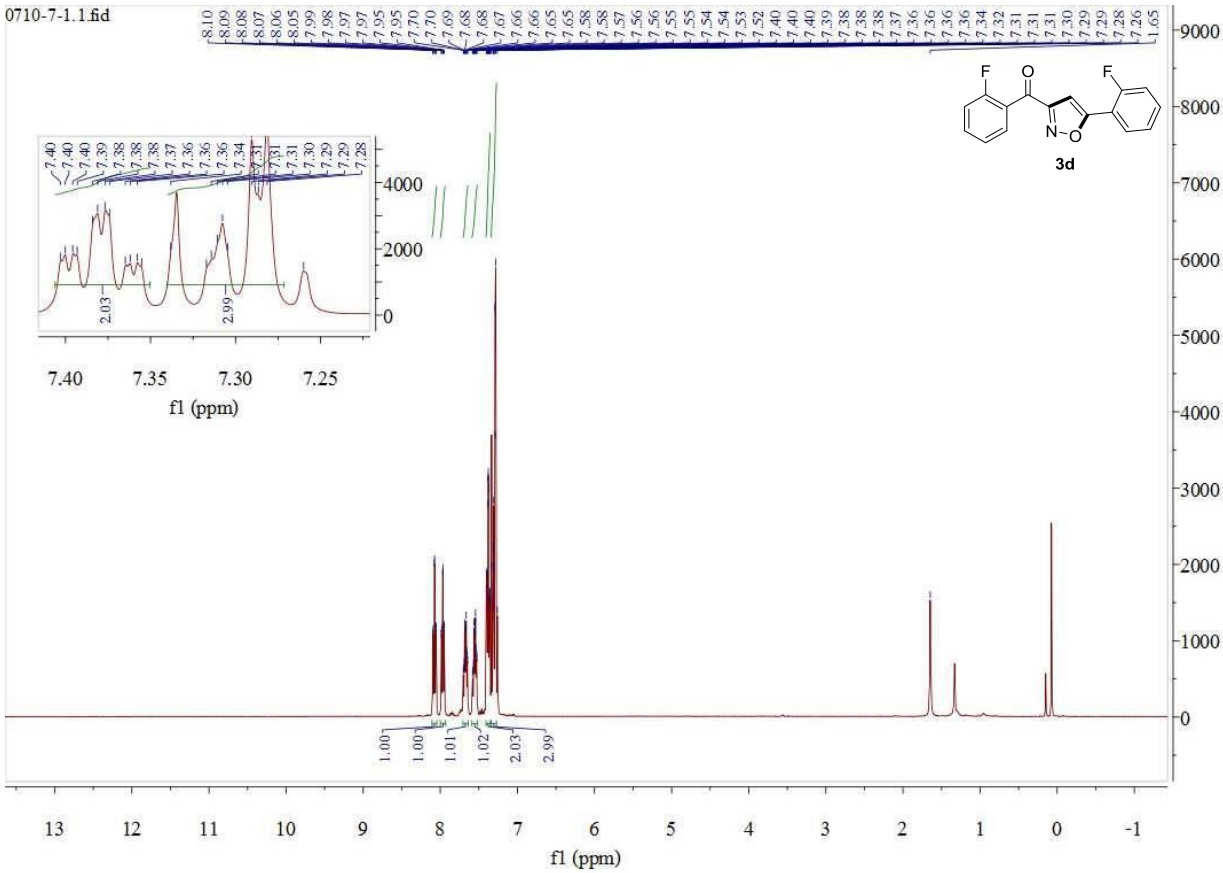
f1 (ppm)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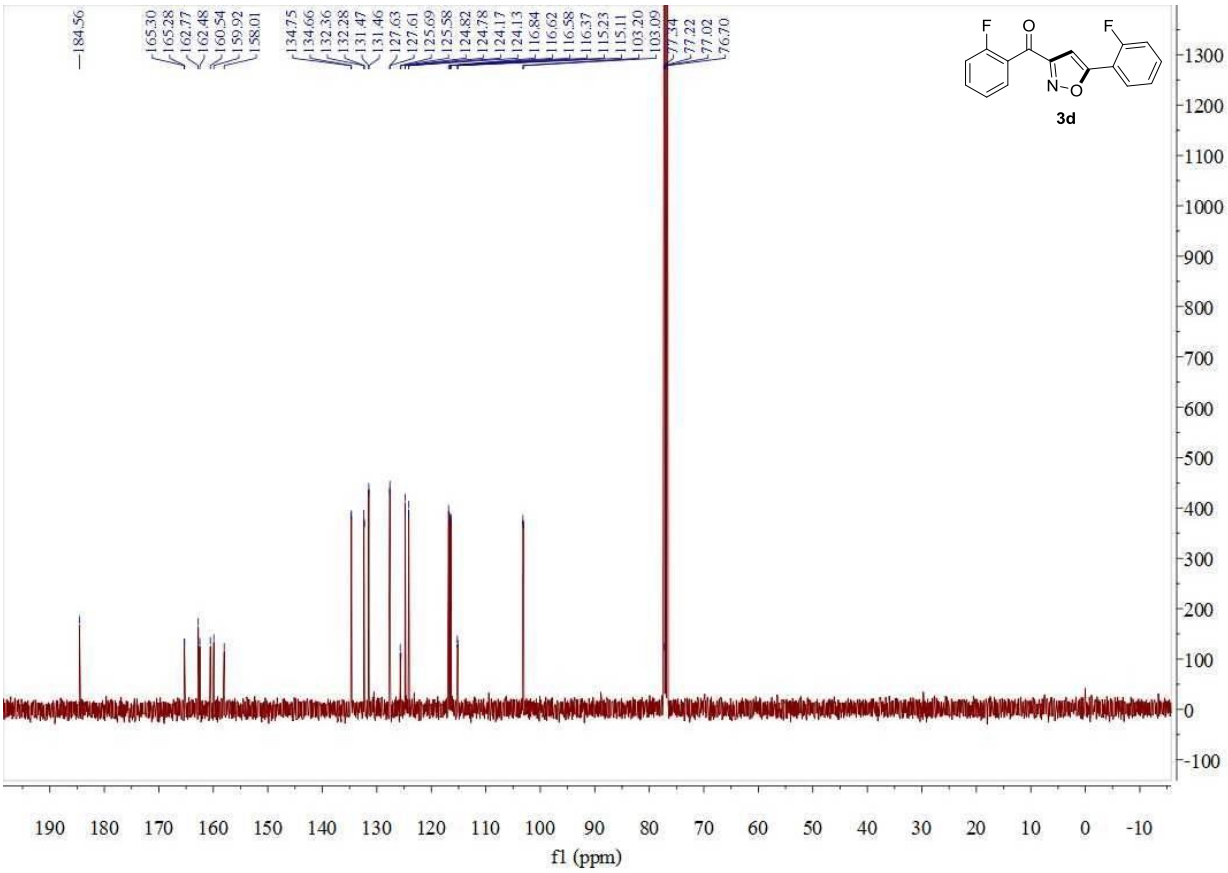
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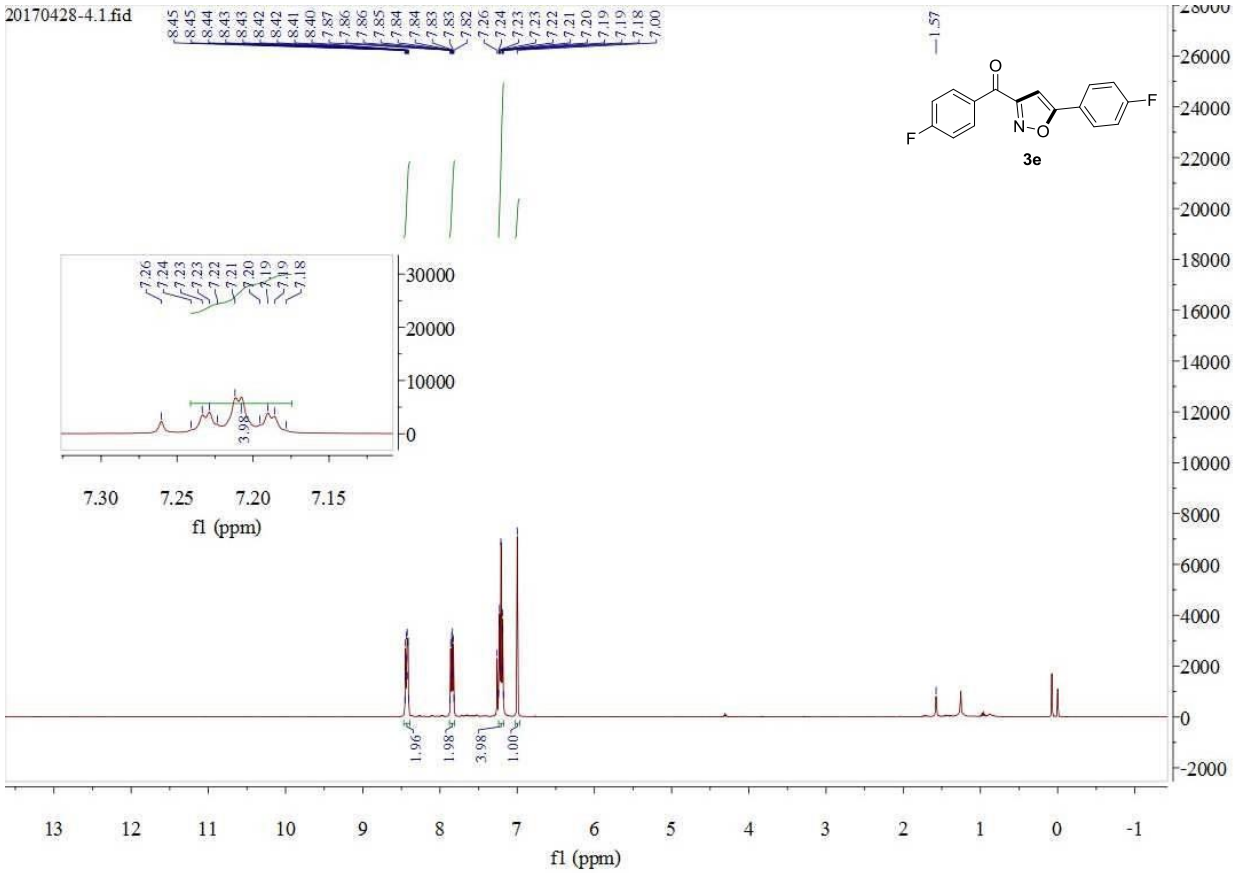
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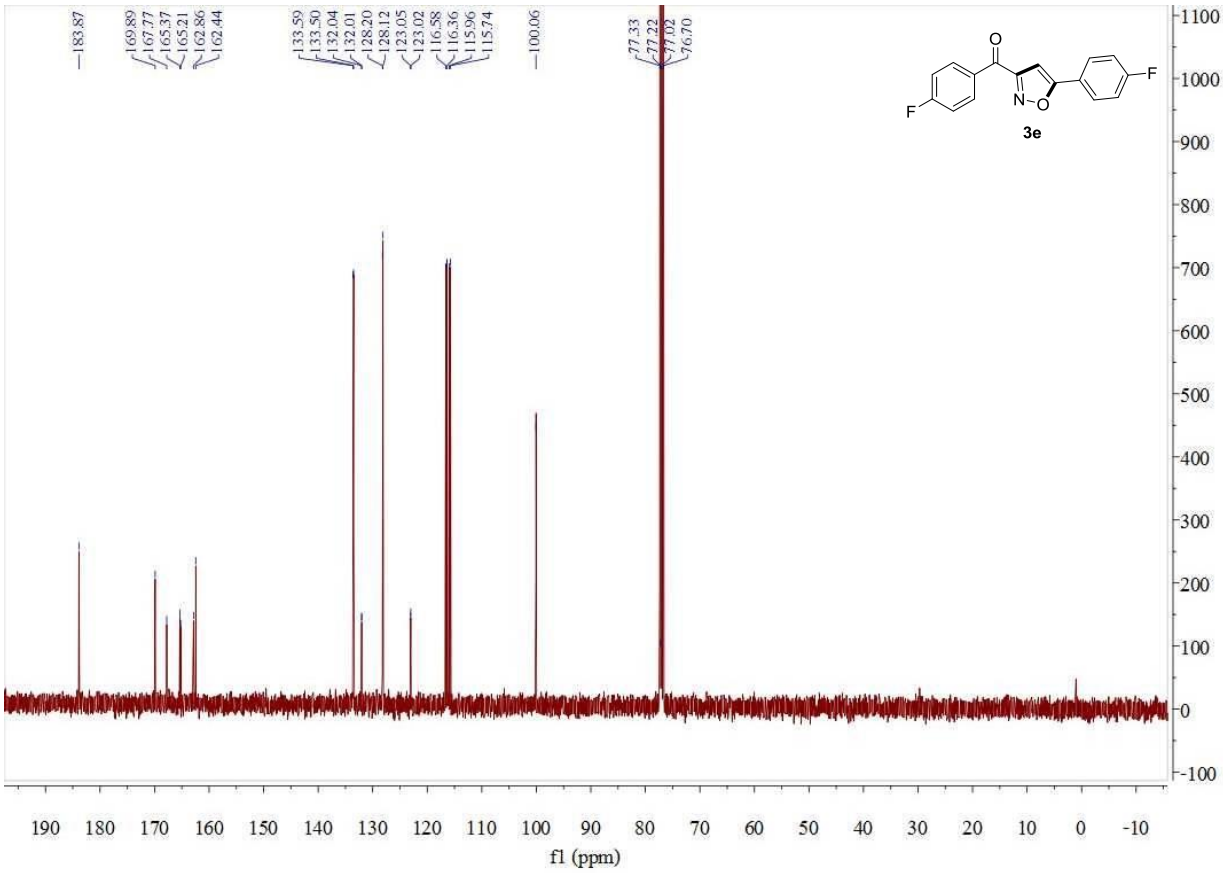
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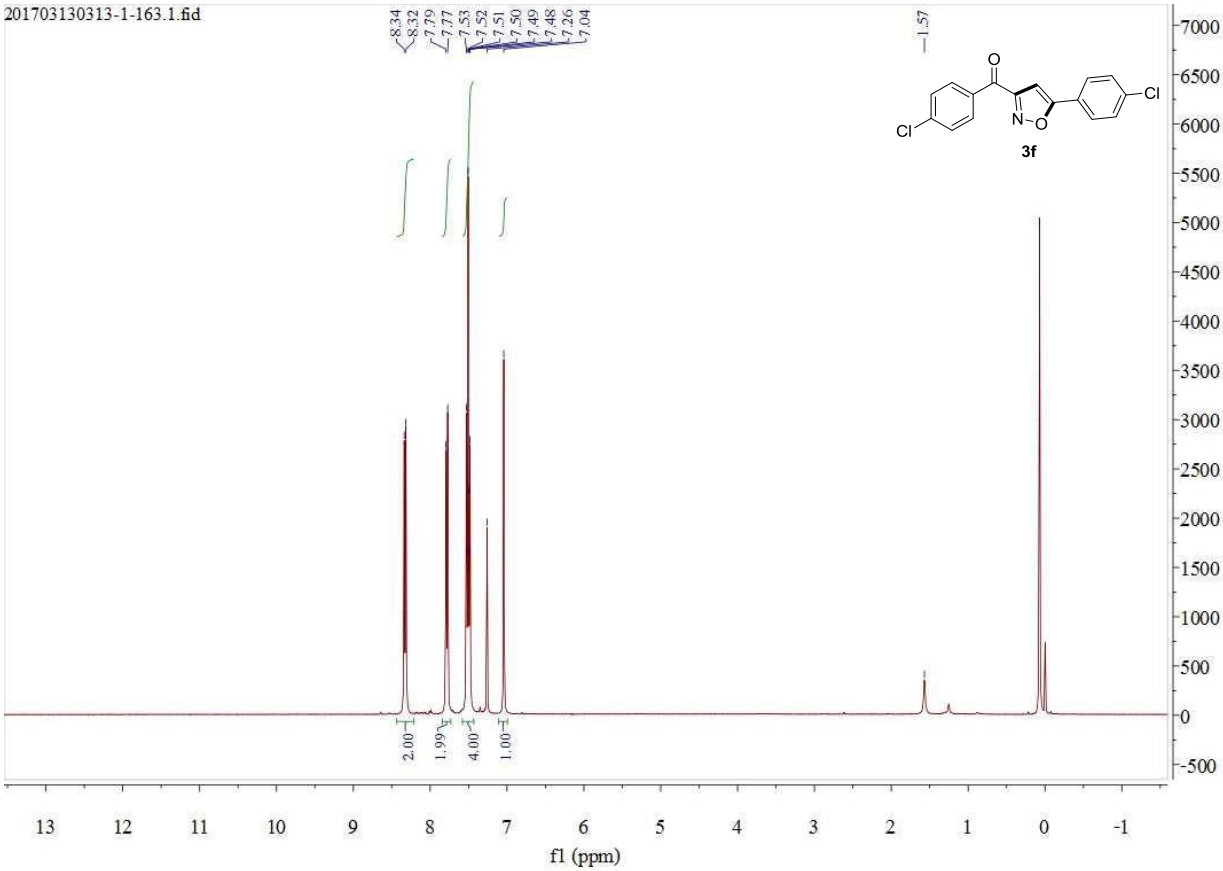
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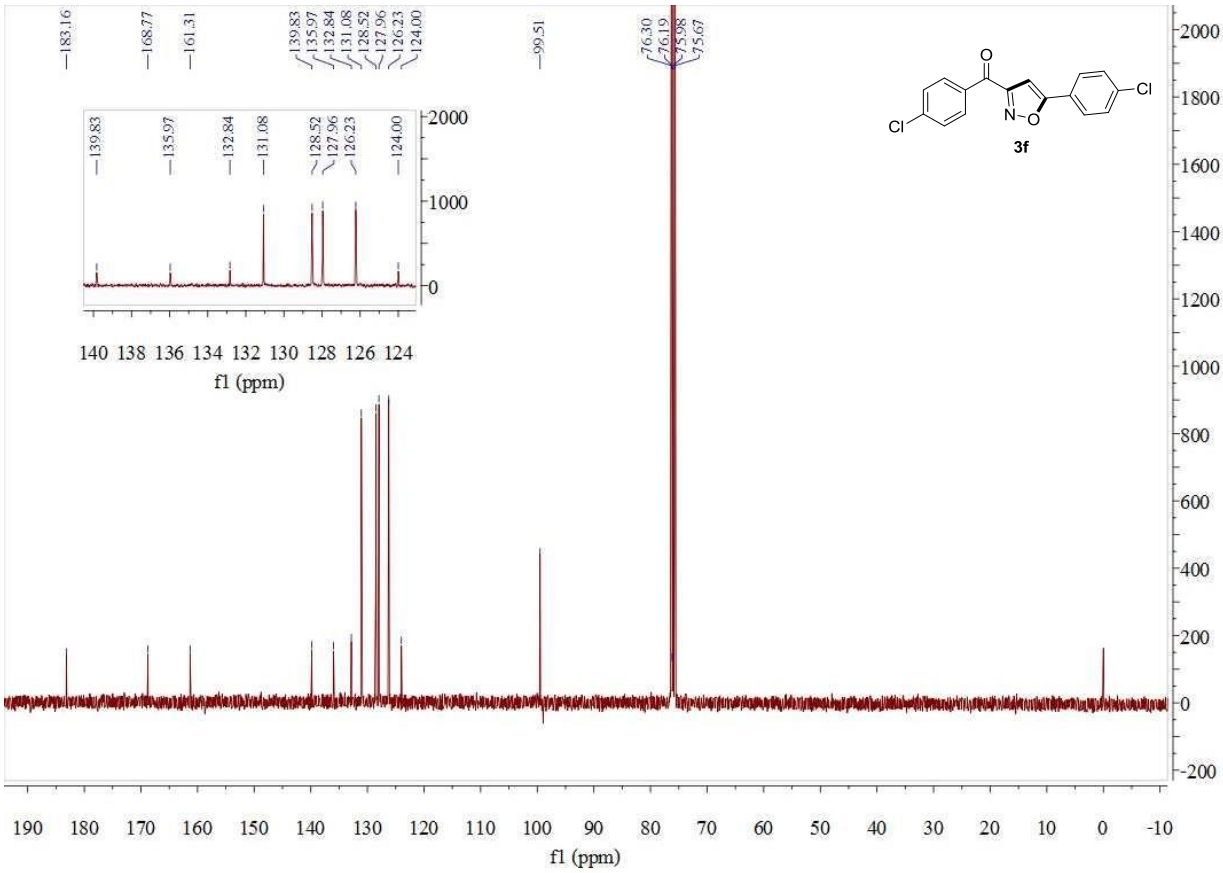
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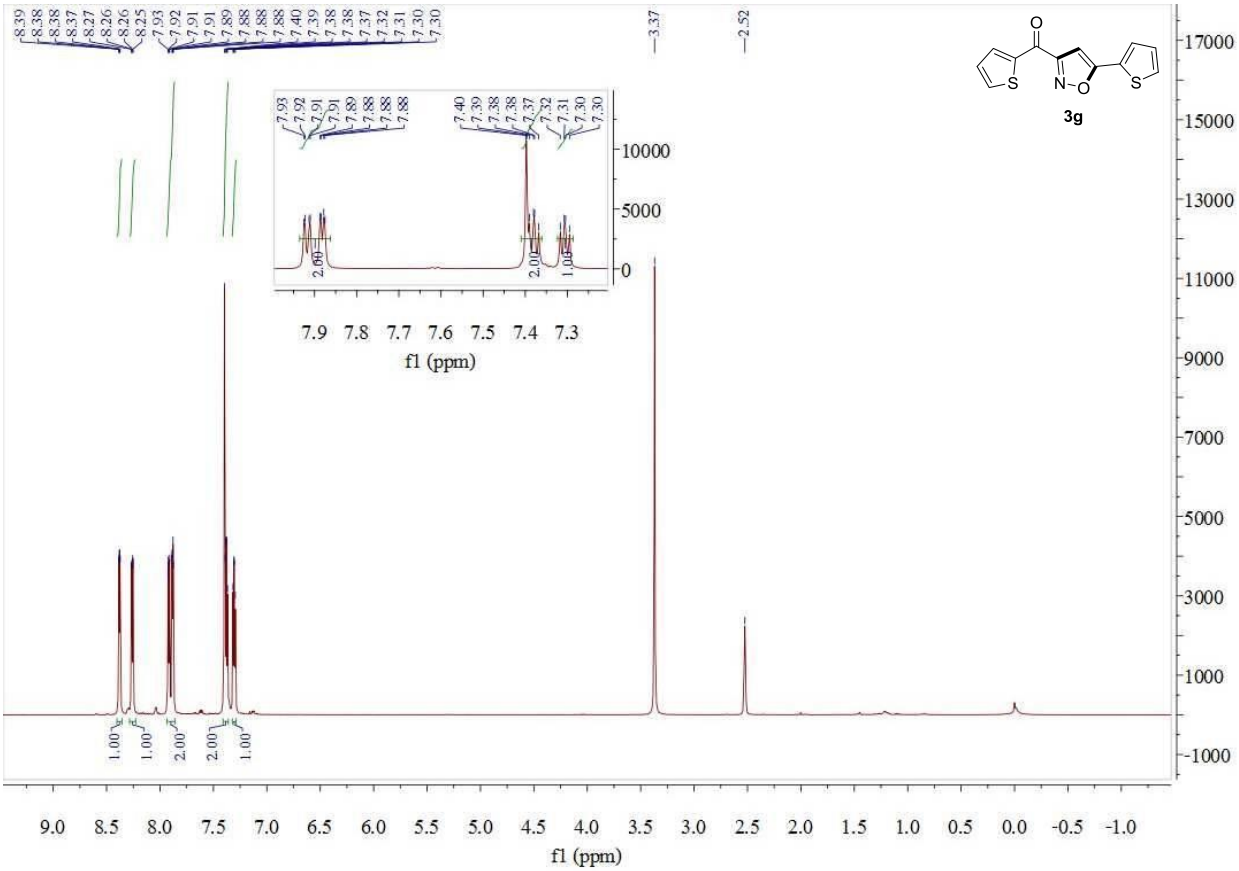
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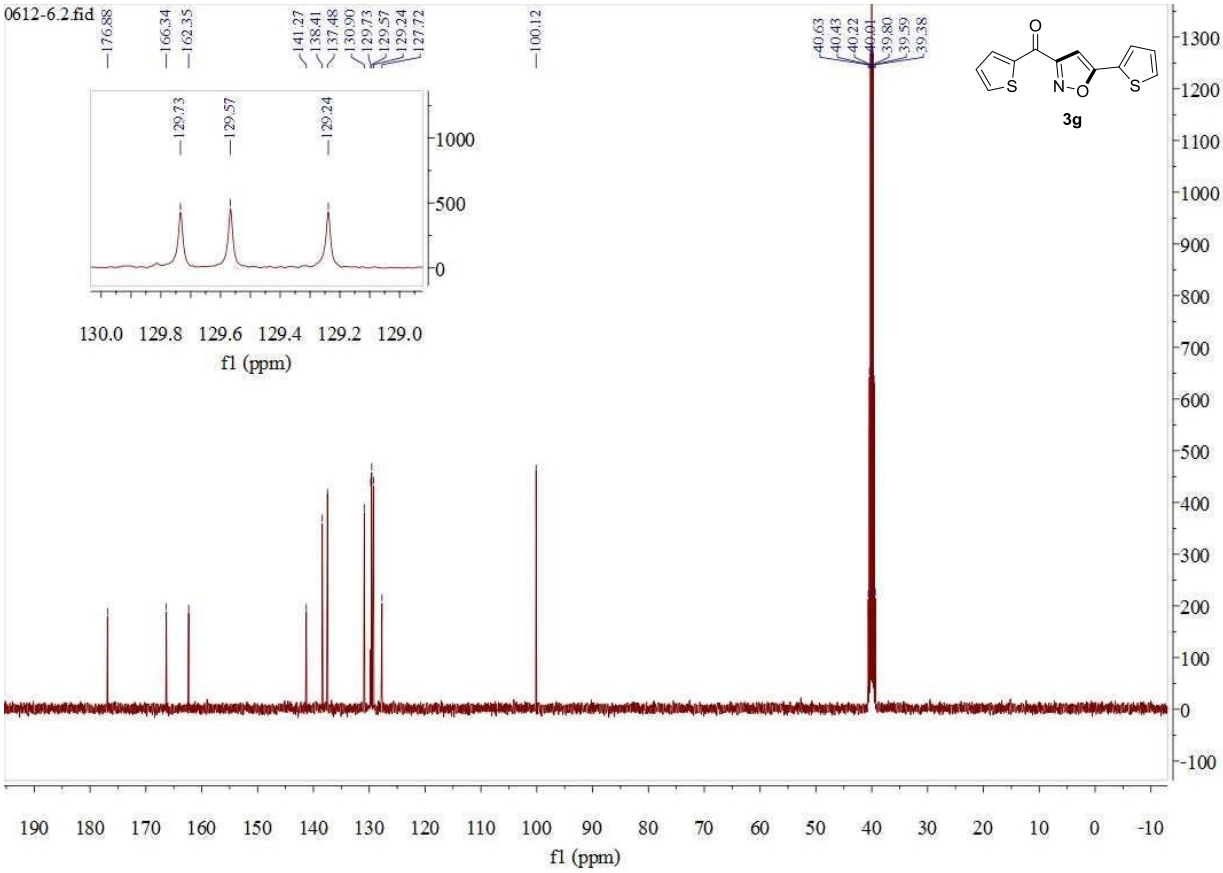
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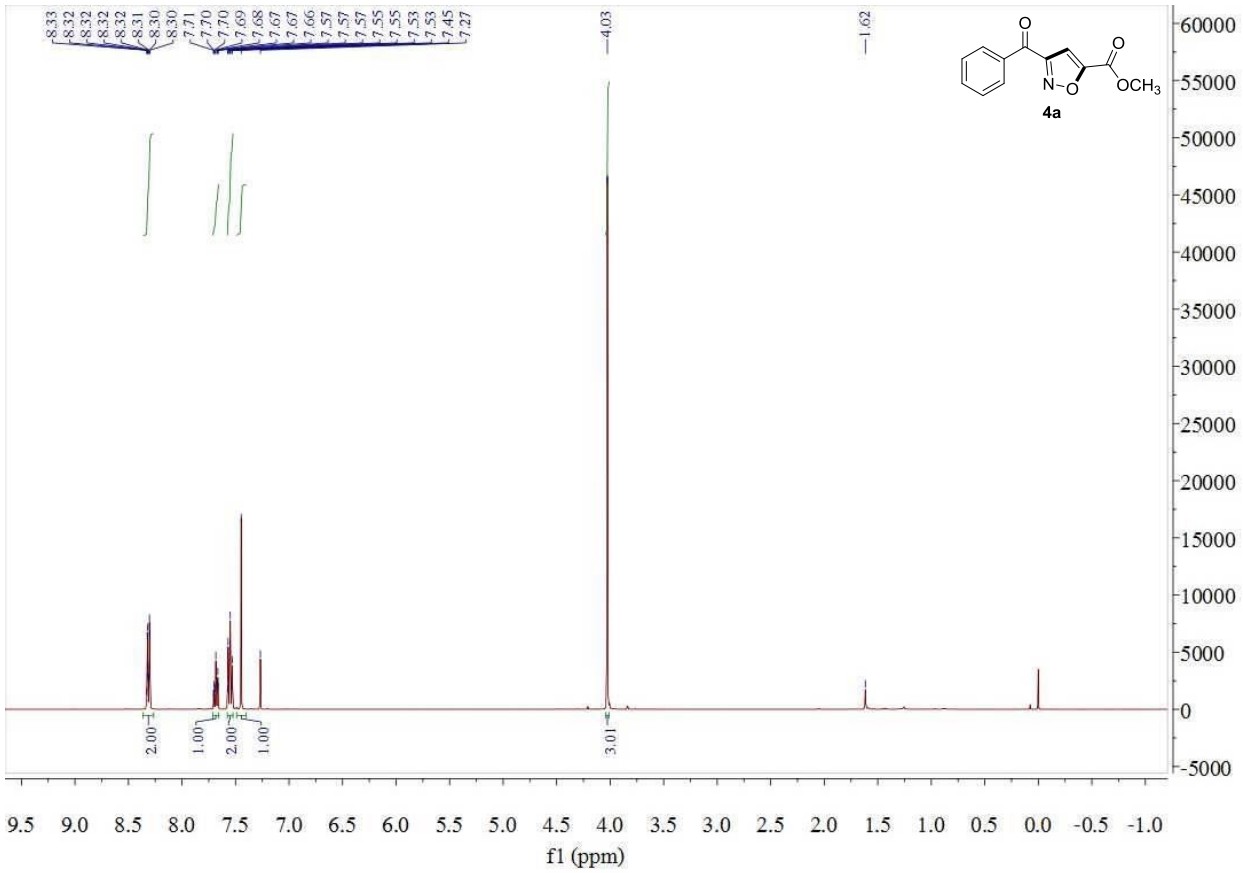
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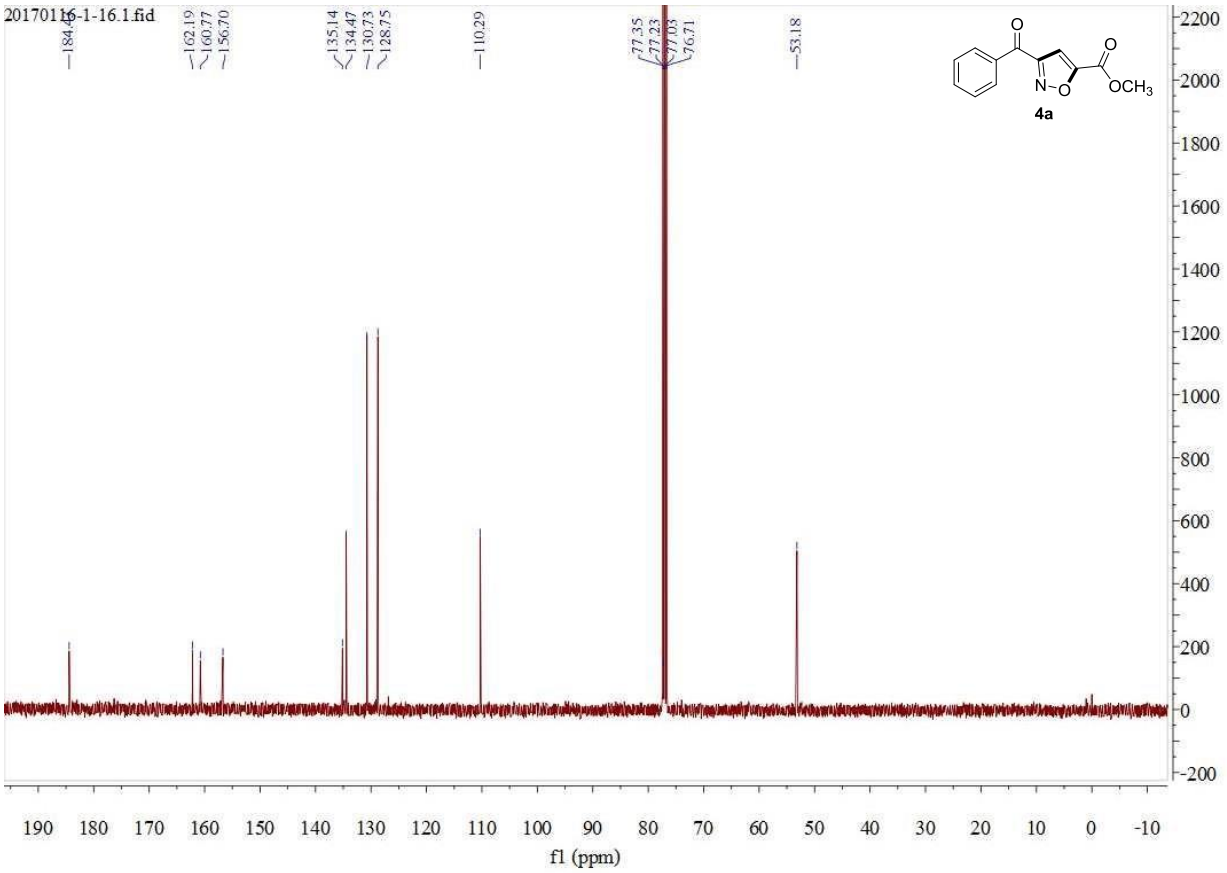
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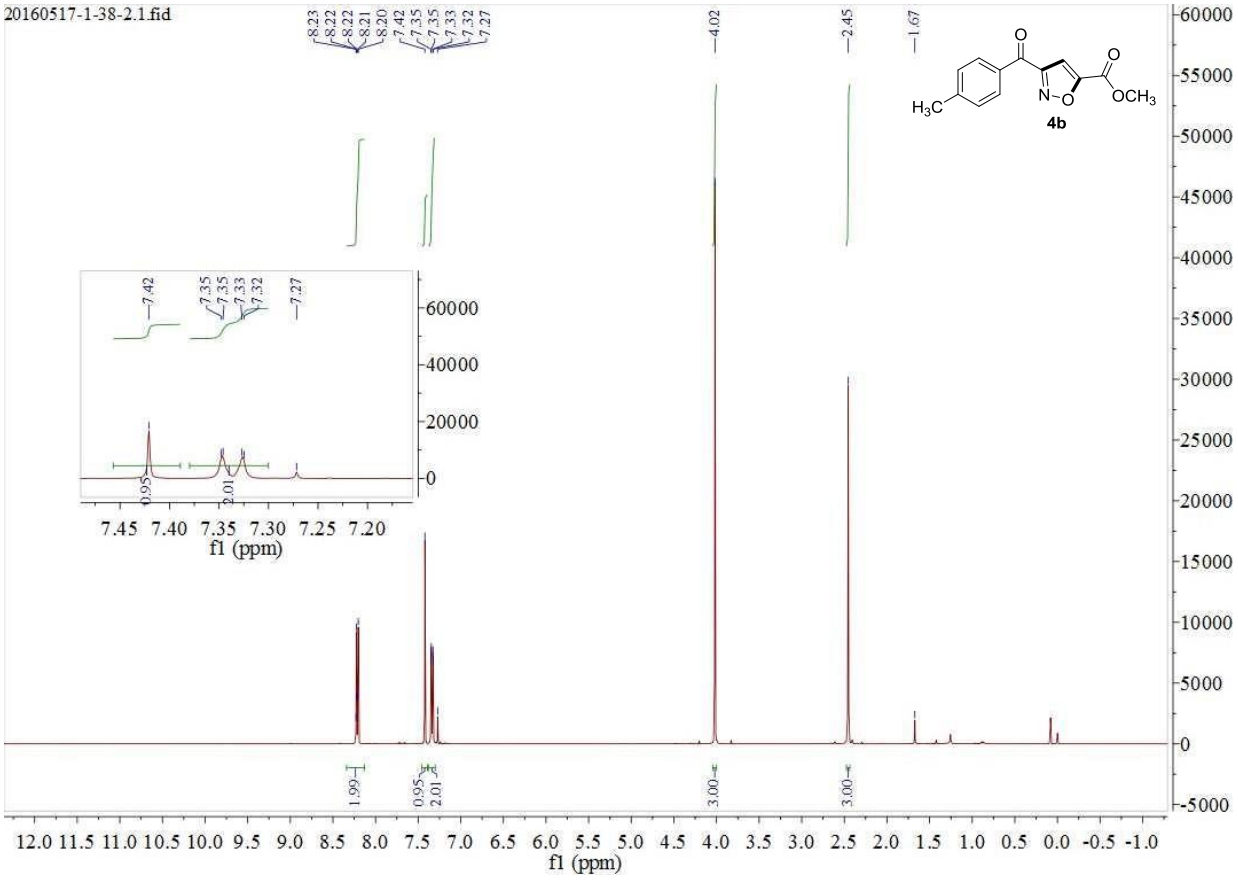
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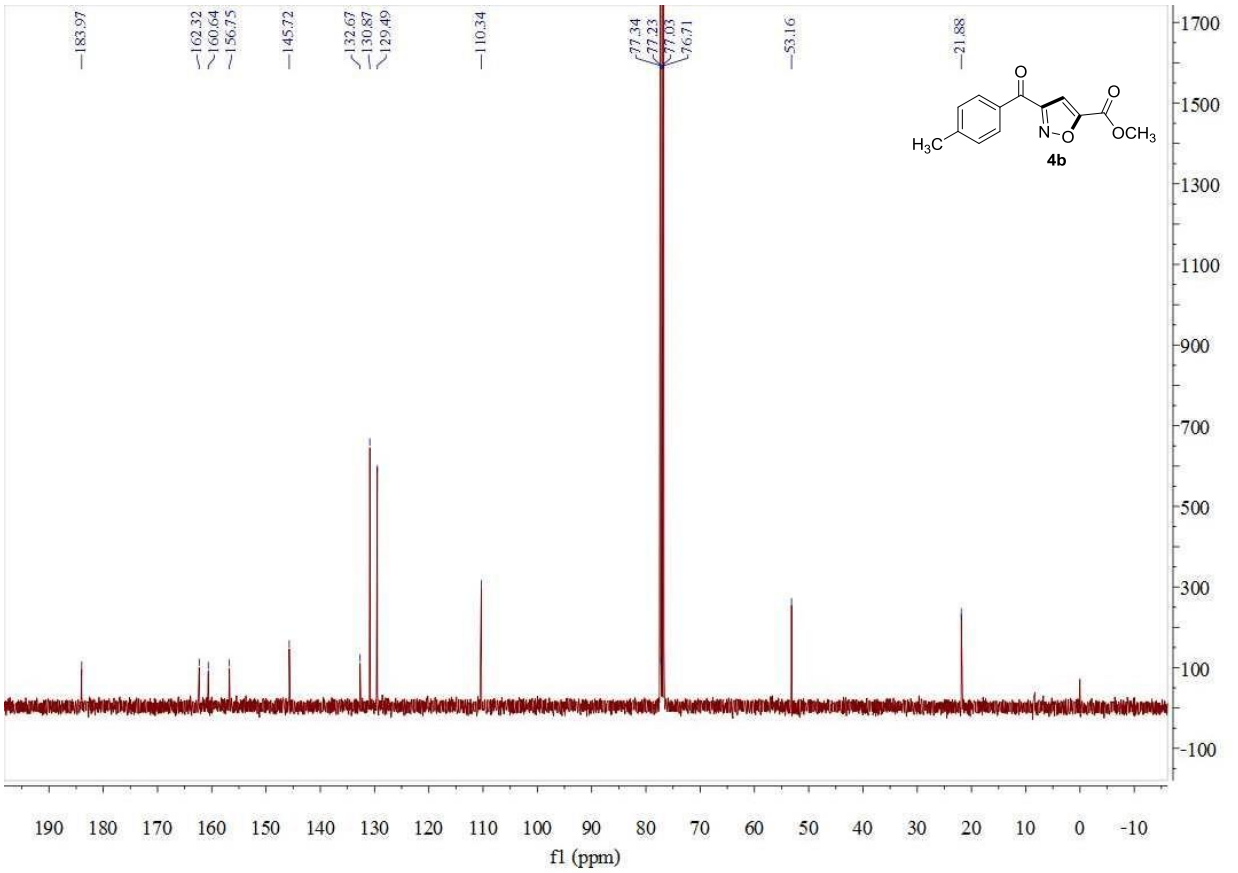
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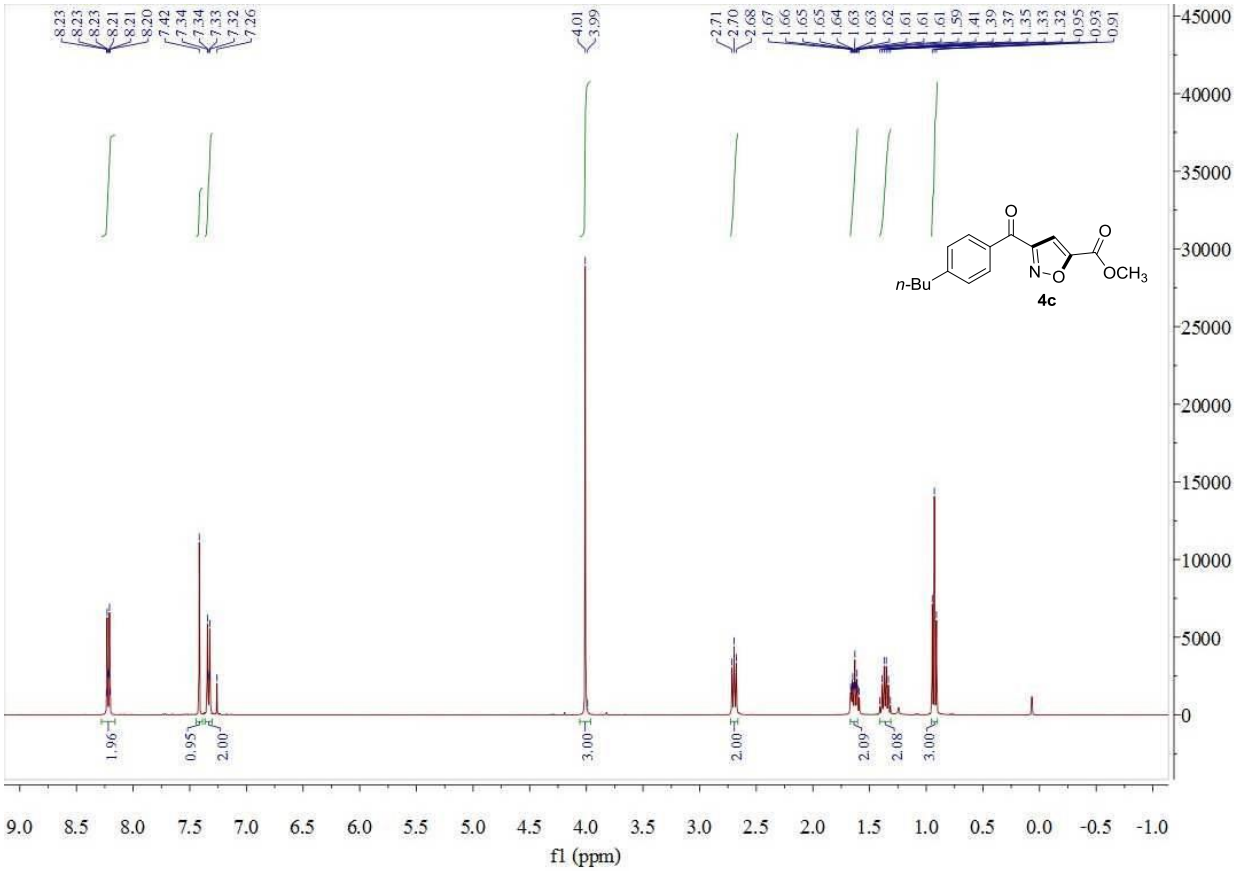
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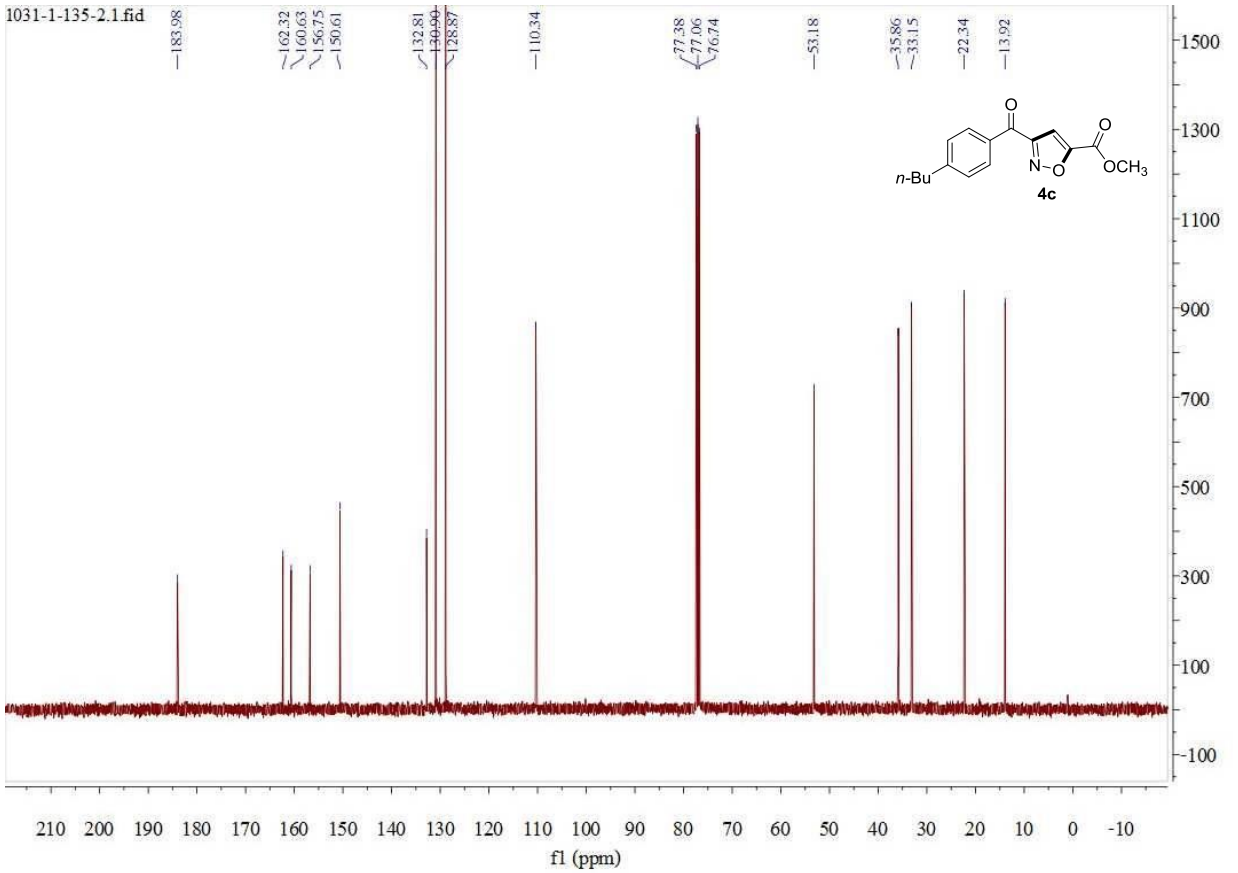
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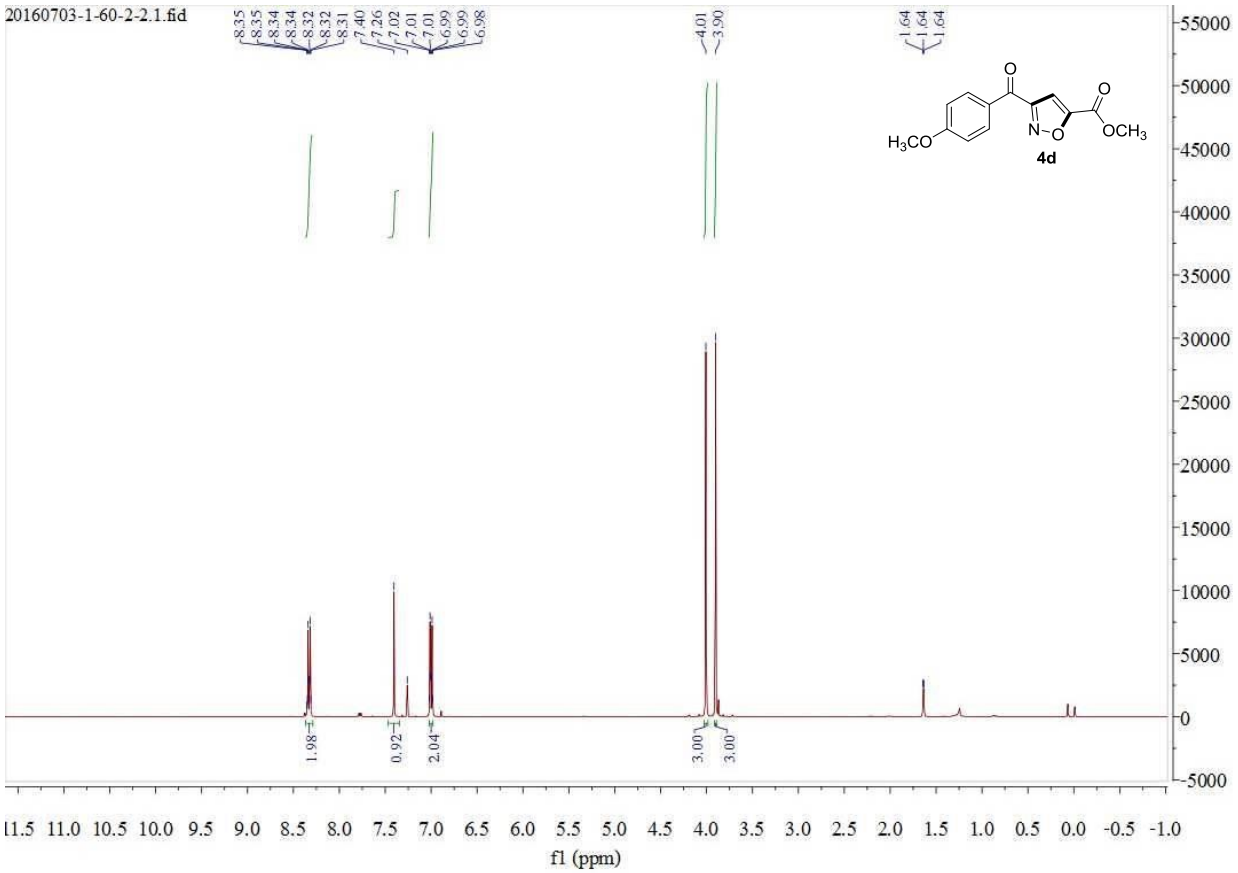
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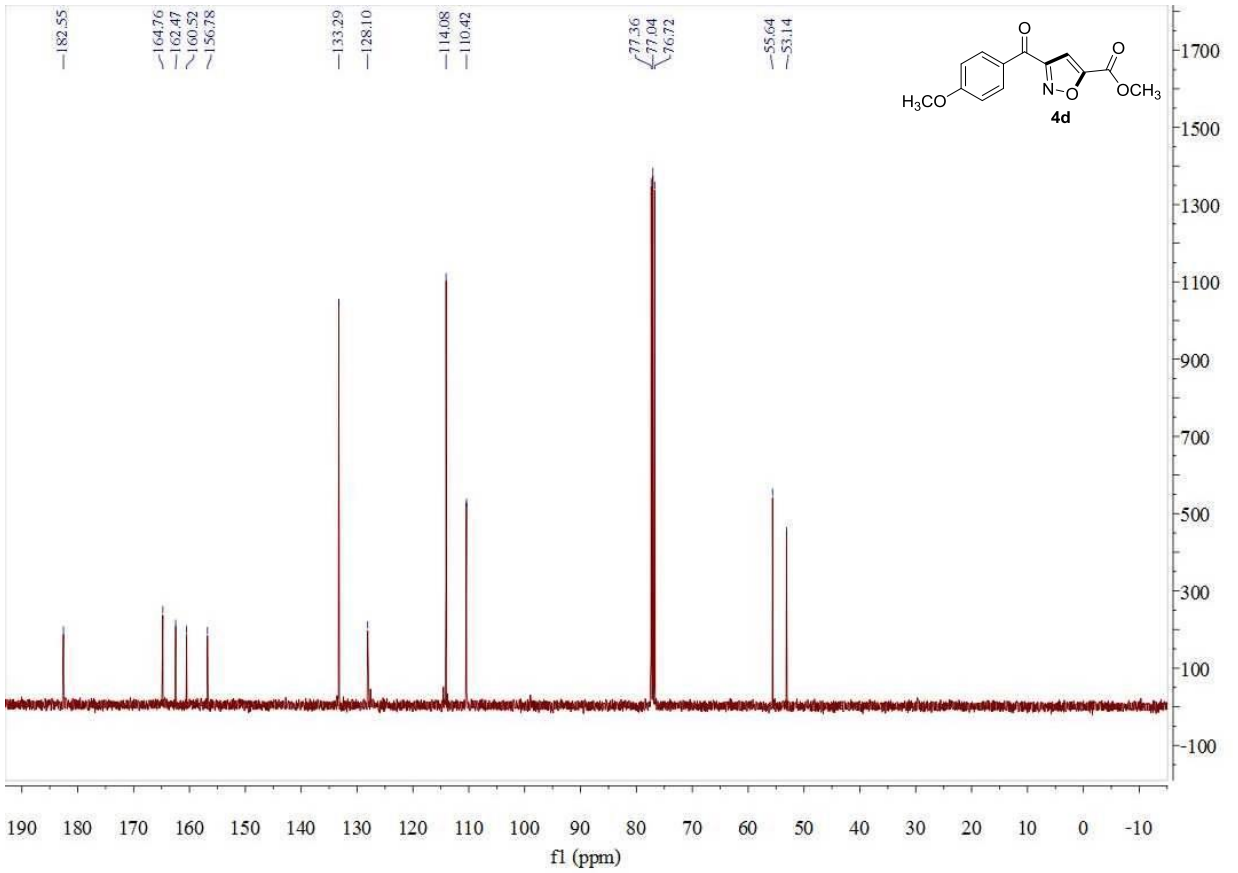
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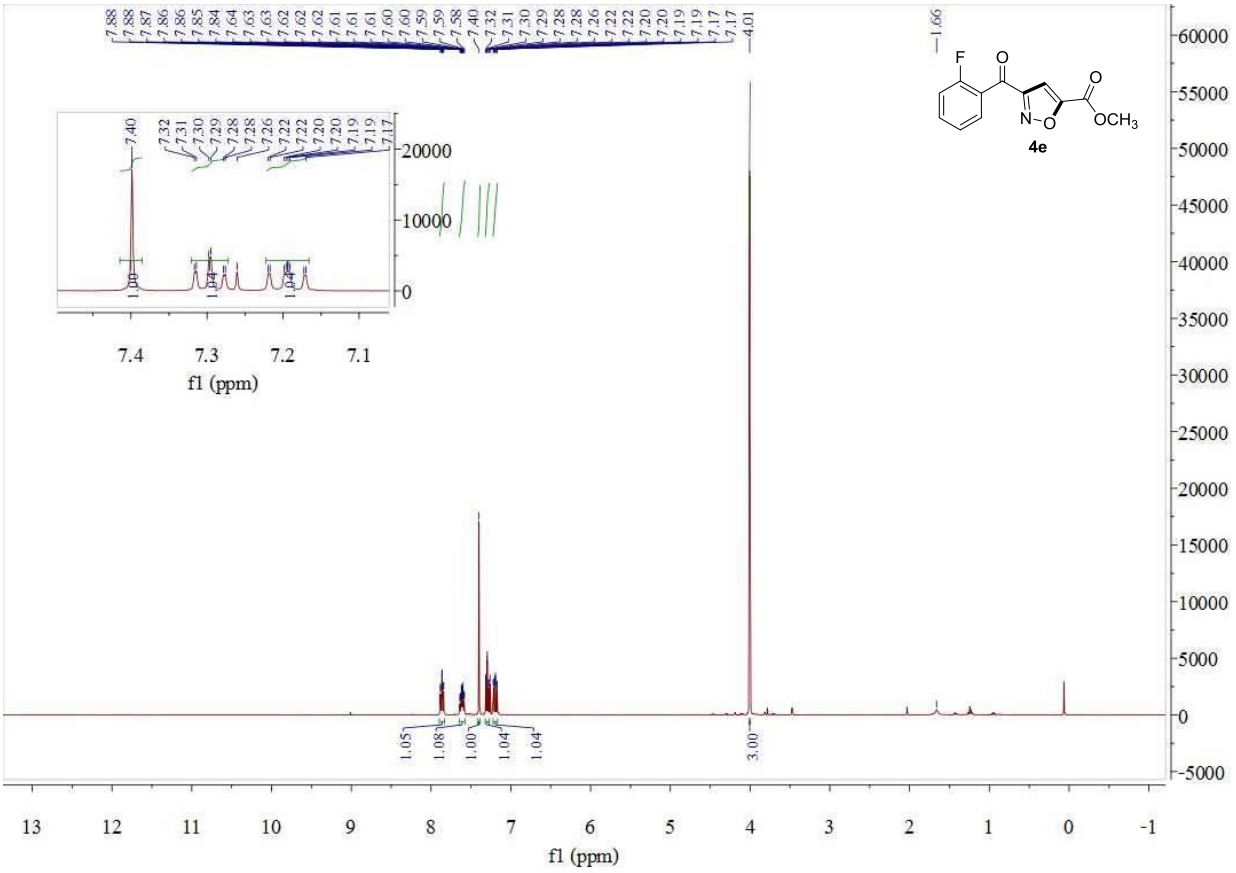
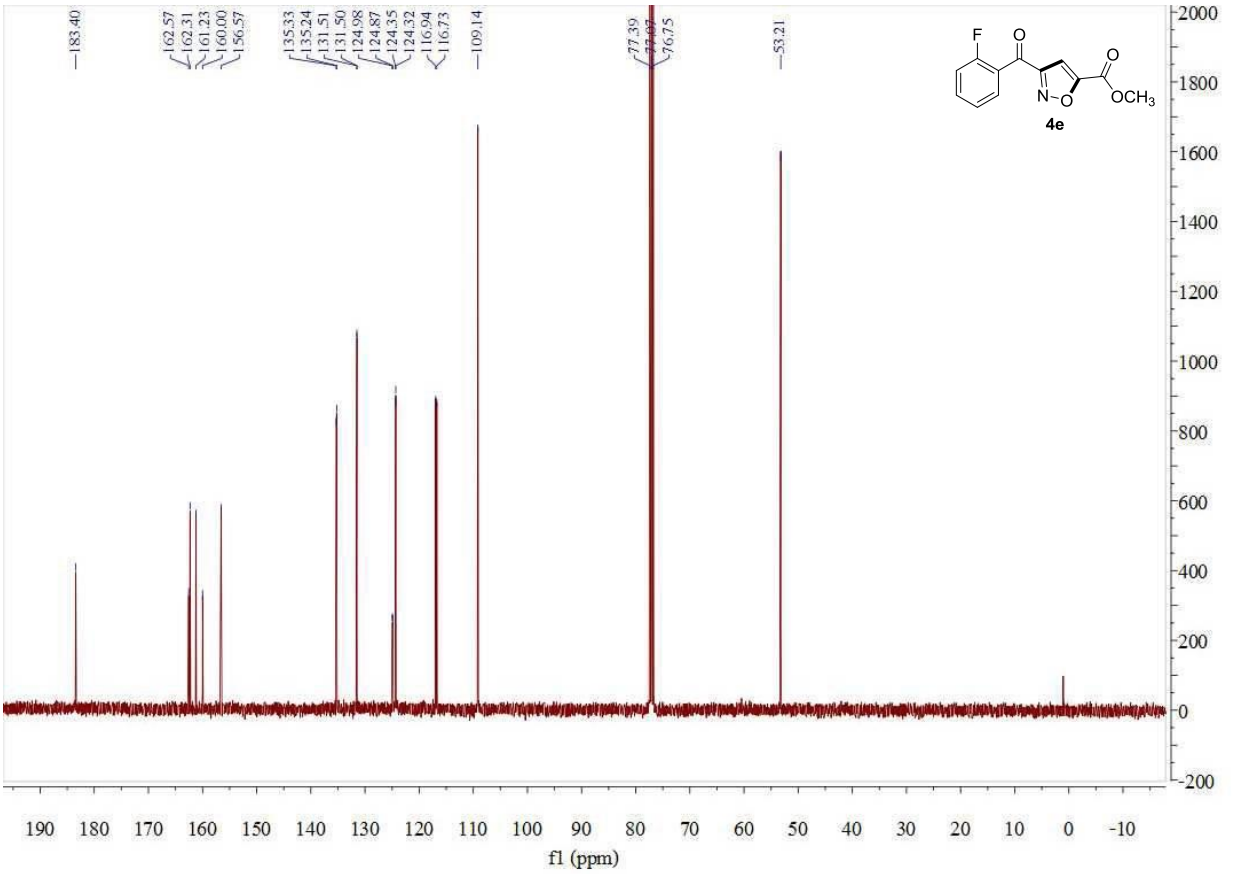
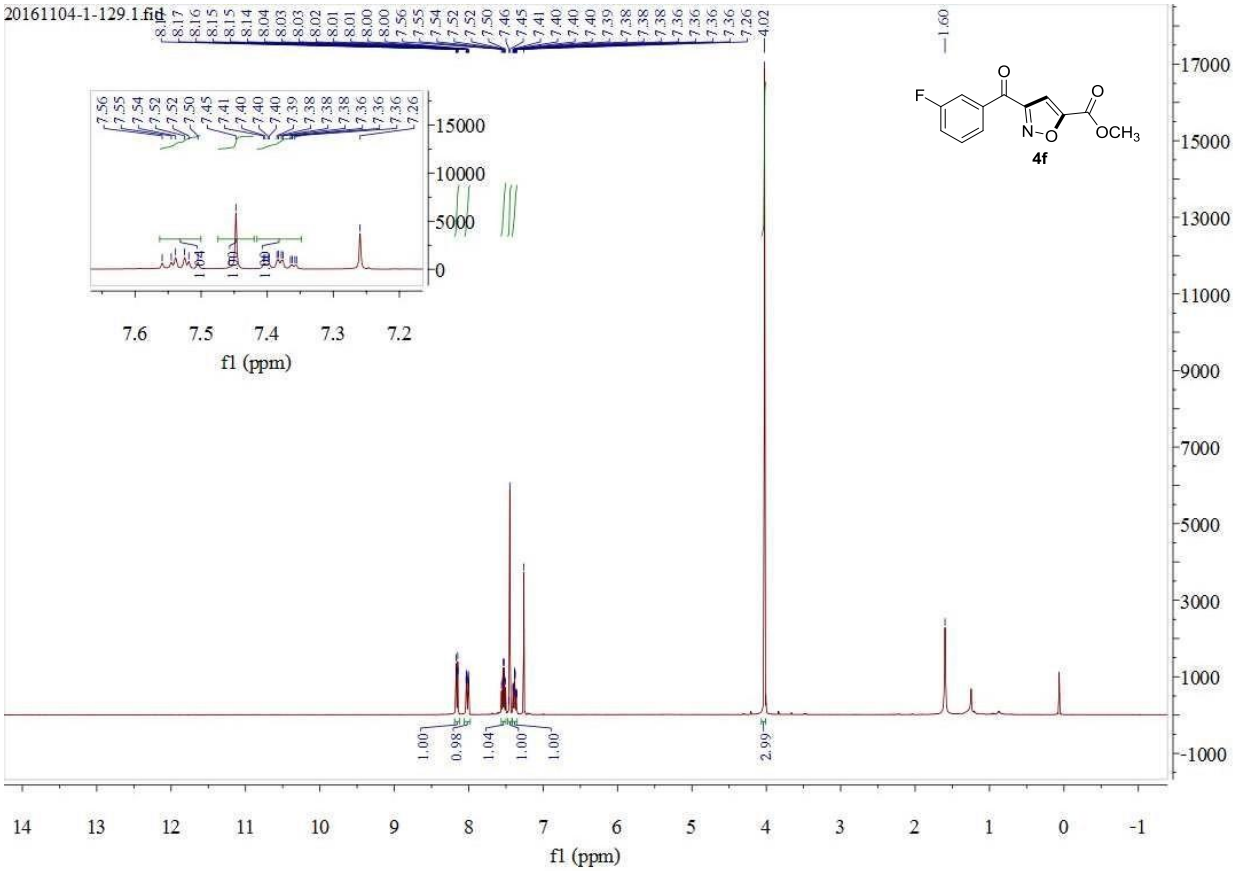
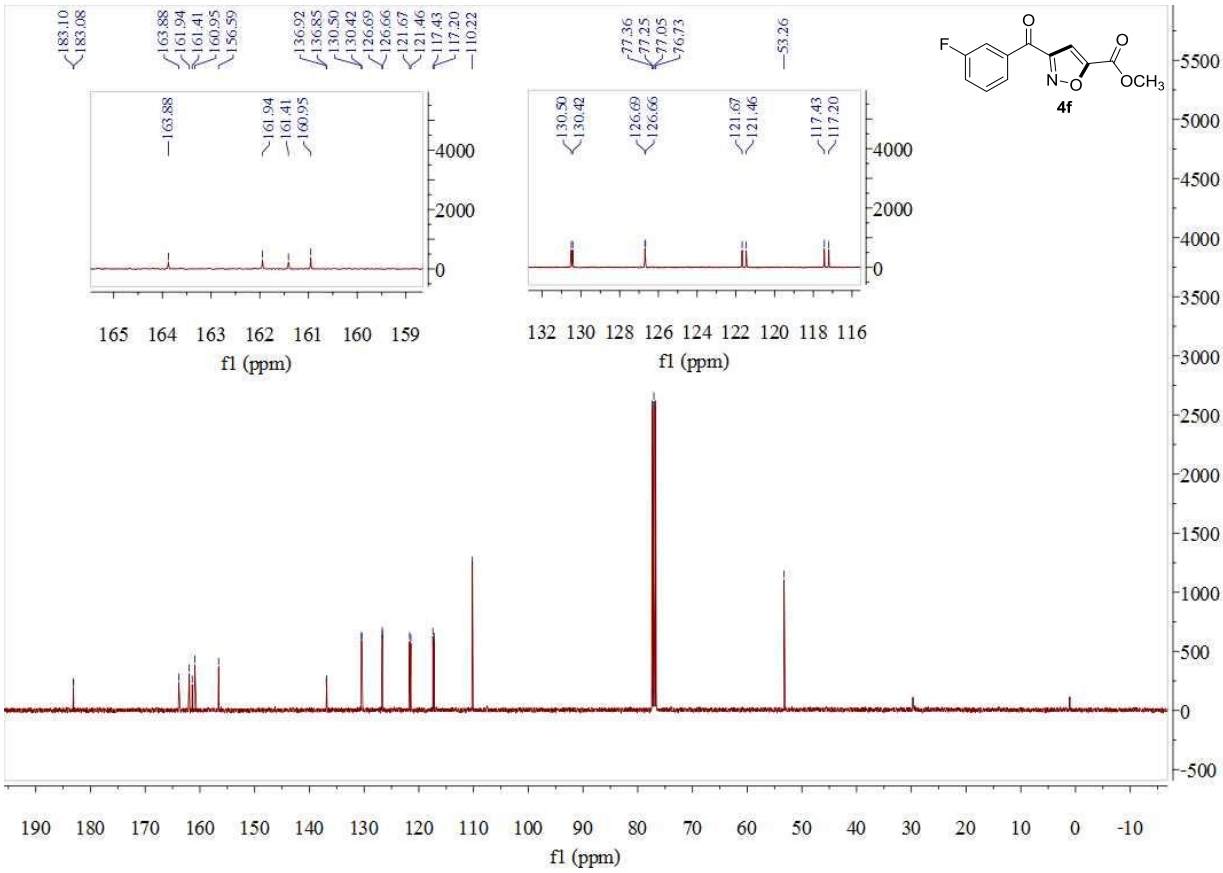
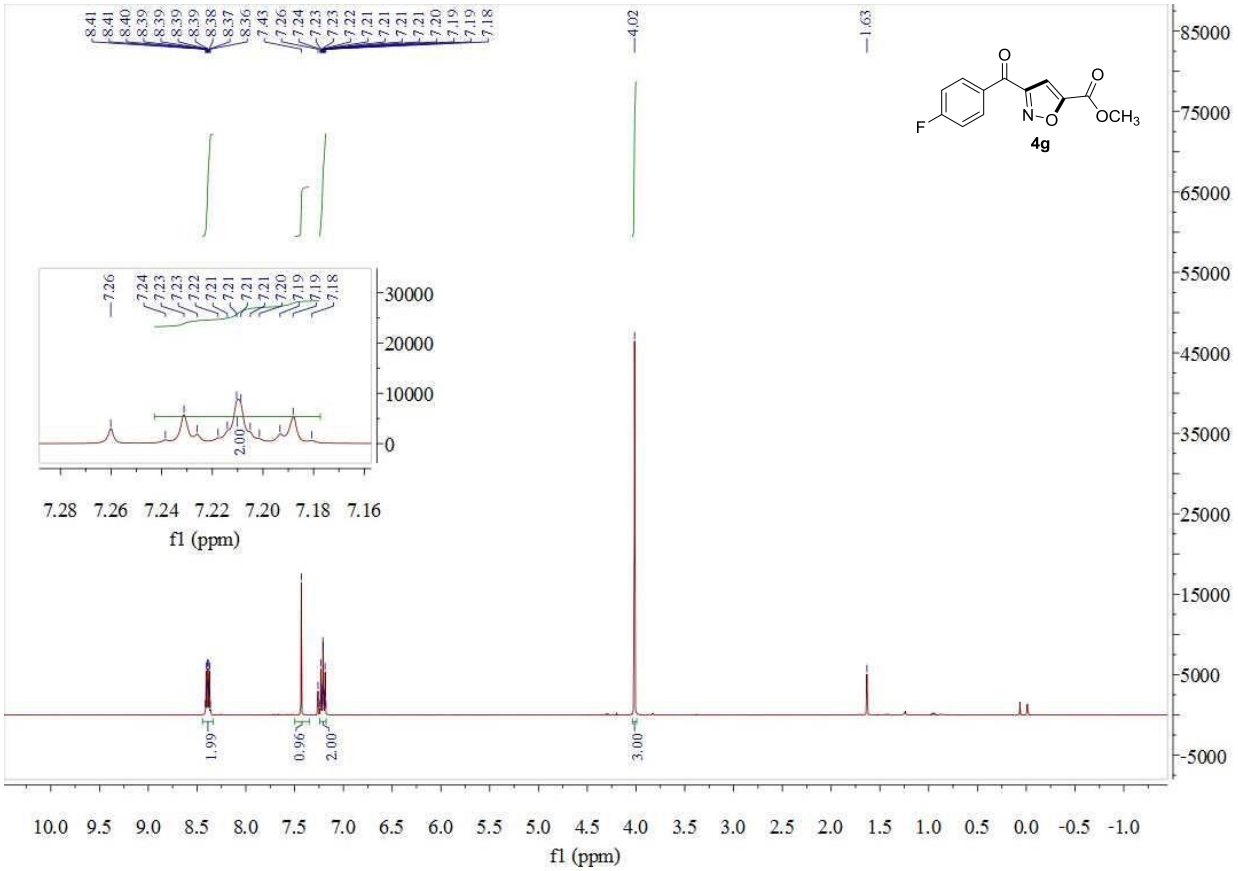
O
O
N
O
OCH3
H3CO
4d
f1 (ppm)

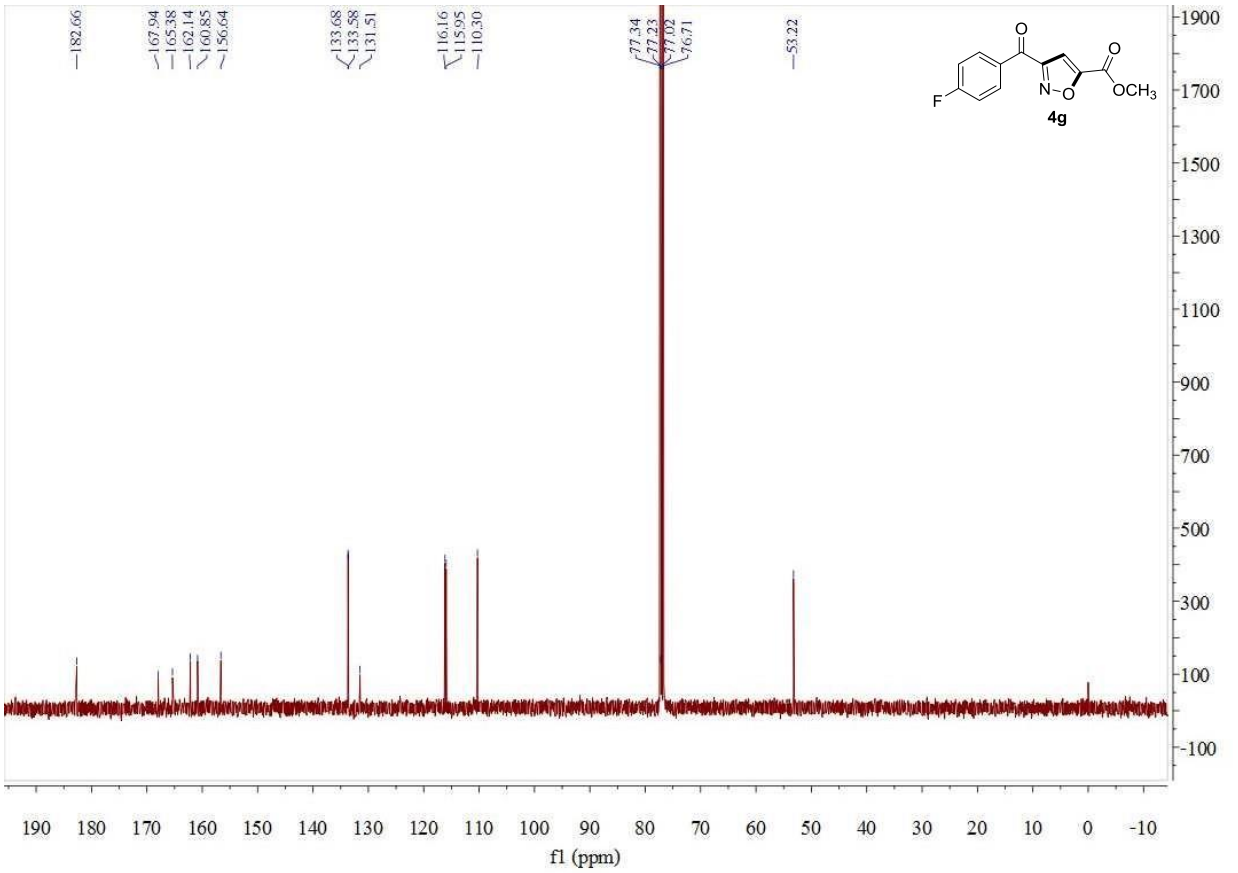

4e
f1 (ppm)

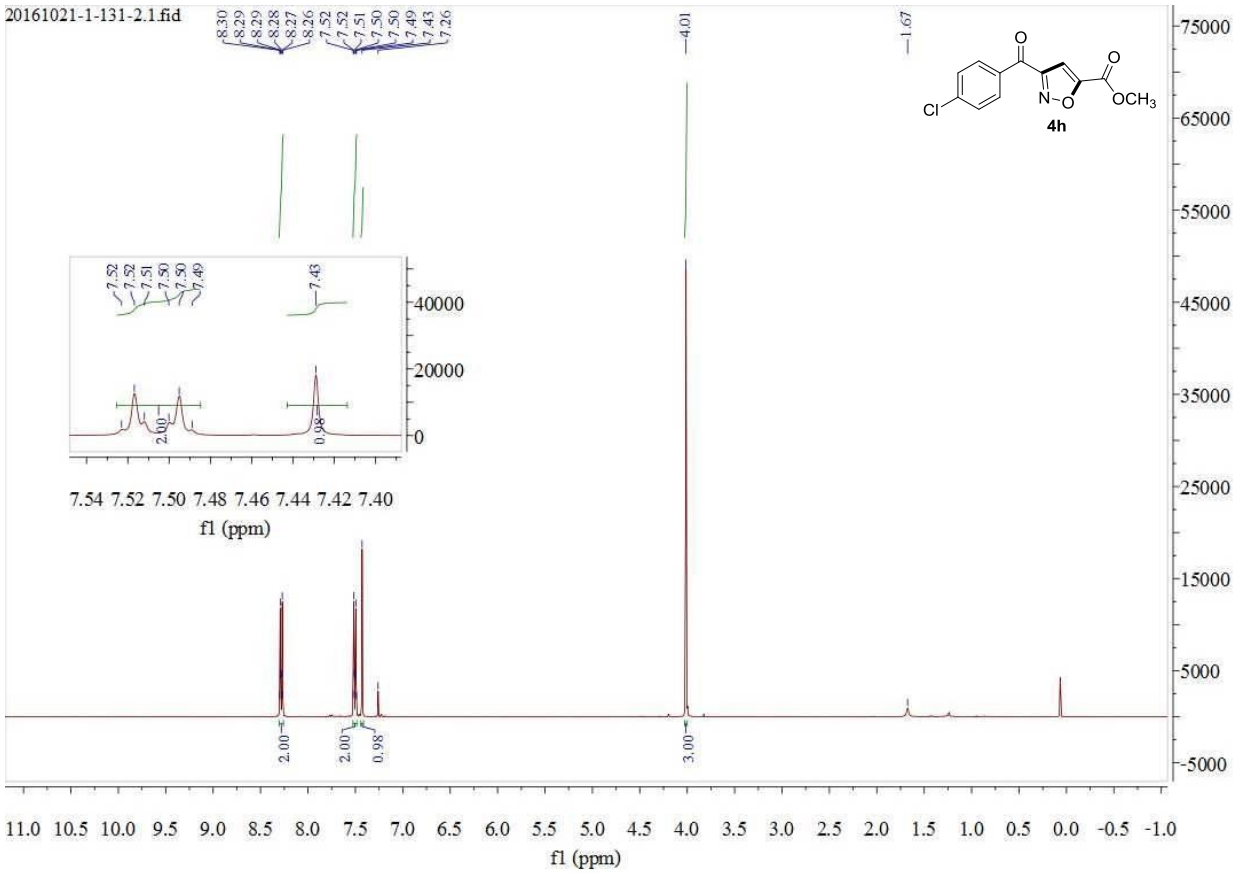

4e
f1 (ppm)

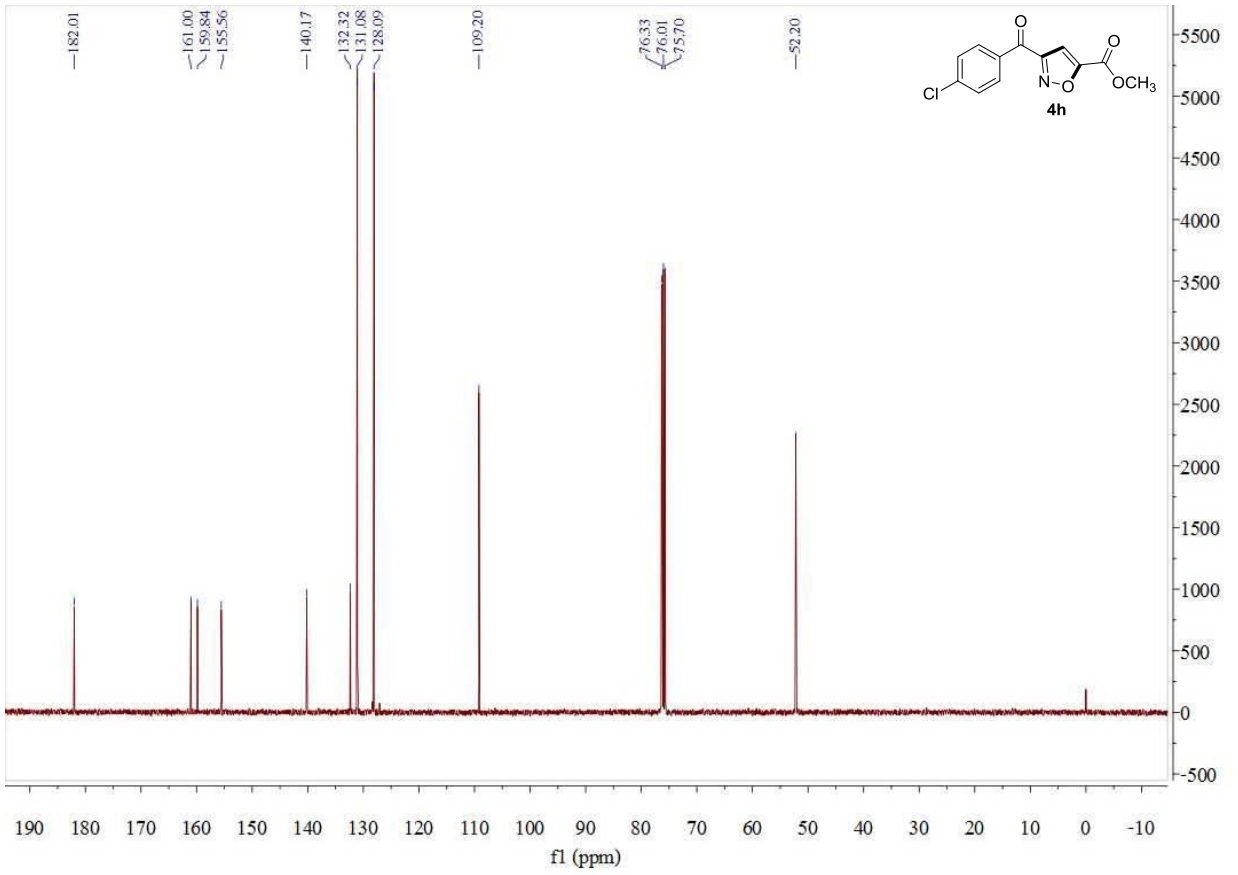

20161104-1-129.1.fid
4f
f1 (ppm)

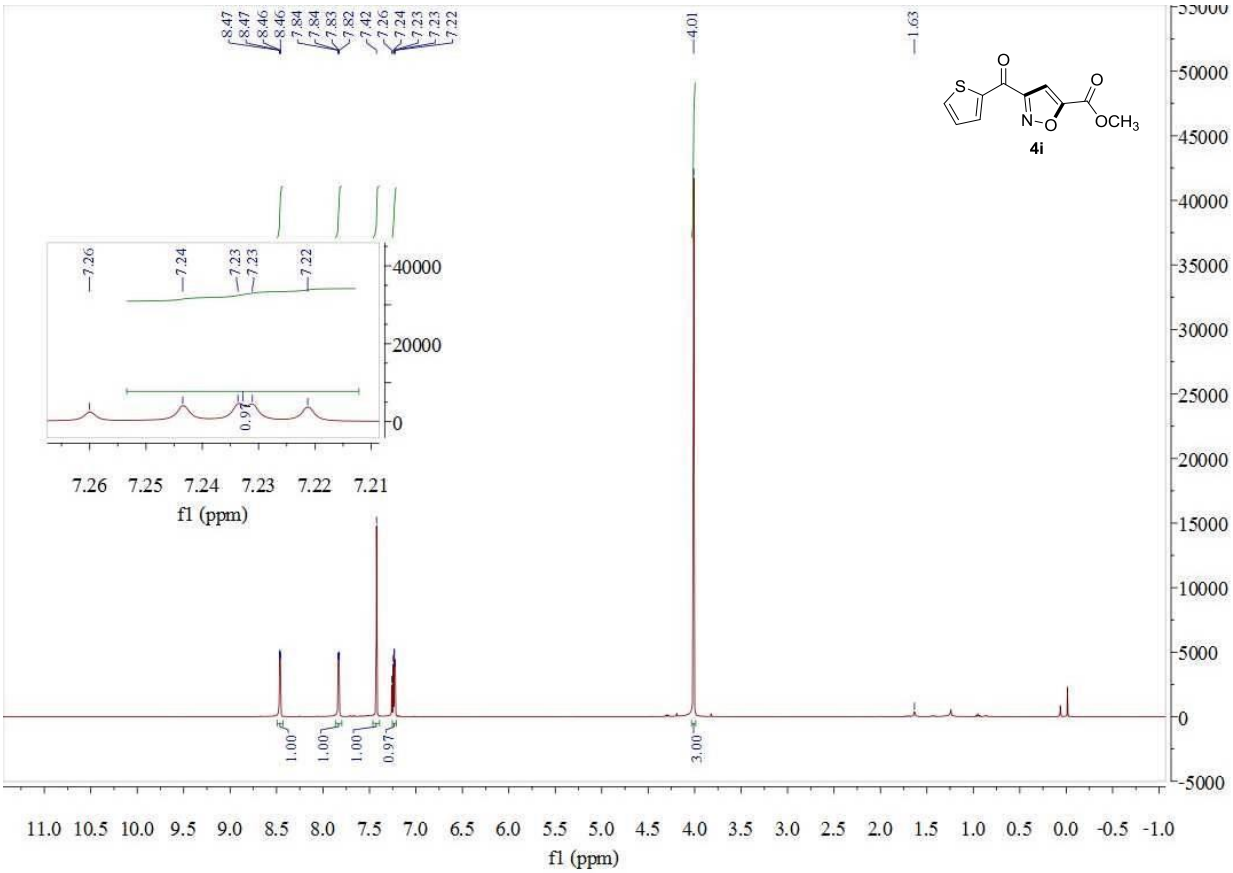

4f
f1 (ppm)

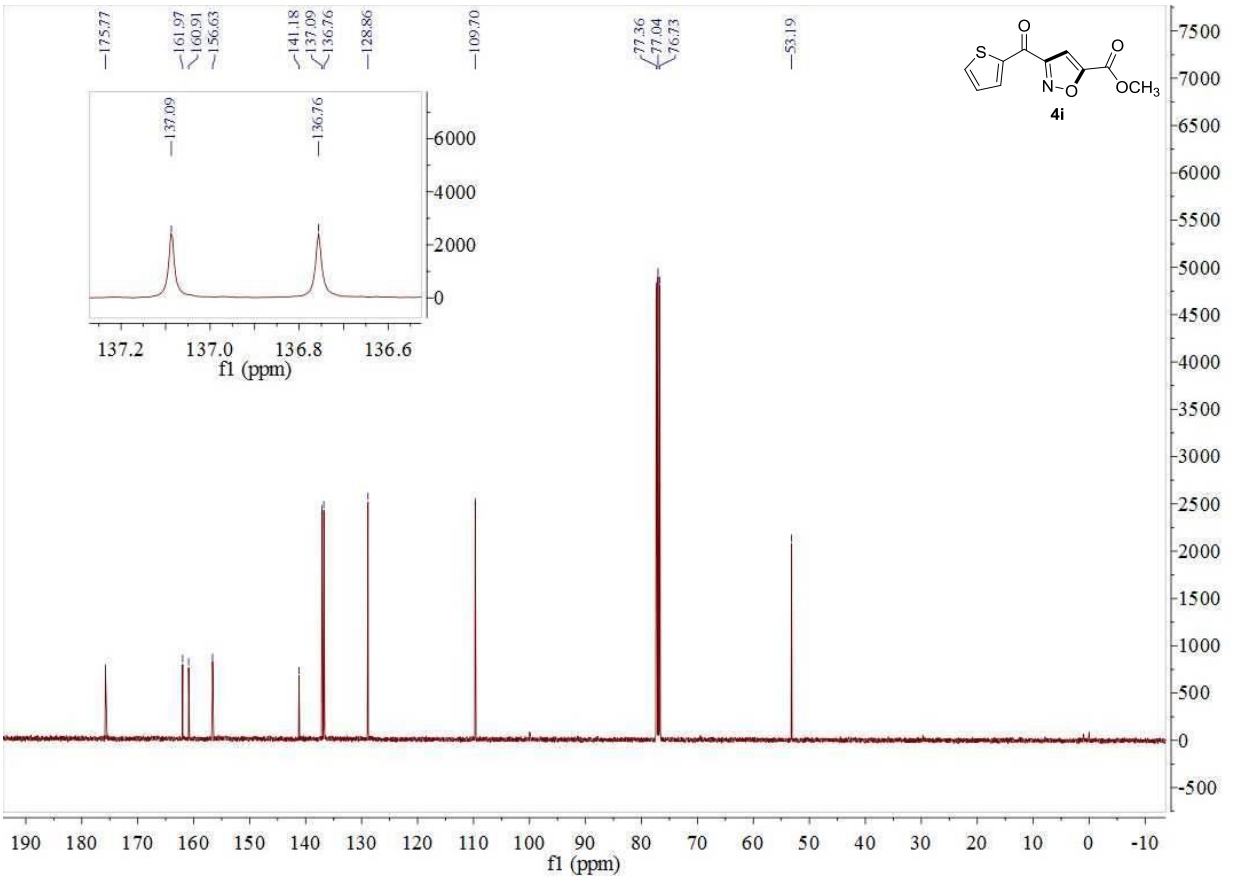

8.41 8.41 8.40 8.39 8.39 8.39 8.39 8.39 8.38 8.37 8.36
7.43 7.26 7.24 7.23 7.23 7.22 7.21 7.21 7.21 7.21 7.20 7.19 7.19 7.18
4.02
1.63
7.26 7.24 7.23 7.23 7.22 7.21 7.21 7.21 7.21 7.20 7.19 7.19 7.18
2.00
7.28 7.26 7.24 7.22 7.20 7.18 7.16
f1 (ppm)
1.99
0.96
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 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 -0.5 -1.0
f1 (ppm)
4g

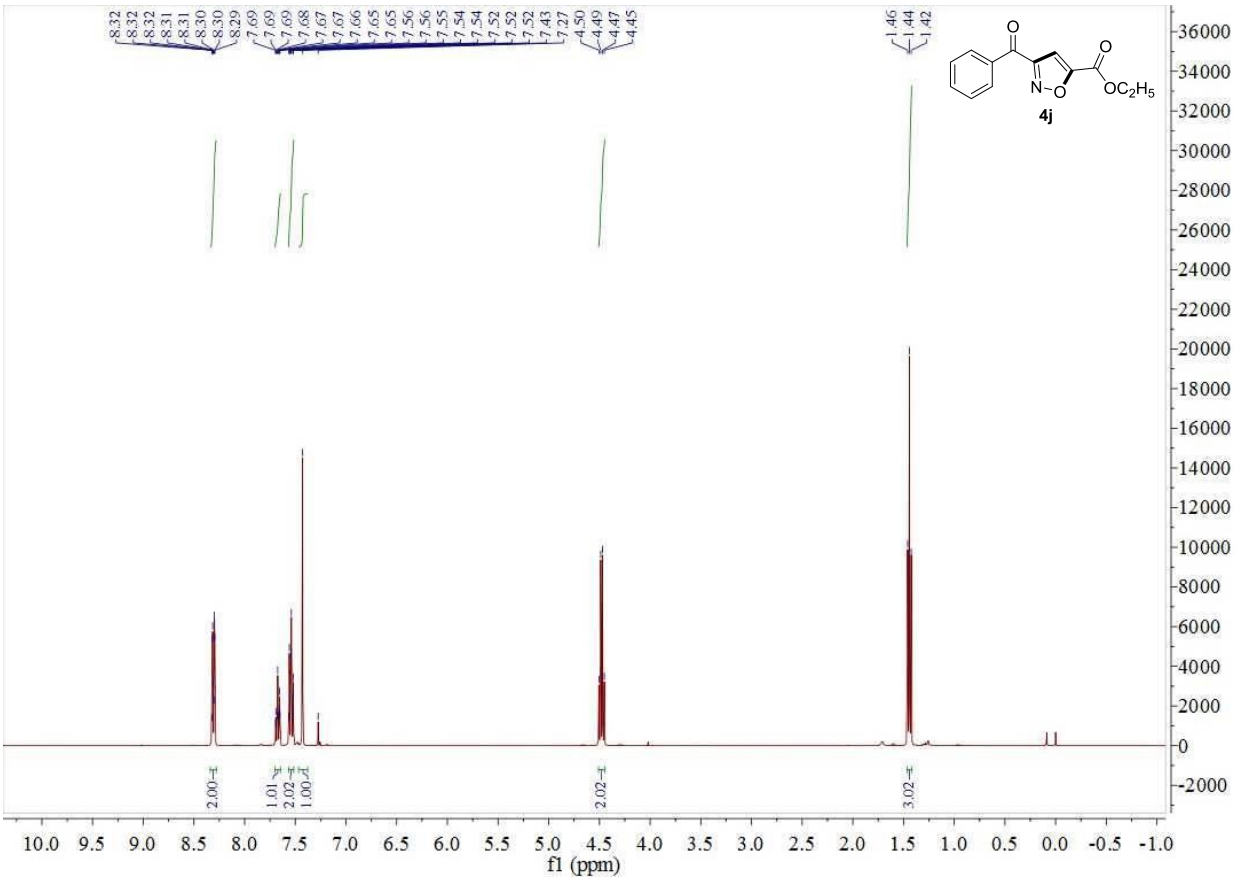

182.66
167.94 165.38 162.14 160.85 156.64
133.68 133.58 131.51
116.16 115.95 110.30
77.34 77.23 77.02 76.71
53.22
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
4g

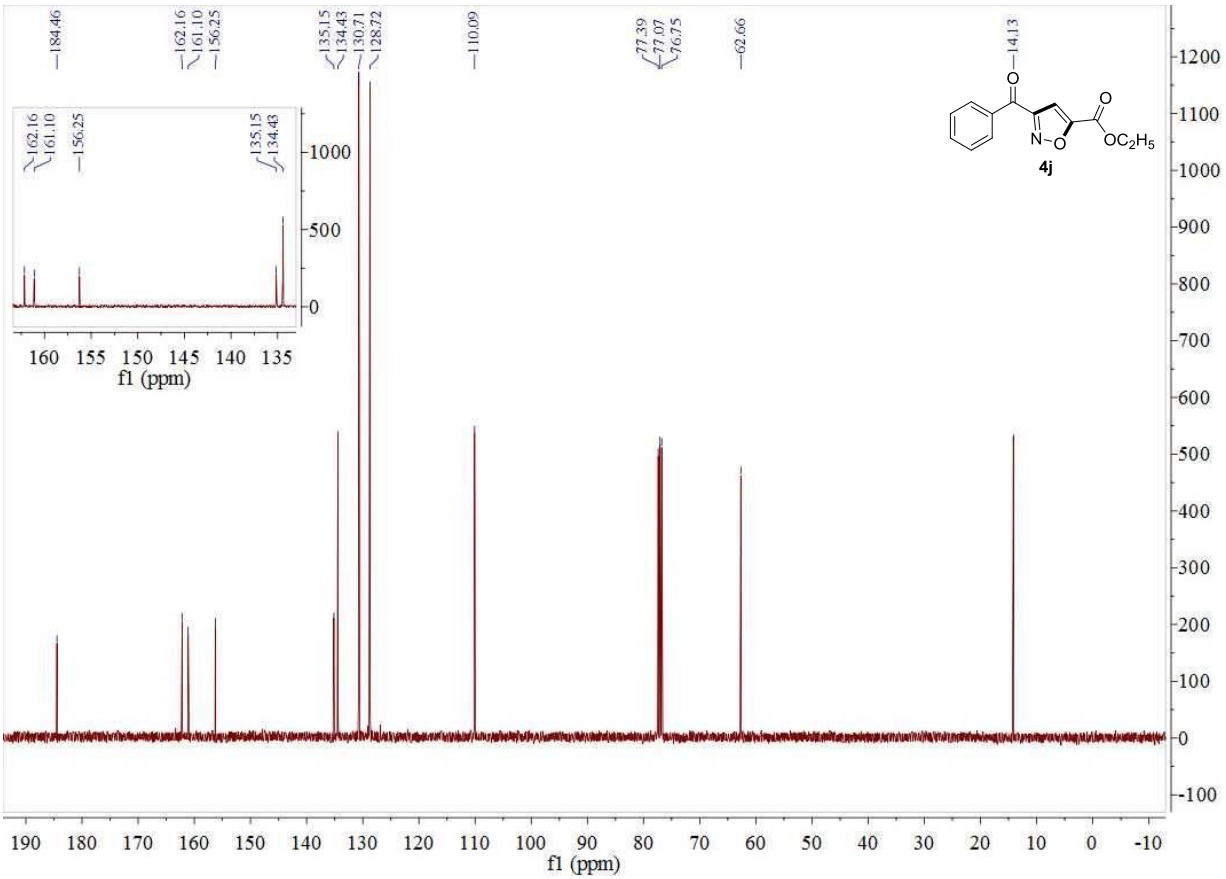

20161021-1-131-2.1.fid
8.30
8.29
8.29
8.28
8.27
8.26
7.52
7.52
7.51
7.50
7.50
7.49
7.43
7.26
4.01
1.67
7.52
7.52
7.51
7.50
7.50
7.49
7.43
2.00
0.98
7.54 7.52 7.50 7.48 7.46 7.44 7.42 7.40
f1 (ppm)
2.00
2.00
0.98
3.00
Cl
N
O
O
O
OCH3
4h
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

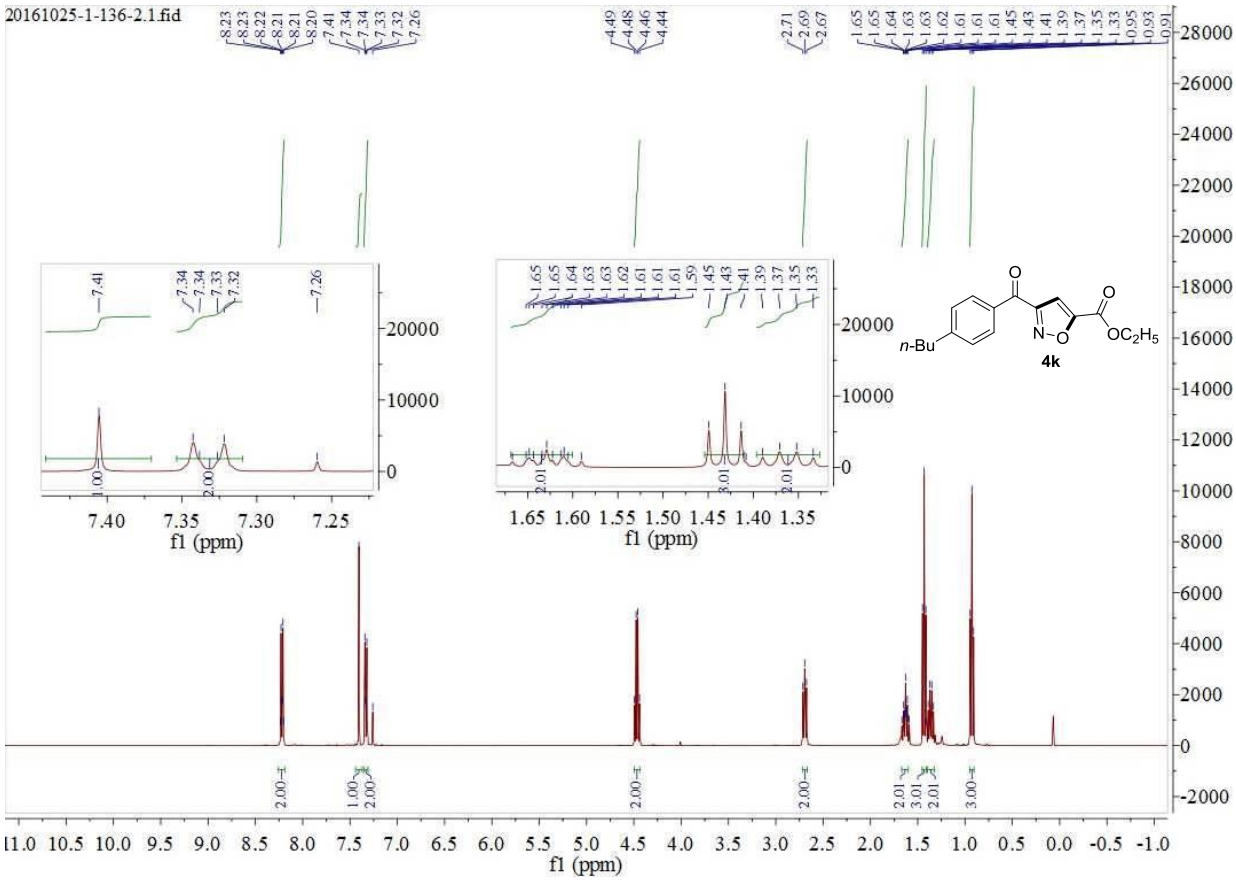

182.01
161.00
159.84
155.56
140.17
132.32
131.08
128.09
109.20
76.33
76.01
75.70
52.20
Cl
N
O
O
O
OCH3
4h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

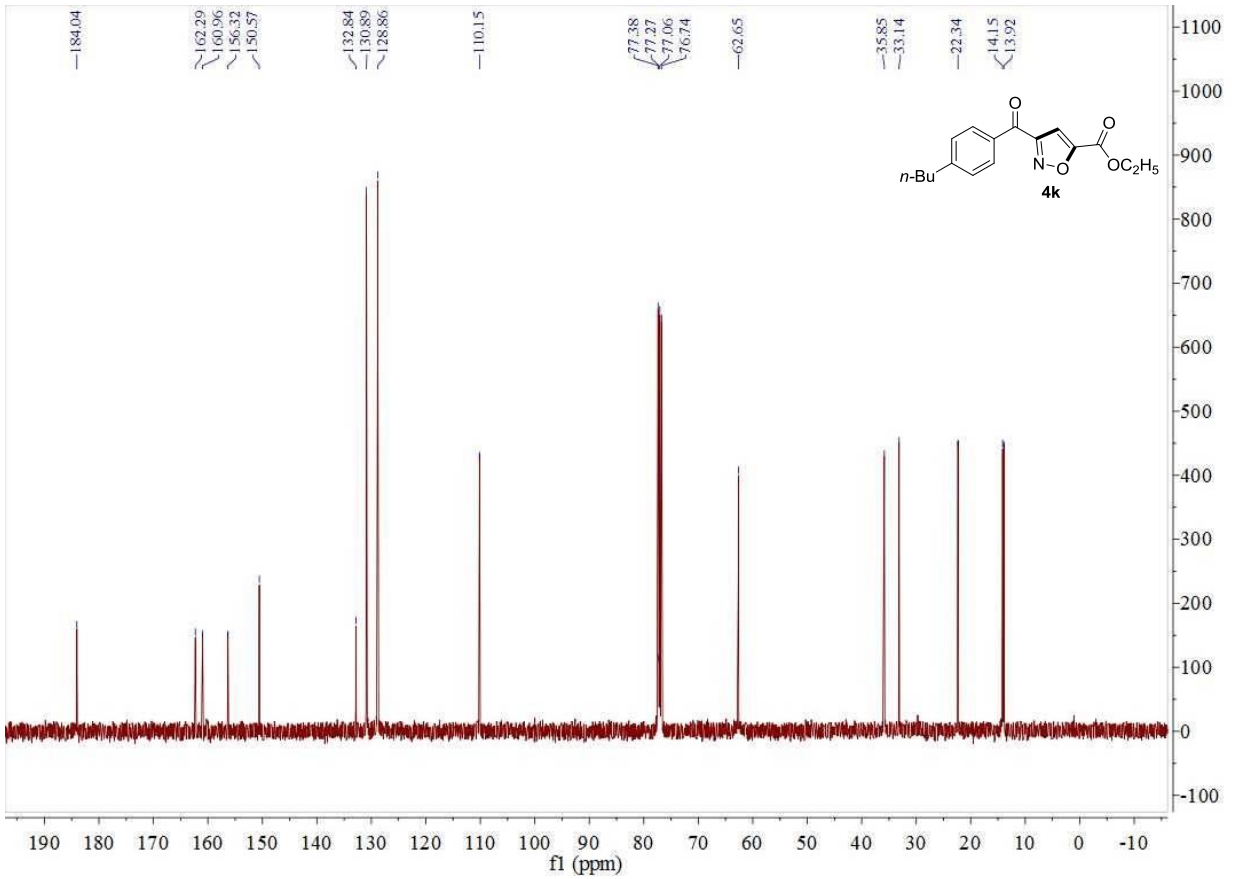

8.47
8.47
8.46
8.46
7.84
7.84
7.83
7.82
7.42
7.26
7.24
7.23
7.23
7.22
4.01
1.63
7.26
7.24
7.23
7.23
7.22
0.97
1.00
1.00
1.00
0.97
3.00
4i
OCH3
7.26 7.25 7.24 7.23 7.22 7.21
f1 (ppm)
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

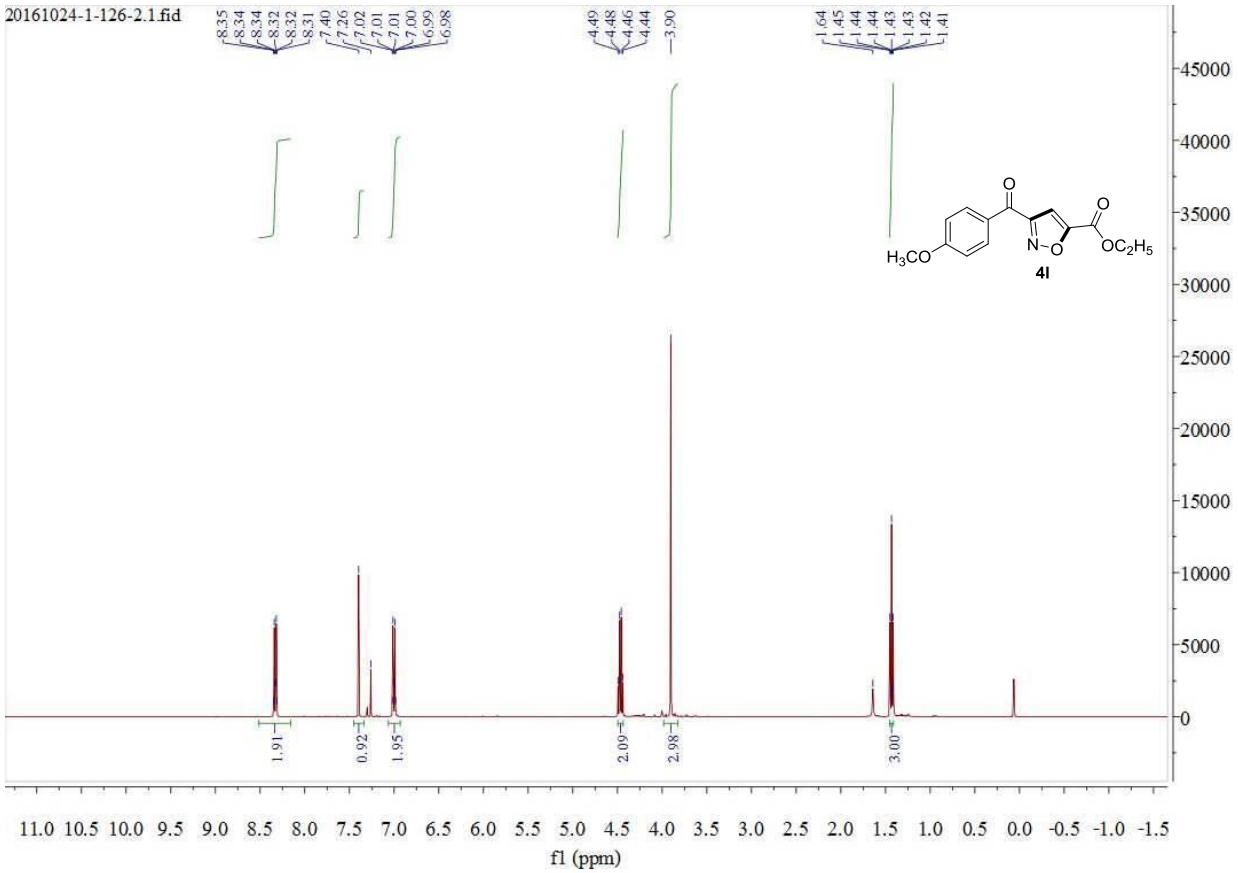

175.77
161.97
160.91
156.63
141.18
137.09
136.76
128.86
109.70
77.36
77.04
76.73
53.19
137.09
136.76
4i
OCH3
137.2 137.0 136.8 136.6
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.32
8.32
8.32
8.31
8.31
8.30
8.30
8.29
7.69
7.69
7.69
7.68
7.67
7.67
7.67
7.66
7.65
7.65
7.56
7.56
7.55
7.54
7.54
7.52
7.52
7.52
7.43
7.27
4.50
4.49
4.47
4.45
1.46
1.44
1.42
4j
2.00
1.01
2.02
1.00
2.02
3.02
f1 (ppm)

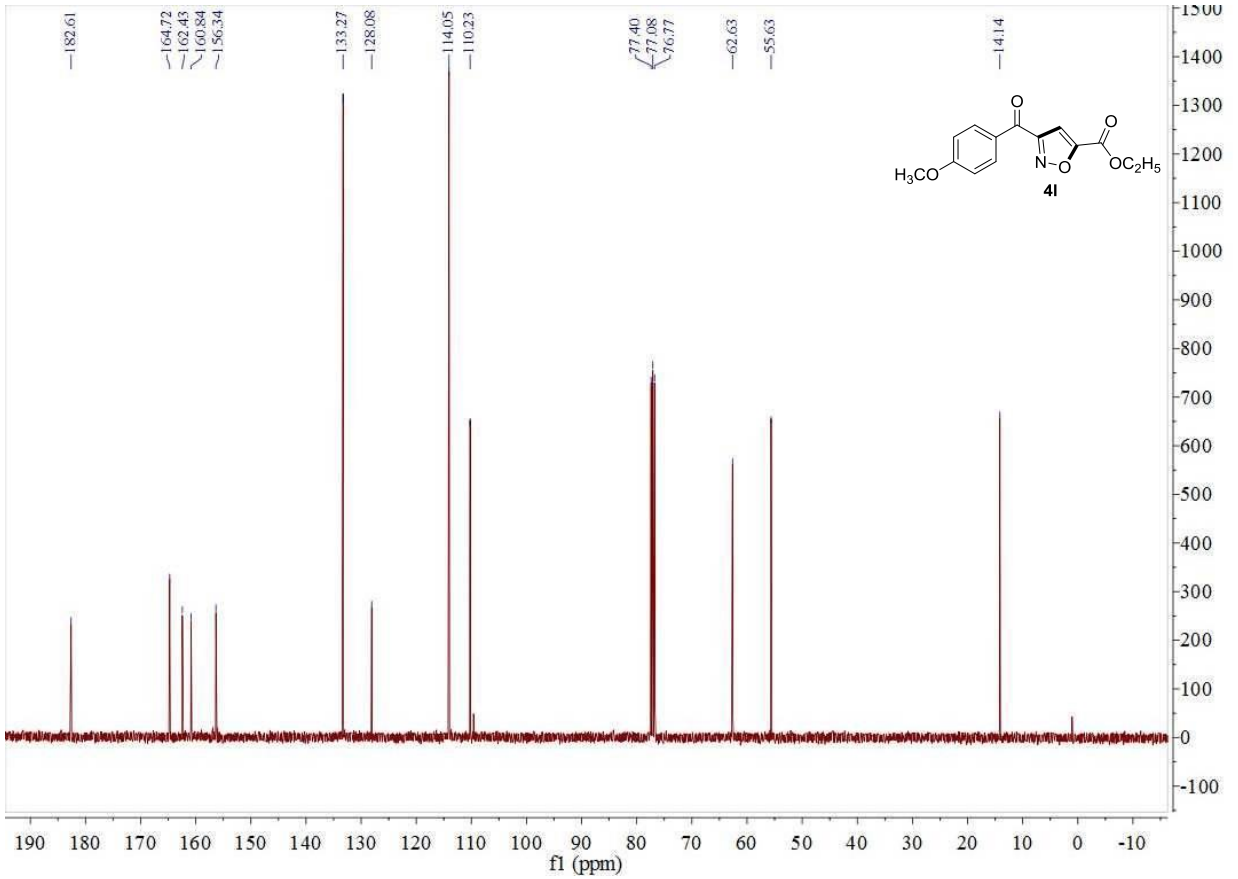

184.46
162.16
161.10
156.25
135.15
134.43
130.71
128.72
110.09
77.39
77.07
76.75
62.66
14.13
4j
f1 (ppm)


20161025-1-136-2.1.fid
8.23 8.23 8.22 8.21 8.21 8.20 7.41 7.34 7.34 7.33 7.33 7.32 7.26
4.49 4.48 4.46 4.44
2.71 2.69 2.67
1.65 1.65 1.64 1.63 1.63 1.62 1.61 1.61 1.61 1.45 1.43 1.41 1.39 1.37 1.35 1.33 0.95 0.93 0.91
7.41 7.34 7.34 7.33 7.32 7.26
1.65 1.65 1.64 1.63 1.63 1.62 1.61 1.61 1.61 1.59 1.45 1.43 1.41 1.39 1.37 1.35 1.33
7.40 7.35 7.30 7.25
f1 (ppm)
1.65 1.60 1.55 1.50 1.45 1.40 1.35
f1 (ppm)
n-Bu
N
O
O
O
OC2H5
4k
2.00 1.00 2.00 2.00 2.00 2.01 3.01 2.01 3.00
f1 (ppm)

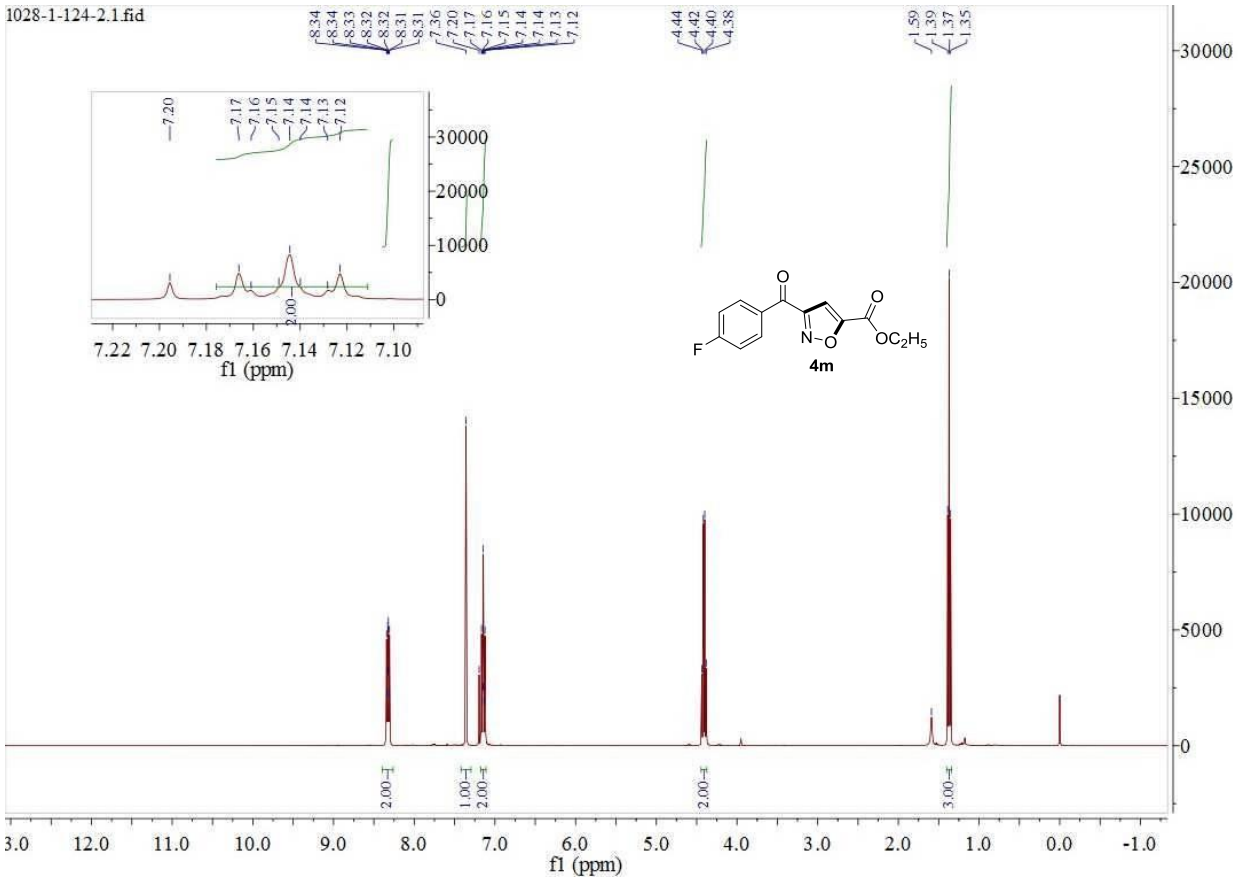

184.04
162.29 160.96 156.32 150.57
132.84 130.89 128.86
110.15
77.38 77.27 77.06 76.74
62.65
35.85 33.14
22.34
14.15 13.92
n-Bu
N
O
O
O
OC2H5
4k
f1 (ppm)


20161024-1-126-2.1.fid
8.35
8.34
8.34
8.32
8.32
8.31
7.40
7.26
7.02
7.01
7.01
7.00
6.99
6.98
4.49
4.48
4.46
4.44
3.90
1.64
1.45
1.44
1.44
1.43
1.43
1.42
1.41
1.91
0.92
1.95
2.09
2.98
3.00
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 -0.5 -1.0 -1.5
f1 (ppm)

O
O
N
O
H_3CO
OC_2H_5
4l


182.61
164.72
162.43
160.84
156.34
133.27
128.08
114.05
110.23
77.40
77.08
76.77
62.63
55.63
14.14
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

O
O
N
O
H_3CO
OC_2H_5
4l


1028-1-124-2.1.fid
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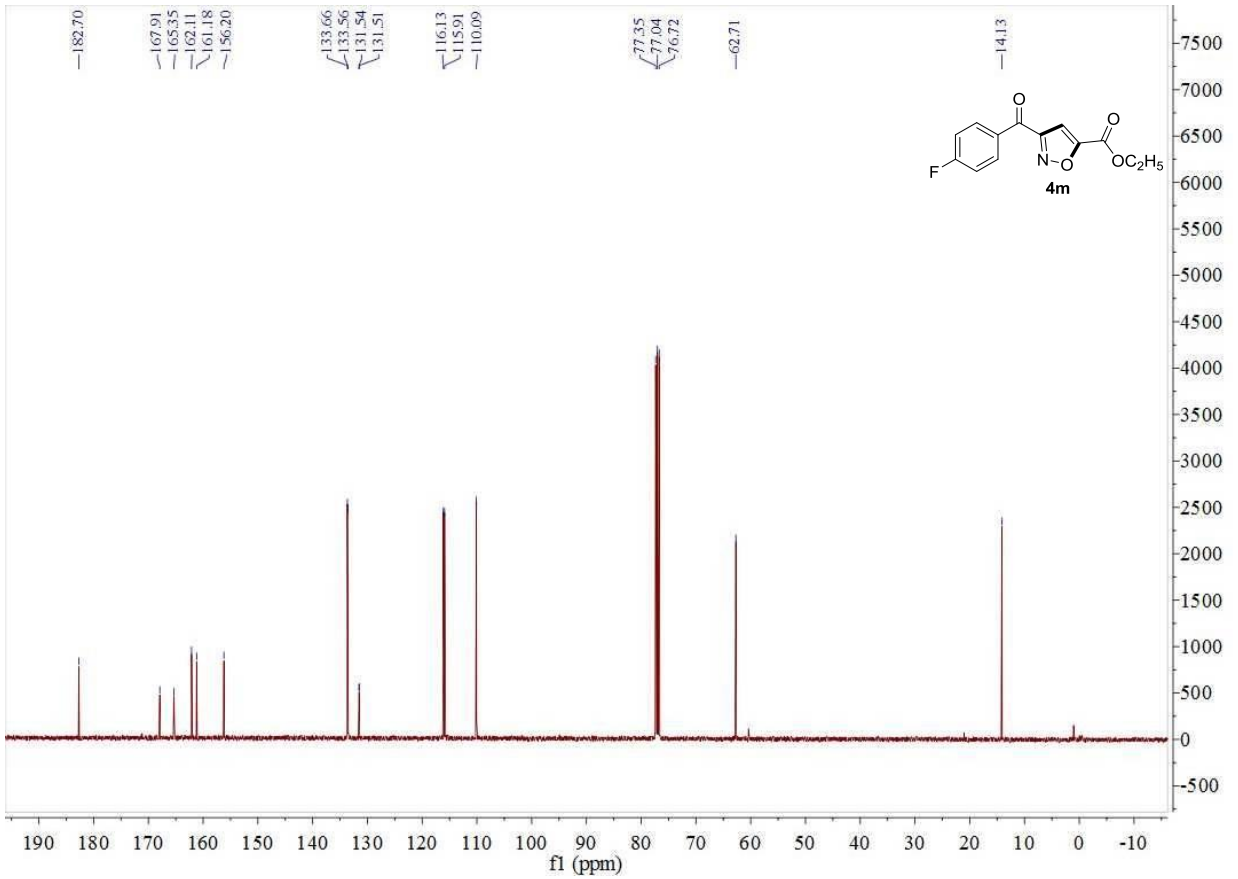
8.33
8.32
8.32
8.31
8.31
7.36
7.20
7.17
7.16
7.15
7.14
7.14
7.13
7.12
4.44
4.42
4.40
4.38
1.59
1.39
1.37
1.35
7.20
7.17
7.16
7.15
7.14
7.14
7.13
7.12
2.00
7.22 7.20 7.18 7.16 7.14 7.12 7.10
f1 (ppm)
O
O
F
N
O
OC2H5
4m
2.00
1.00
2.00
2.00
3.00
f1 (ppm)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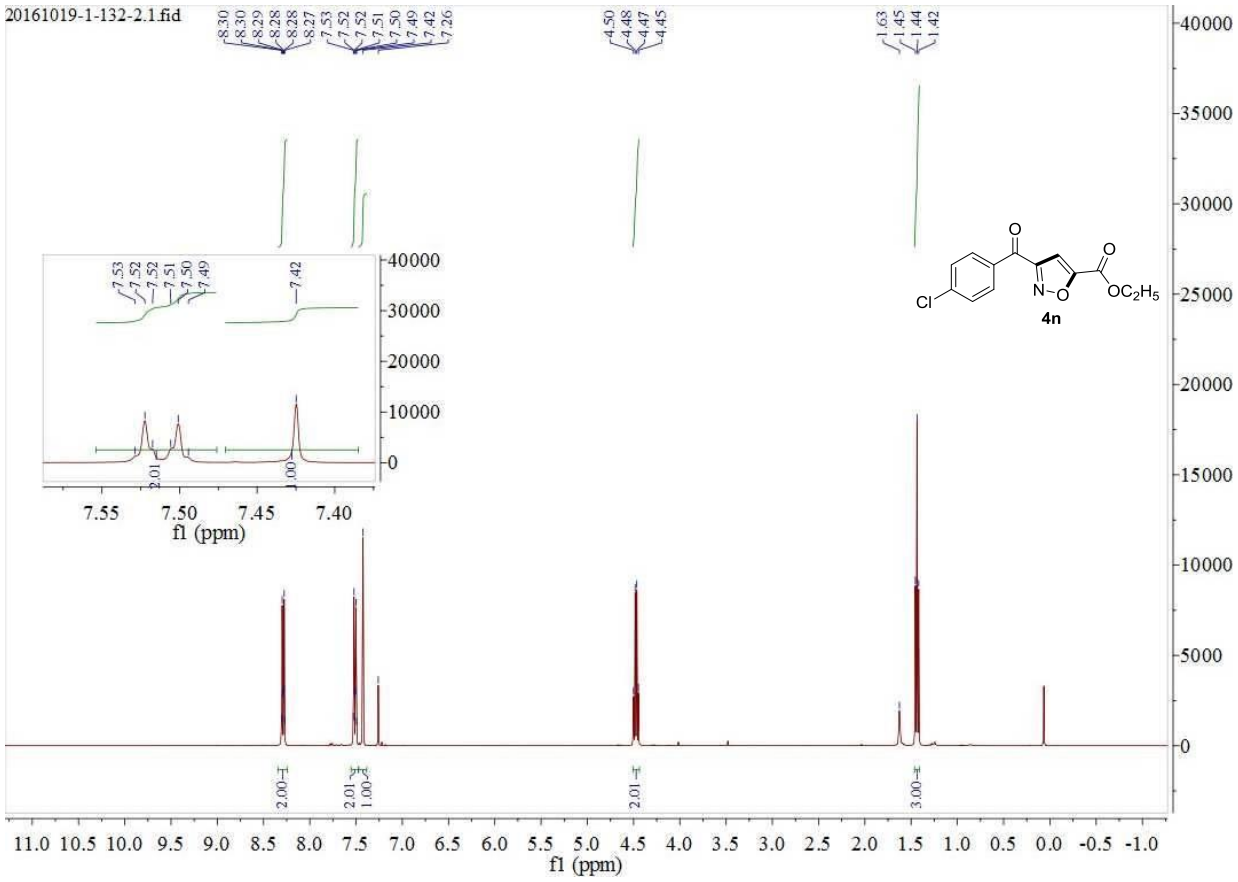
167.91
165.35
162.11
161.18
156.20
133.66
133.56
131.54
131.51
116.13
115.91
110.09
77.35
77.04
76.72
62.71
14.13
O
O
F
N
O
OC2H5
4m
f1 (ppm)


20161019-1-132-2.1.fid
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8.28
8.27
7.53
7.52
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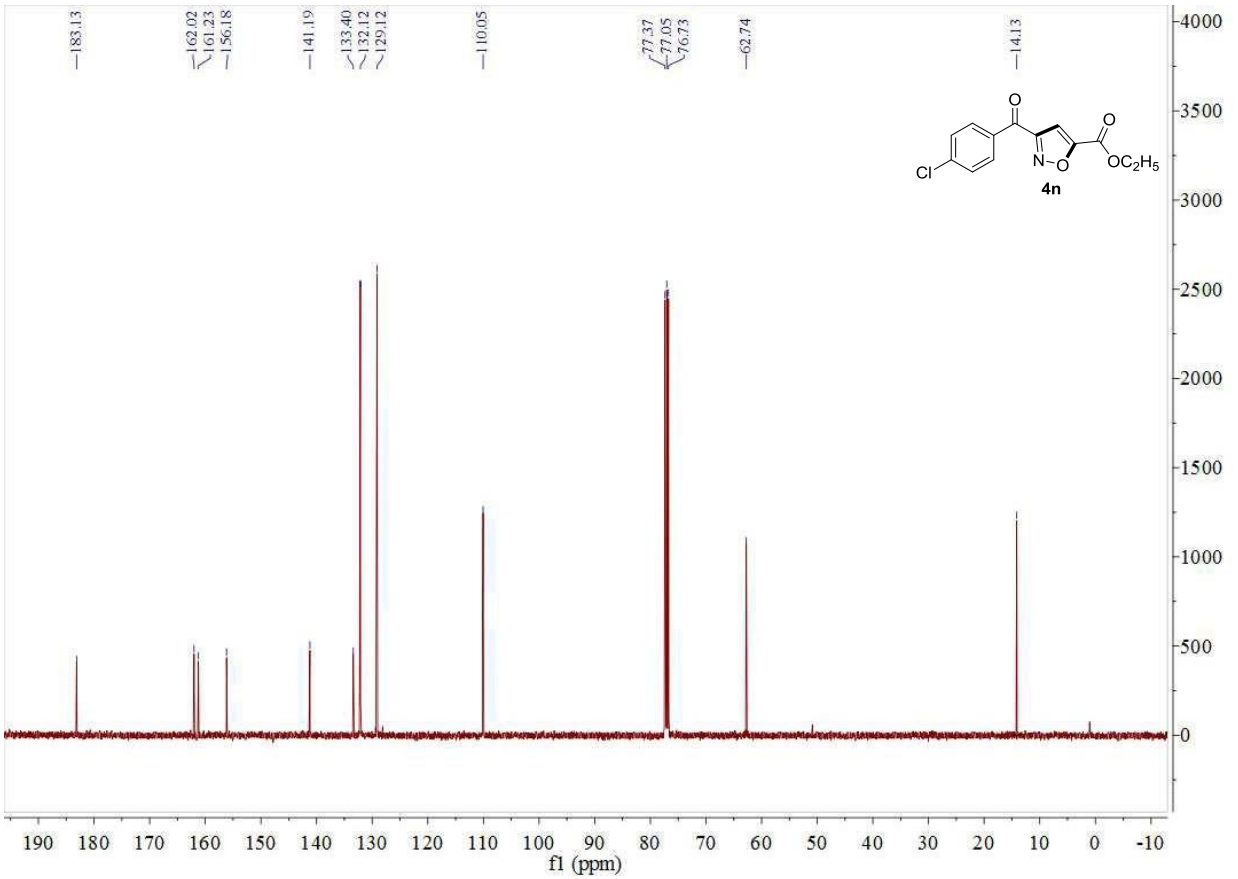
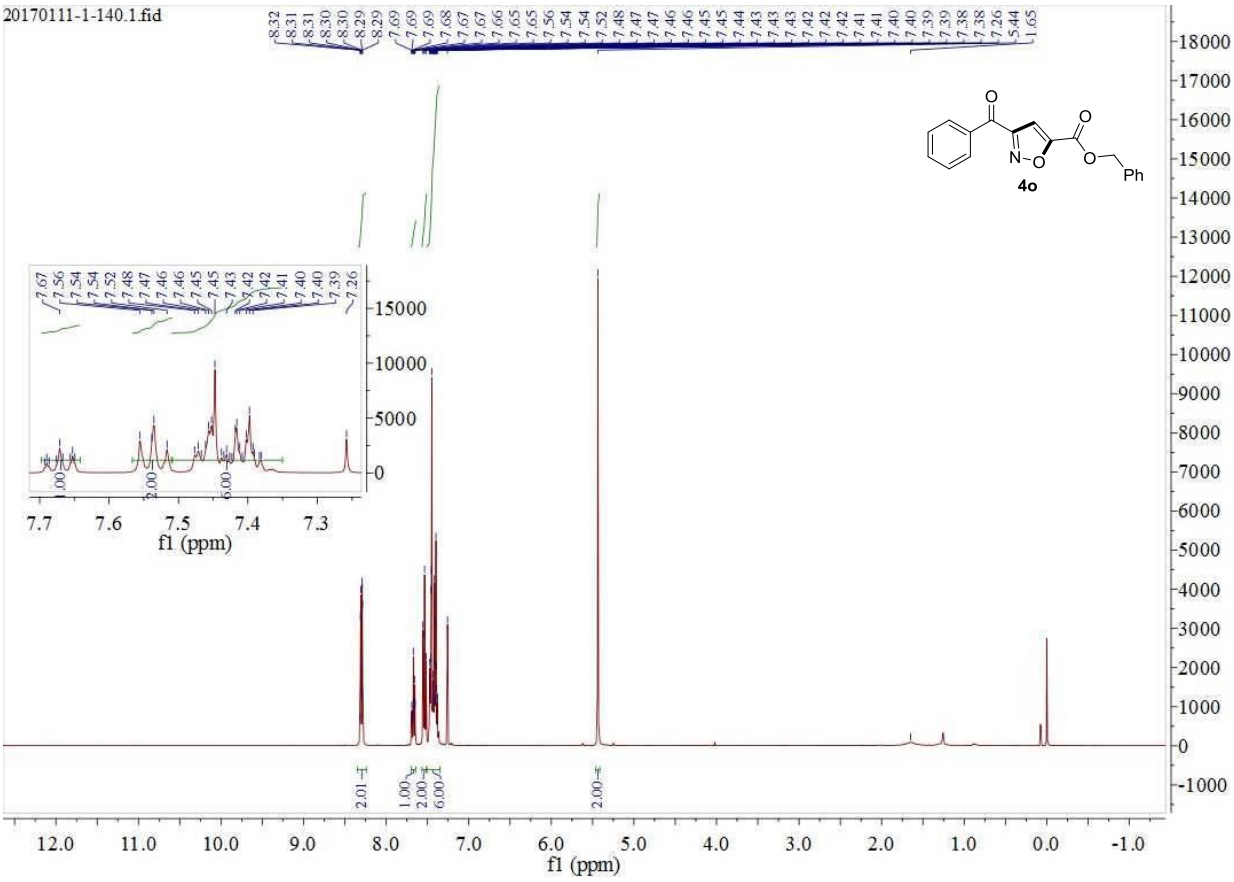
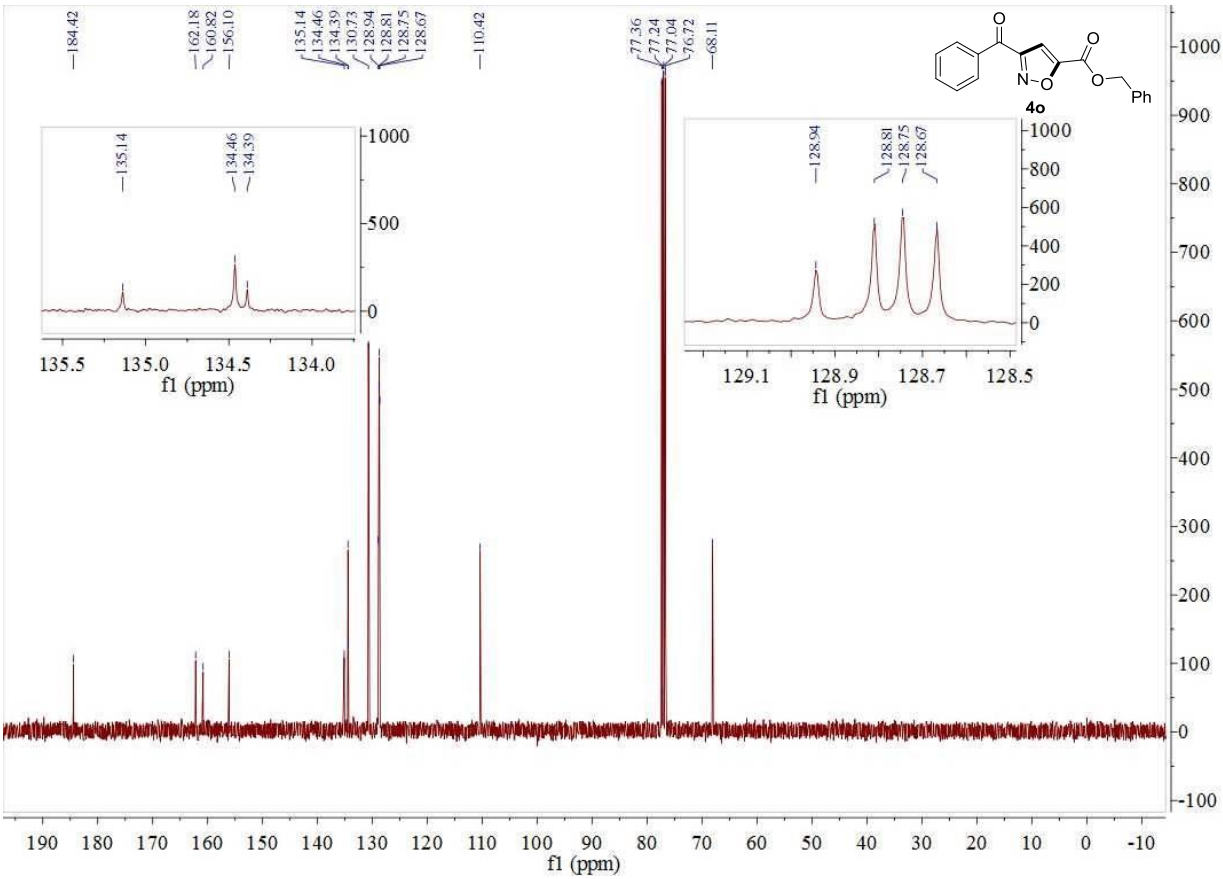
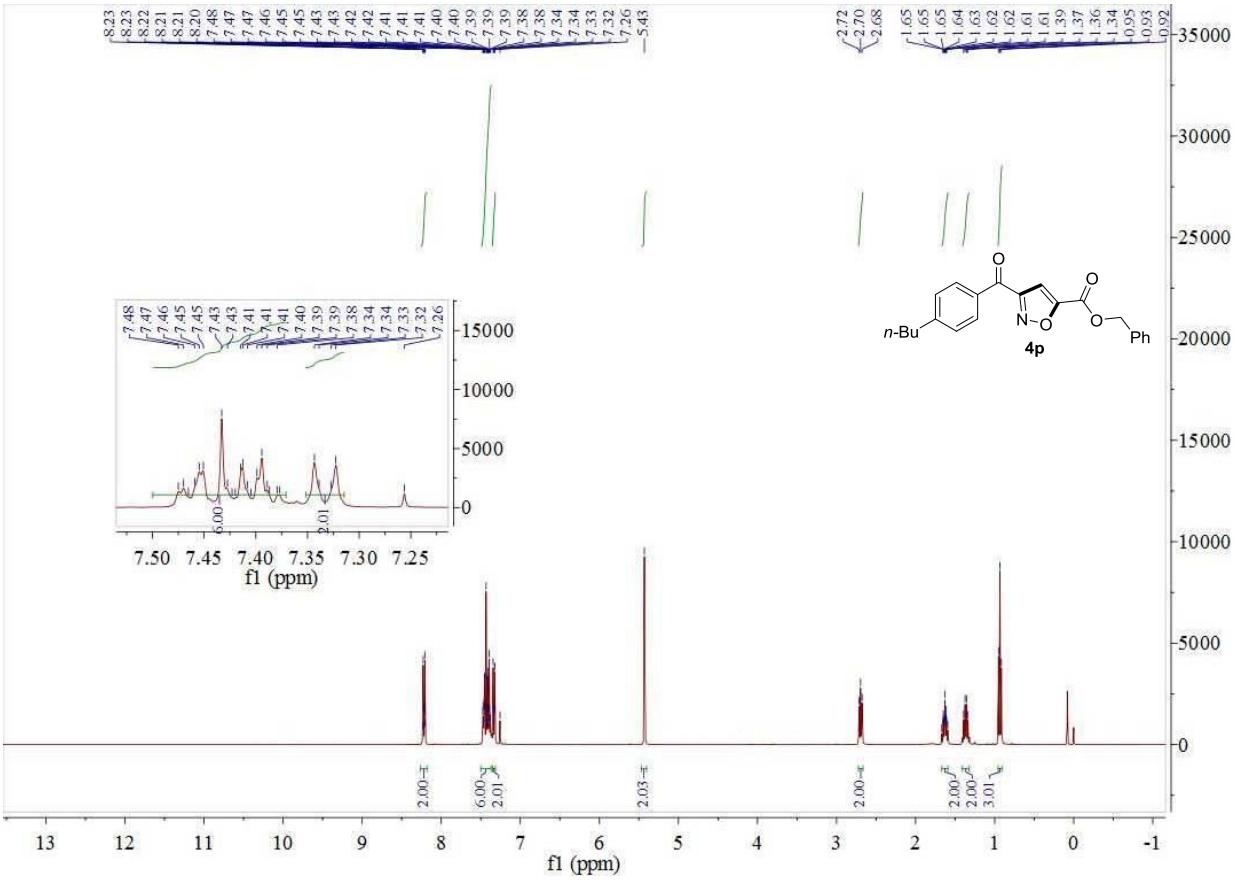
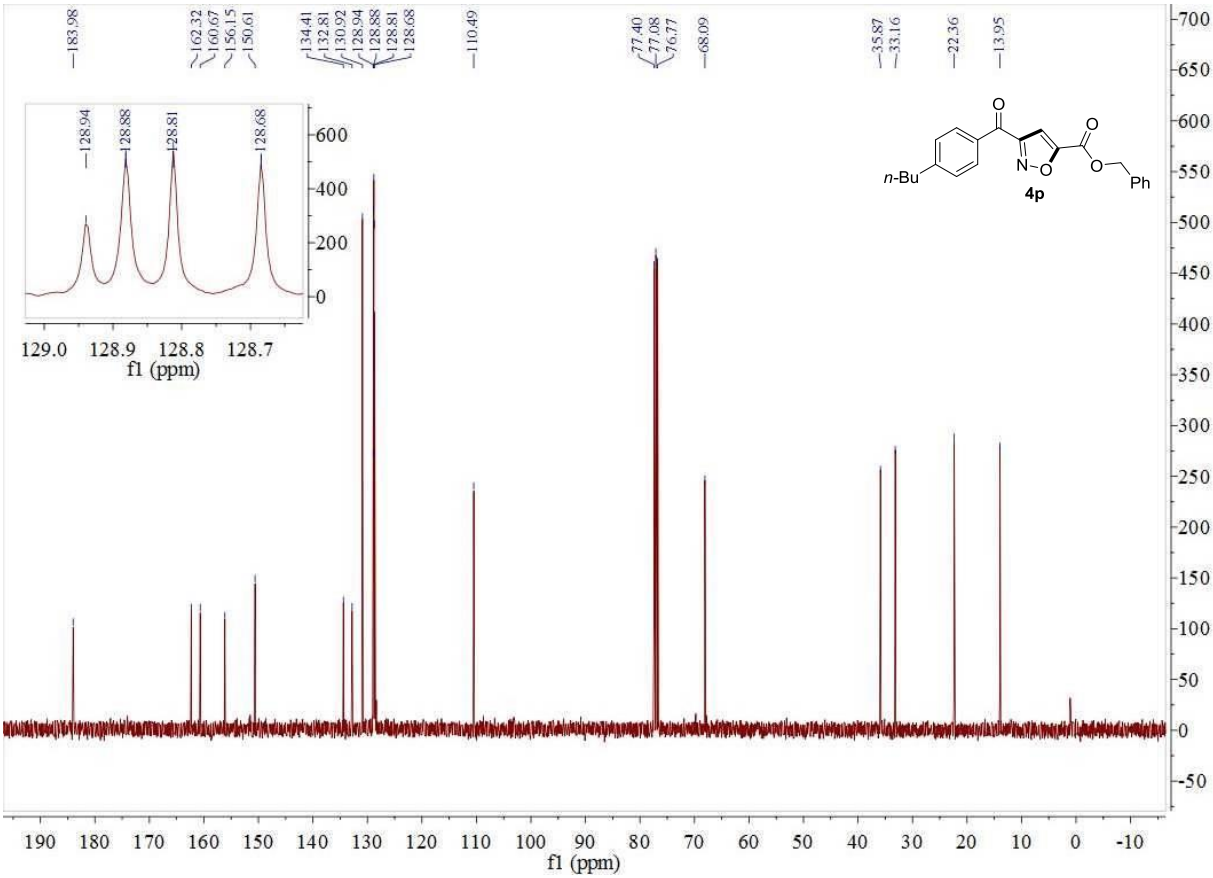
7.51
7.50
7.49
7.42
7.26
4.50
4.48
4.47
4.45
1.63
1.45
1.44
1.42
7.53
7.52
7.52
7.51
7.50
7.49
7.42
2.01
1.00
7.55
7.50
7.45
7.40
f1 (ppm)
2.00
2.01
1.00
2.01
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
Cl
N
O
O
O
OC2H5
4n

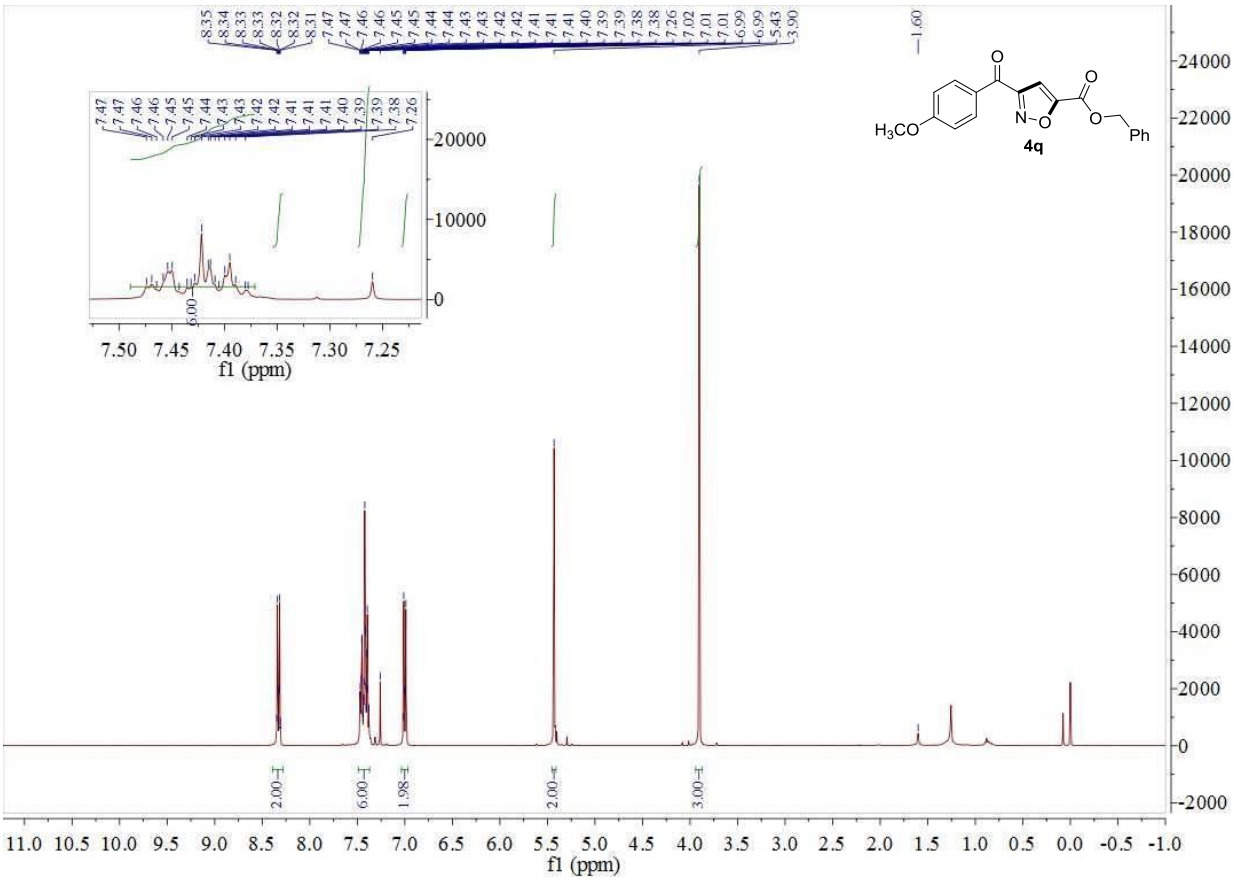

183.13
162.02
161.23
156.18
141.19
133.40
132.12
129.12
110.05
77.37
77.05
76.73
62.74
14.13
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
Cl
N
O
O
O
OC2H5
4n

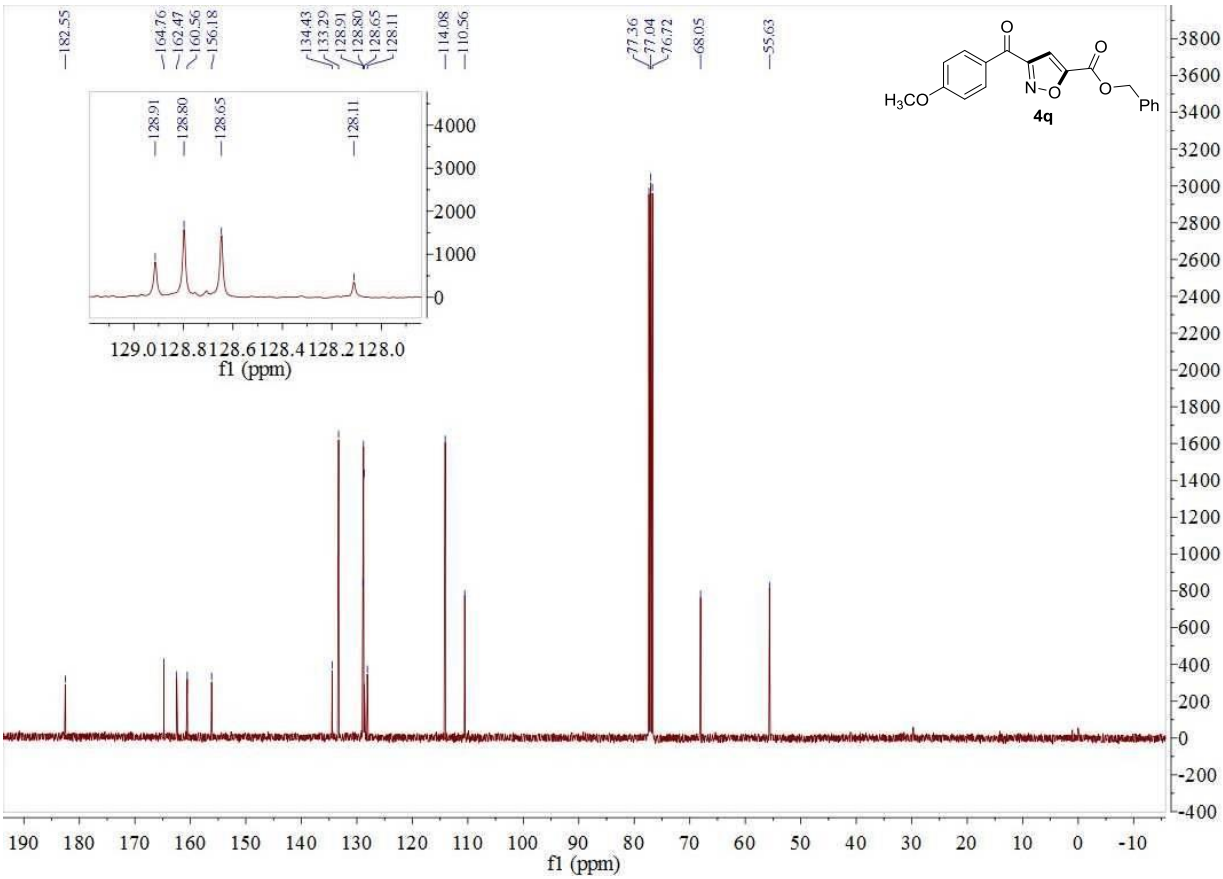

20170111-1-140.1.fid
4o
Ph
f1 (ppm)

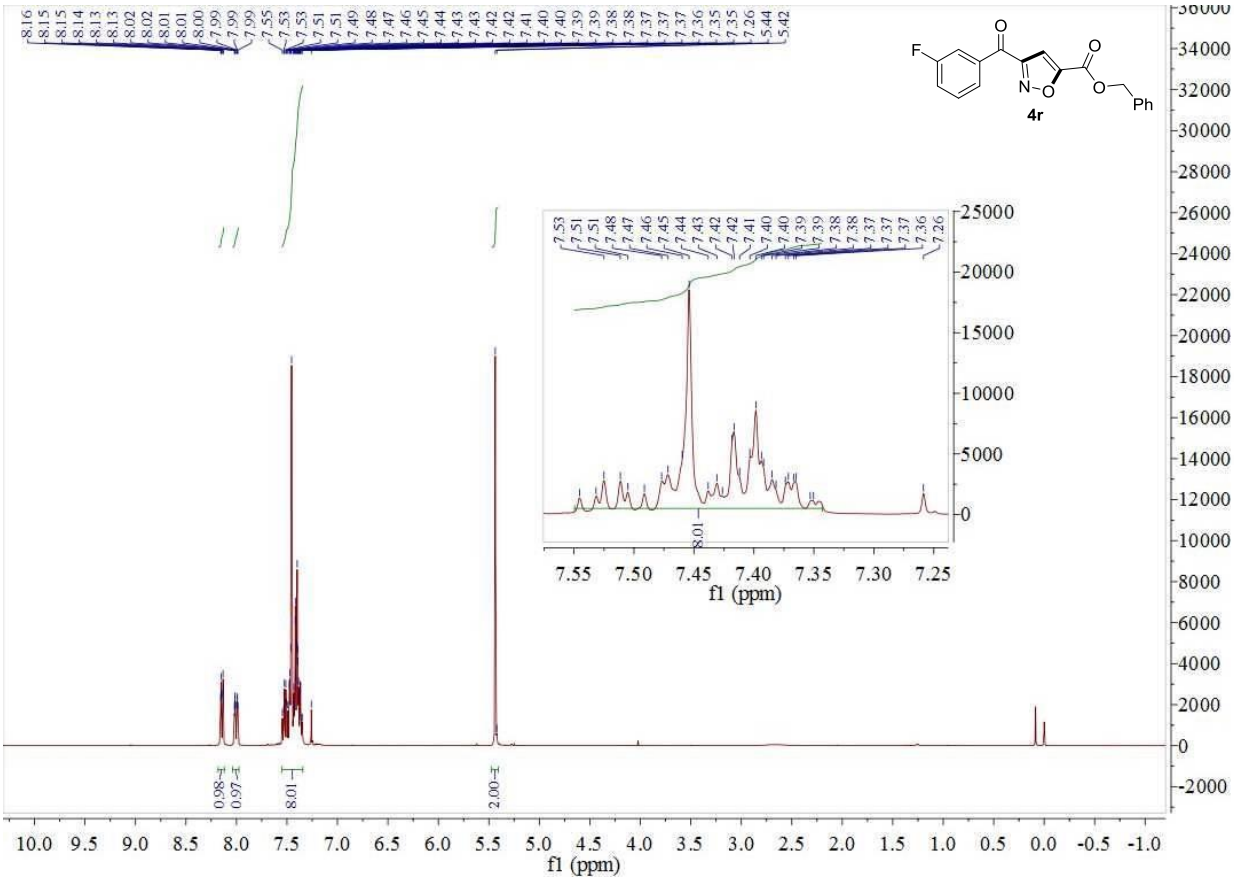

4o
Ph
f1 (ppm)

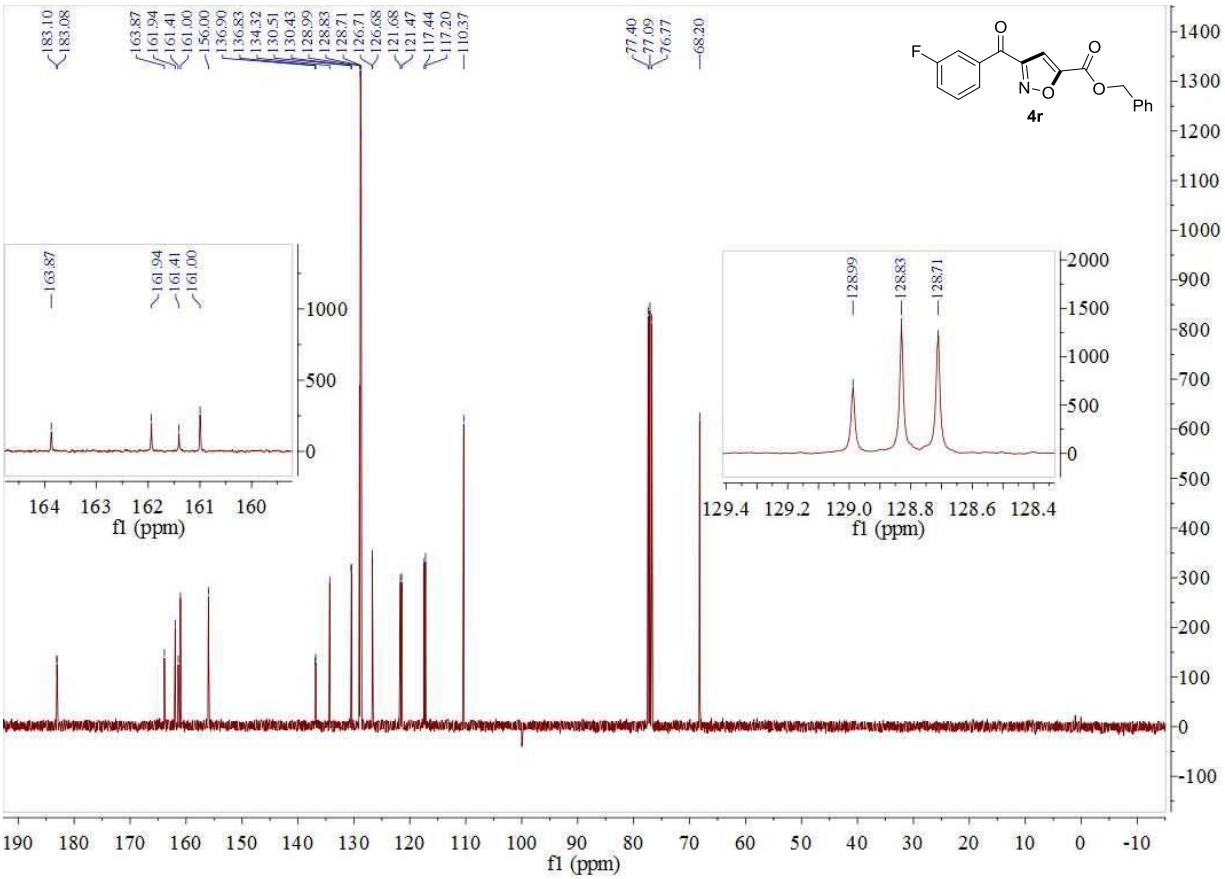

n-Bu
Ph
4p
f1 (ppm)

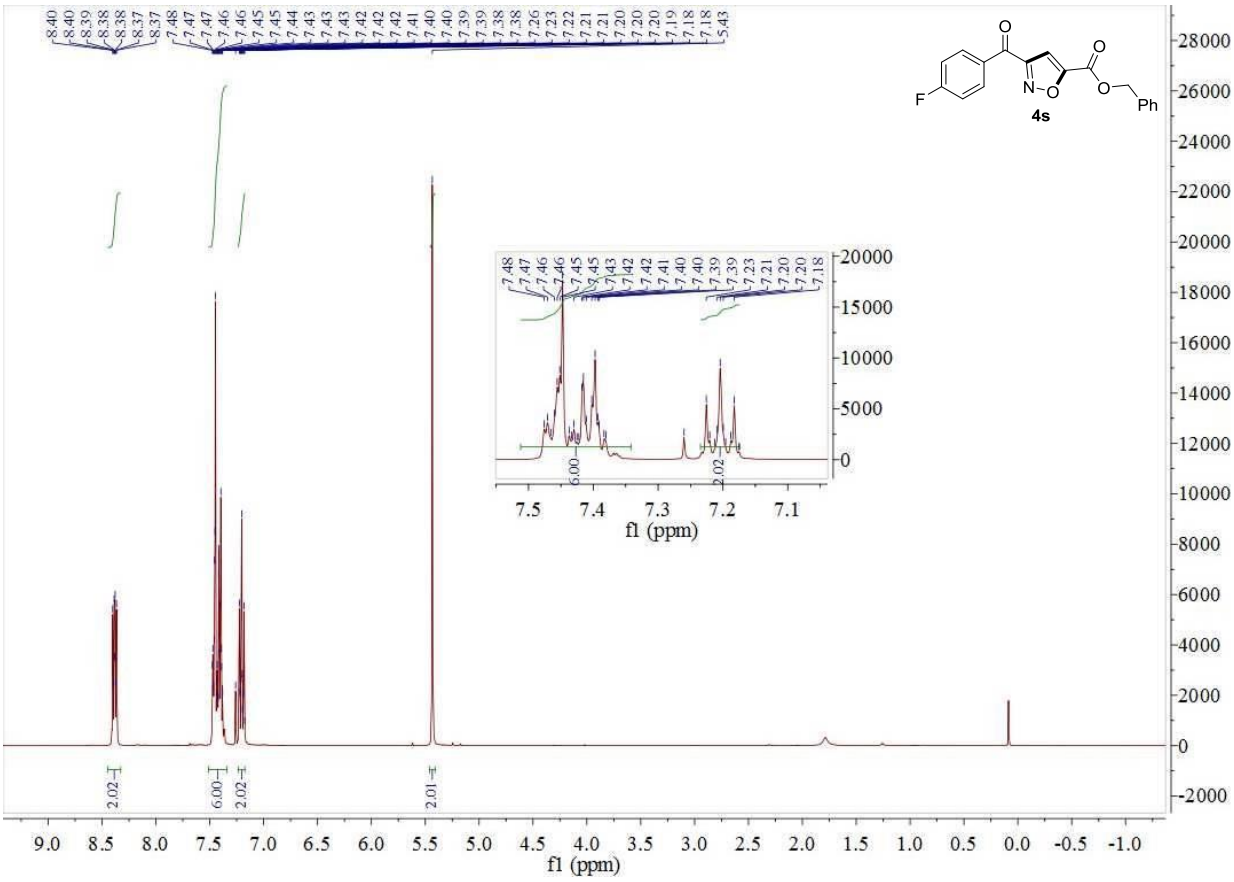

n-Bu
Ph
4p
f1 (ppm)

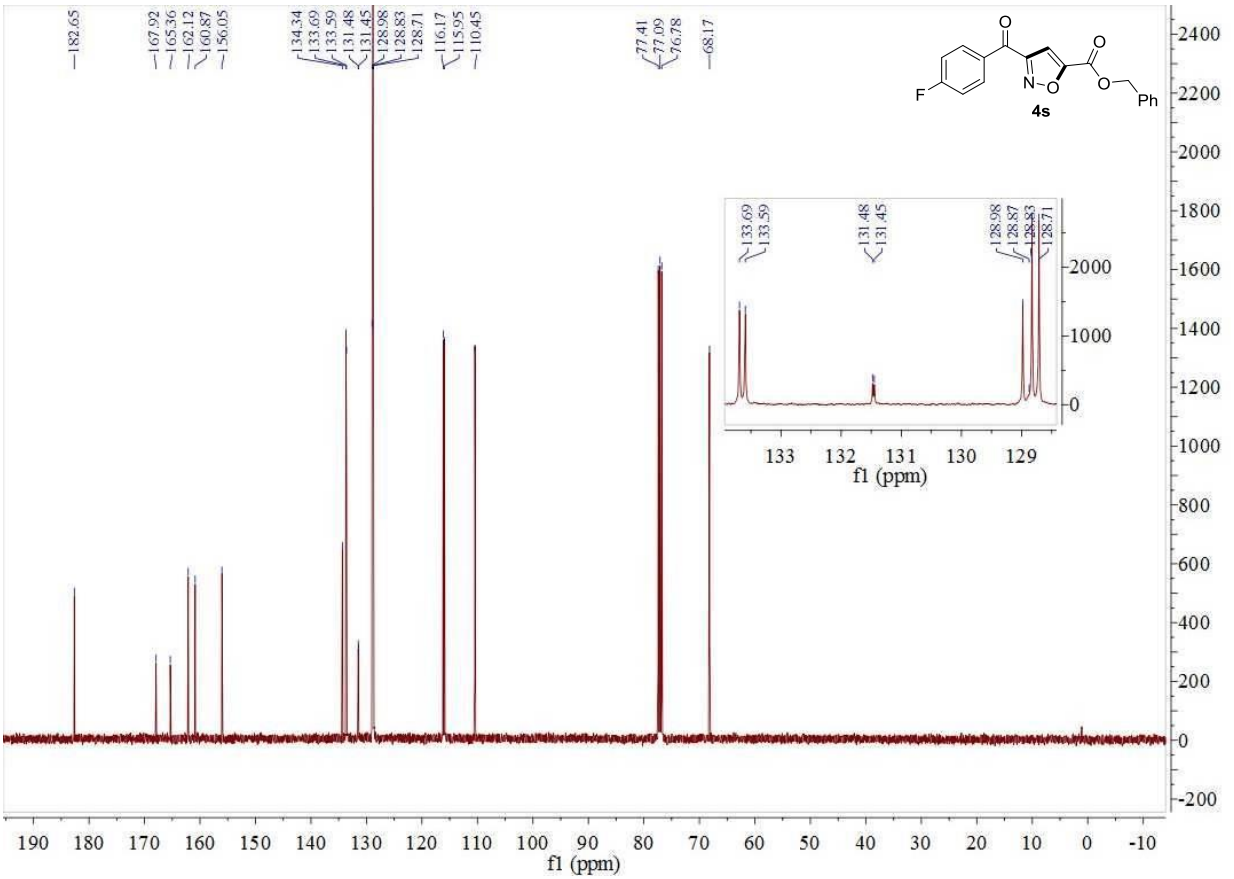

4q
f1 (ppm)

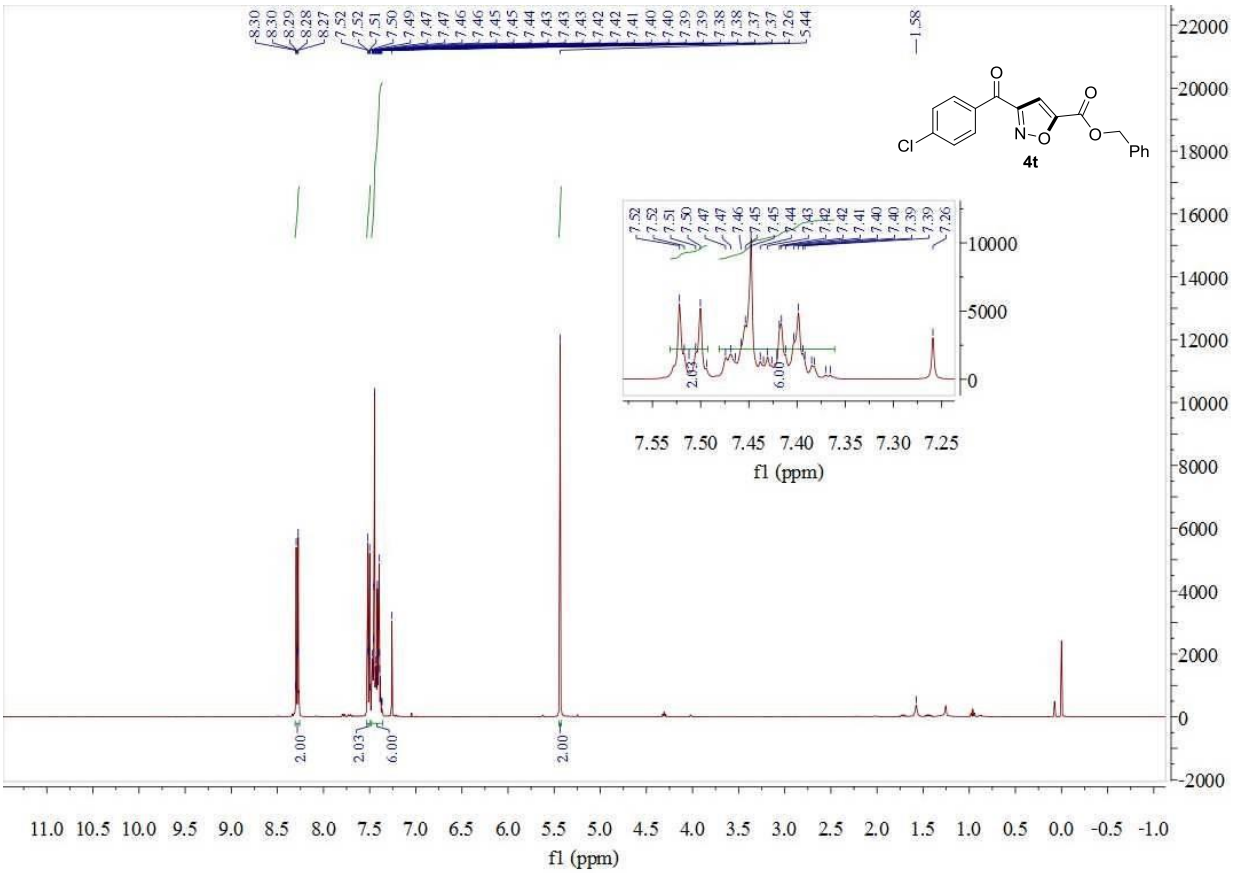

4q
f1 (ppm)

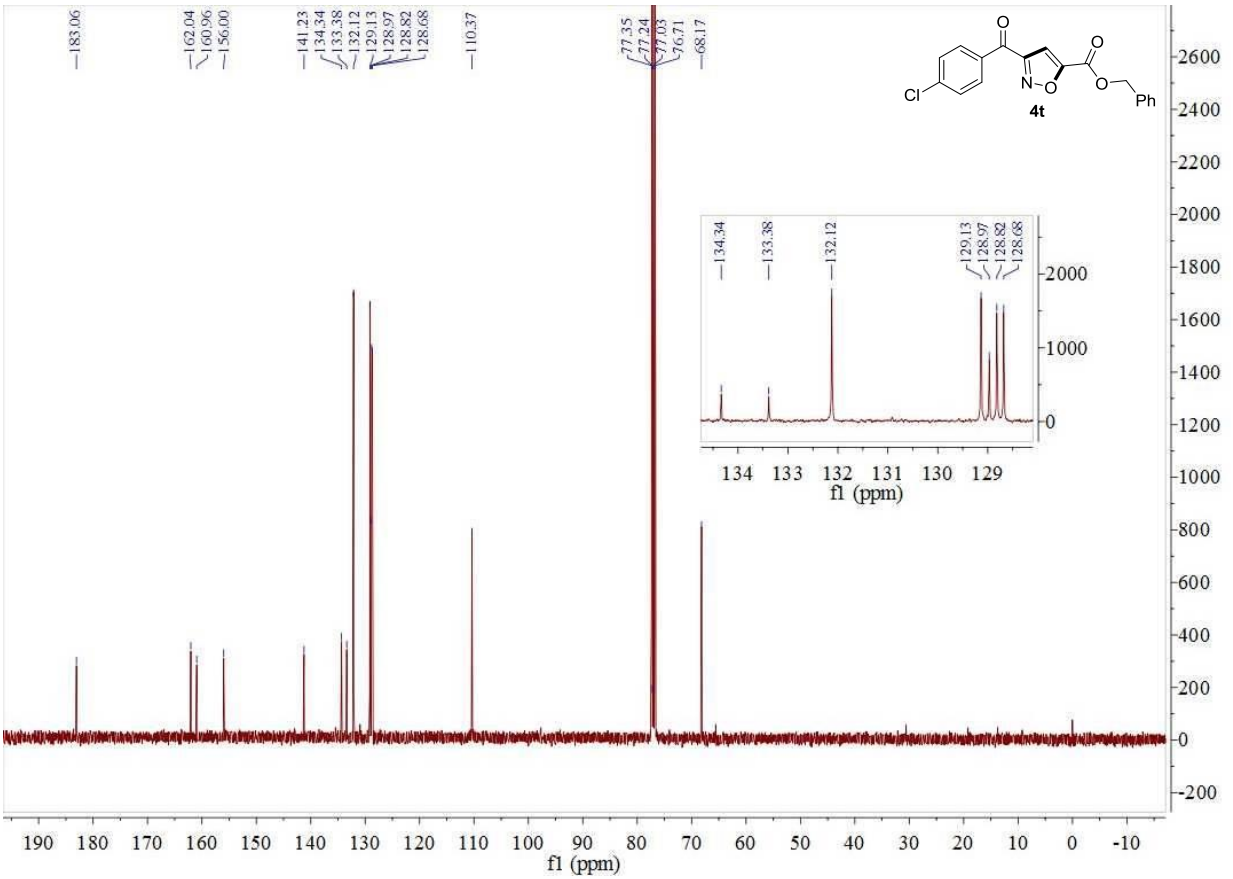

4r
f1 (ppm)

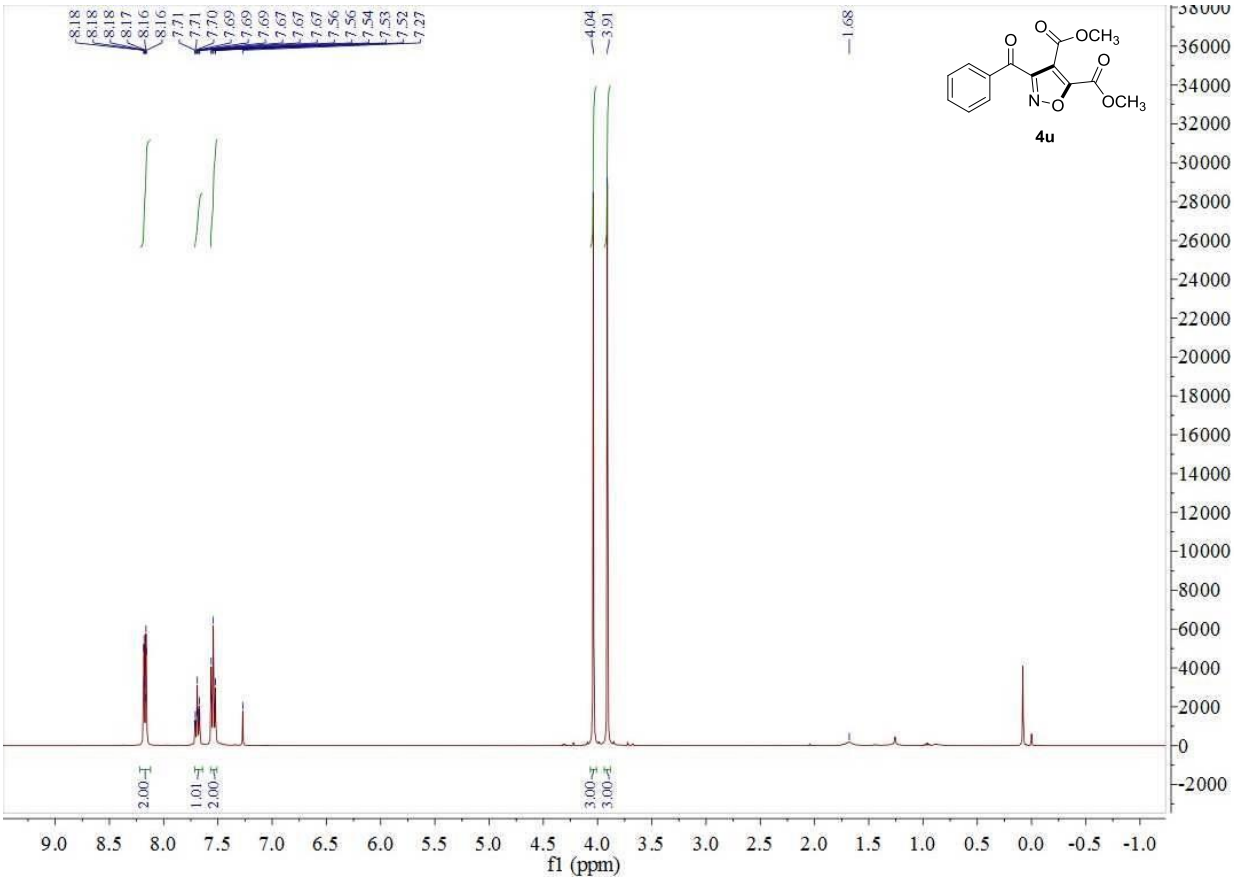

4r
f1 (ppm)


4s
f1 (ppm)


4s
f1 (ppm)


4t
f1 (ppm)


4t
f1 (ppm)


8.18
8.18
8.18
8.17
8.16
8.16
7.71
7.71
7.70
7.69
7.69
7.69
7.67
7.67
7.67
7.56
7.56
7.54
7.53
7.52
7.27
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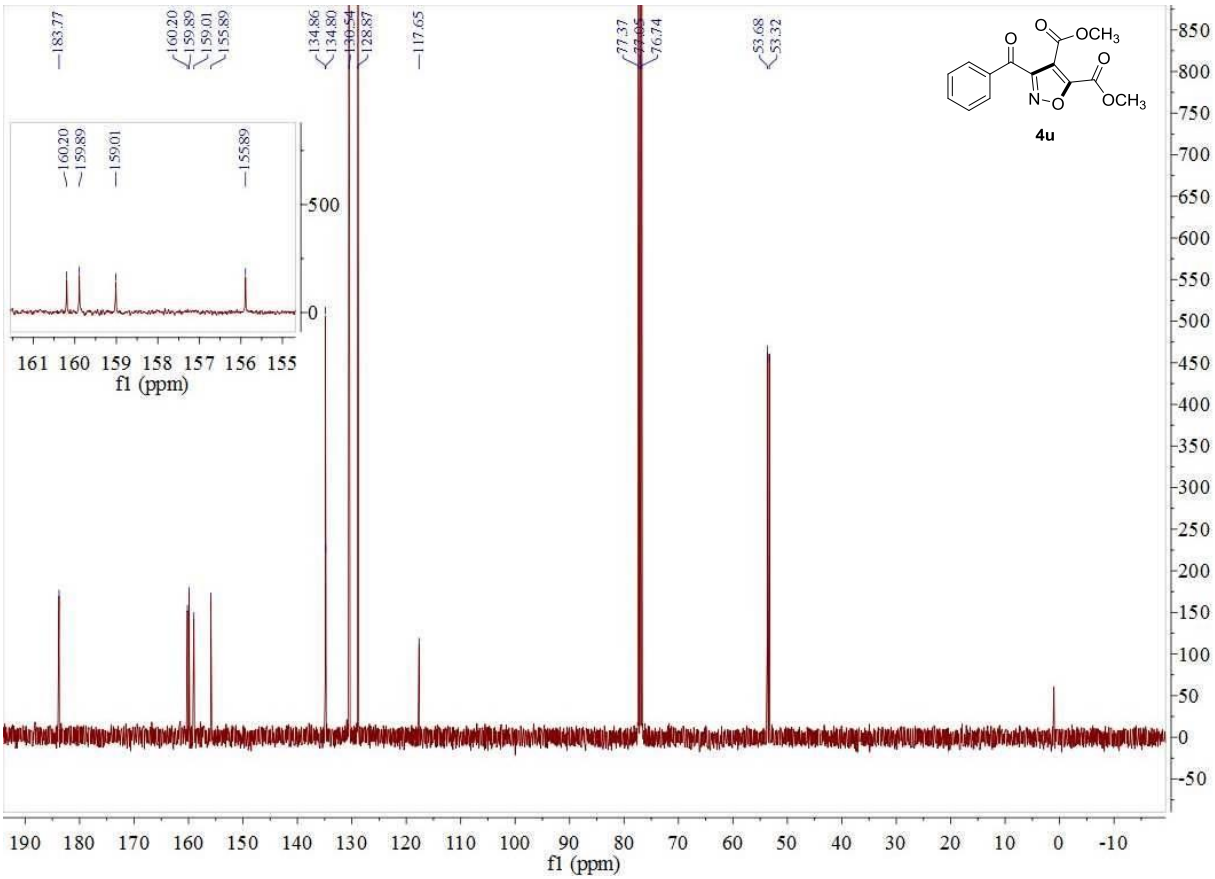
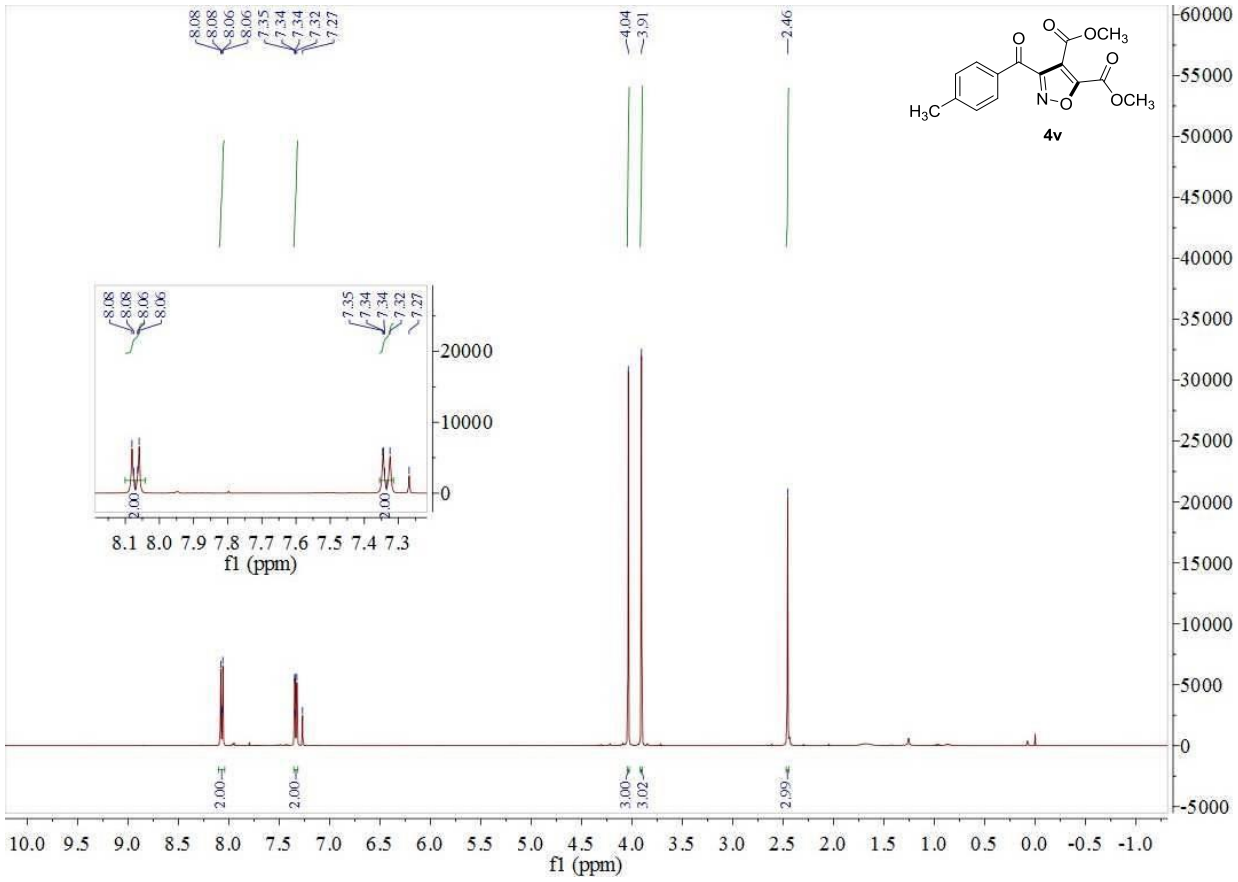
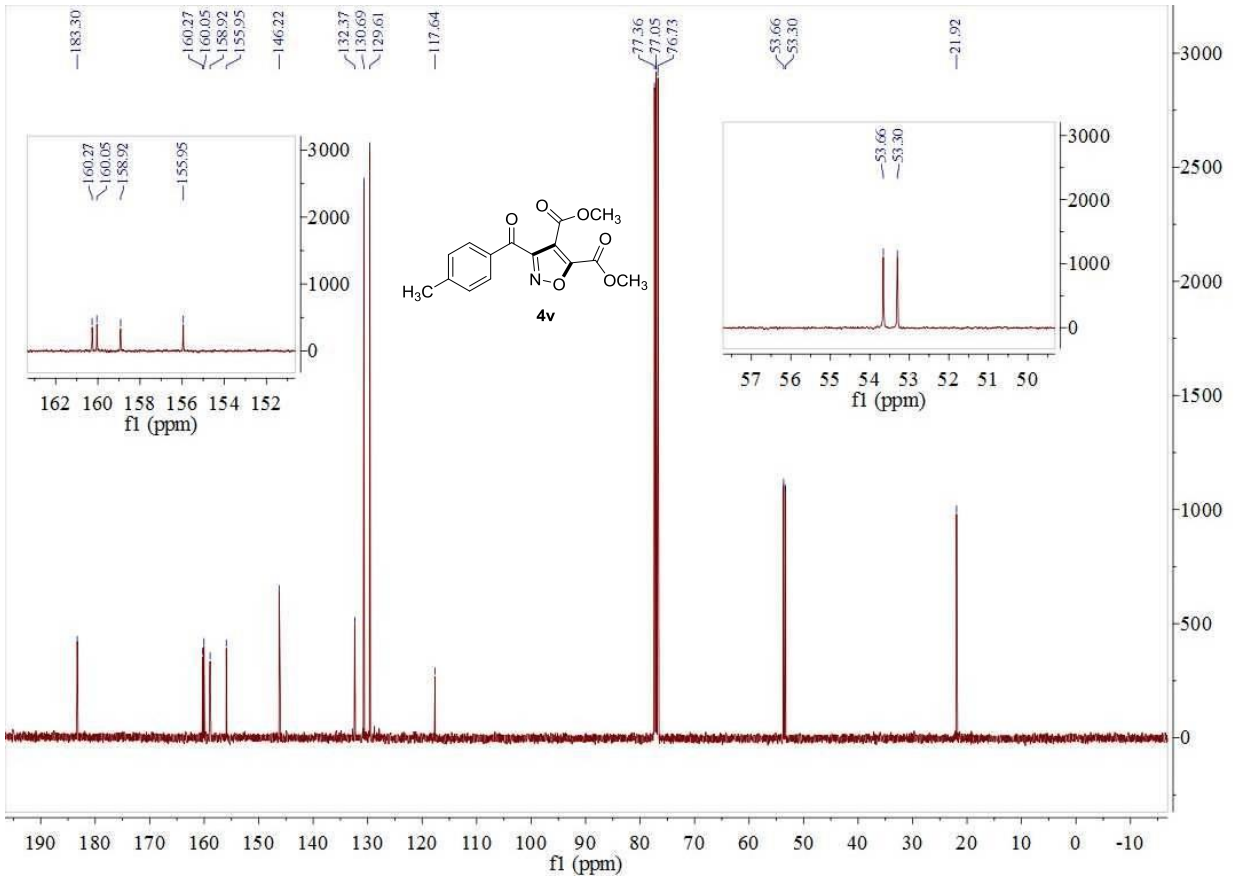
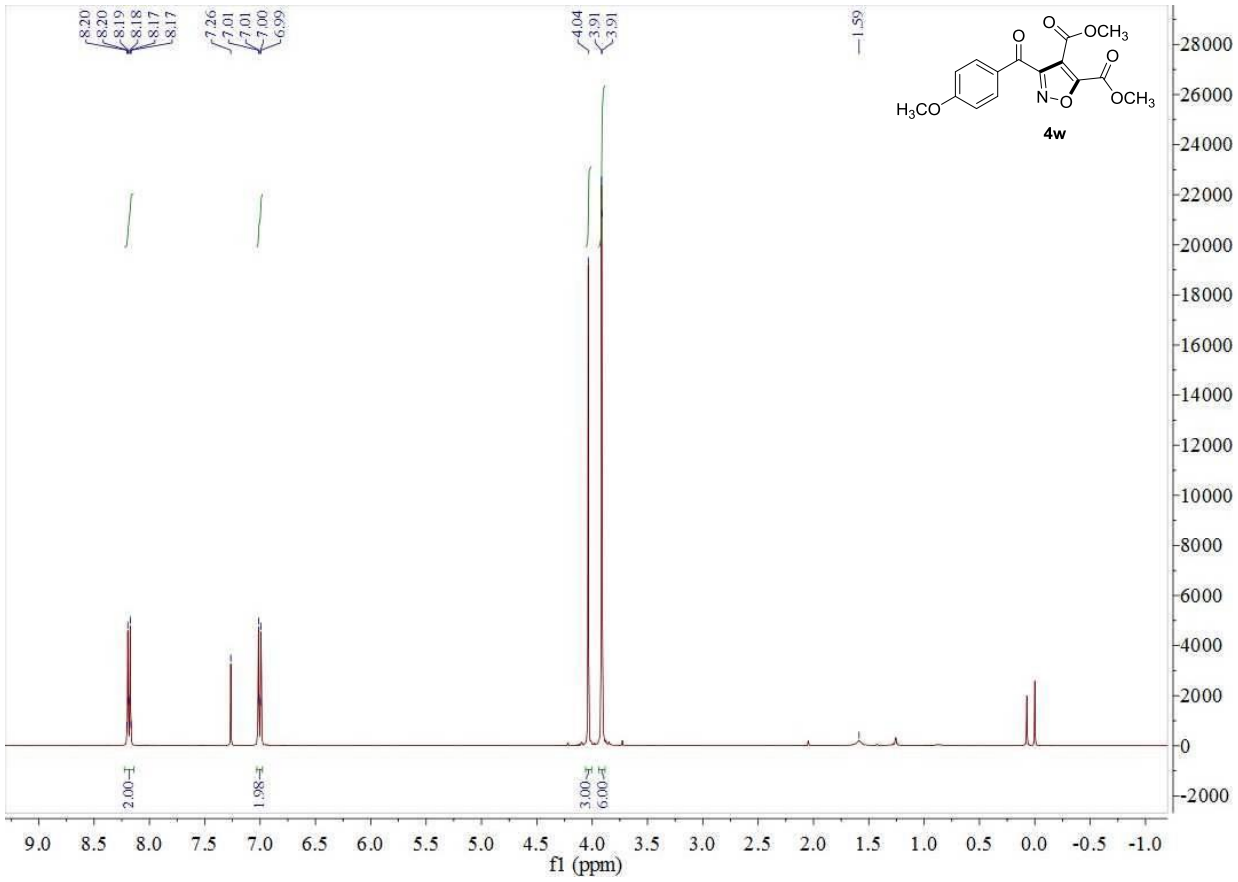
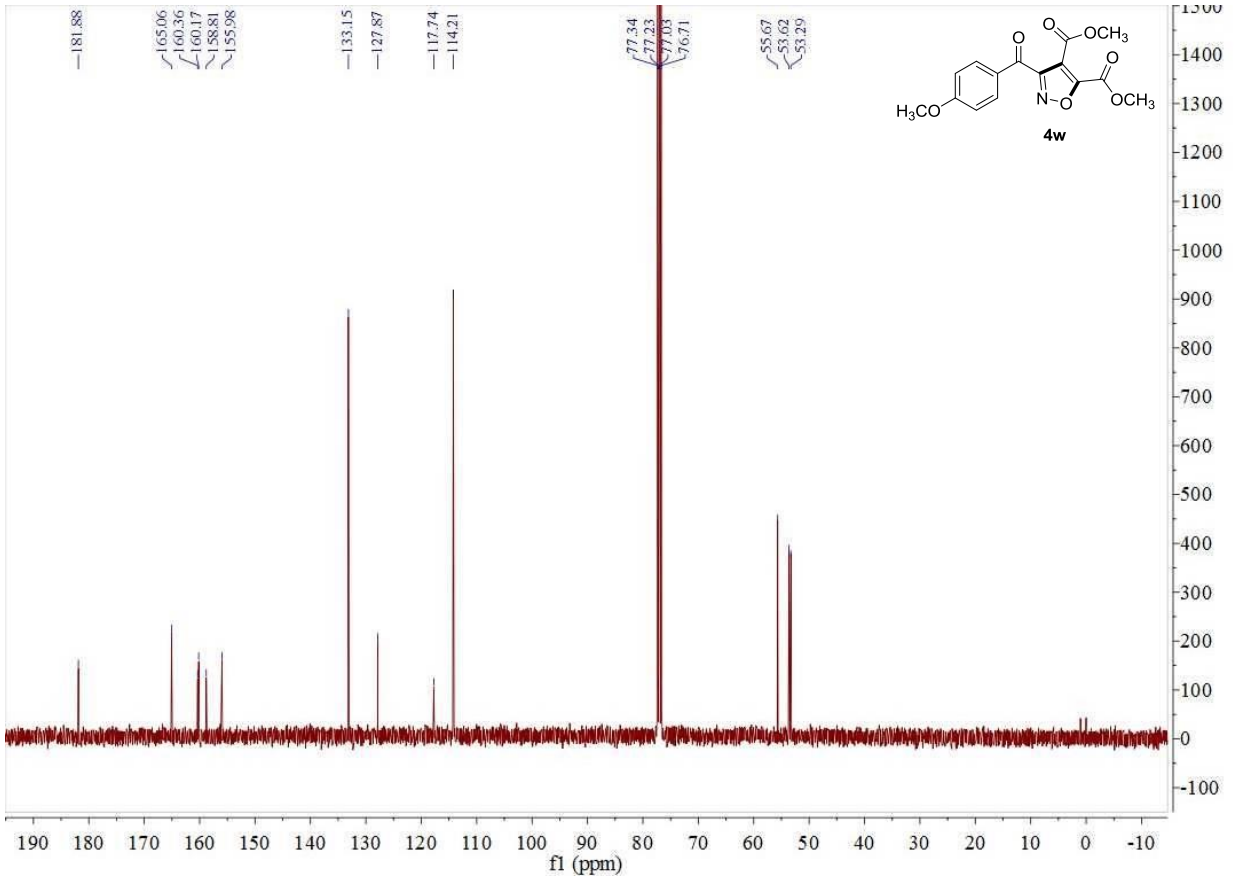
3.00
3.00
OCH3
OCH3
4u
f1 (ppm)

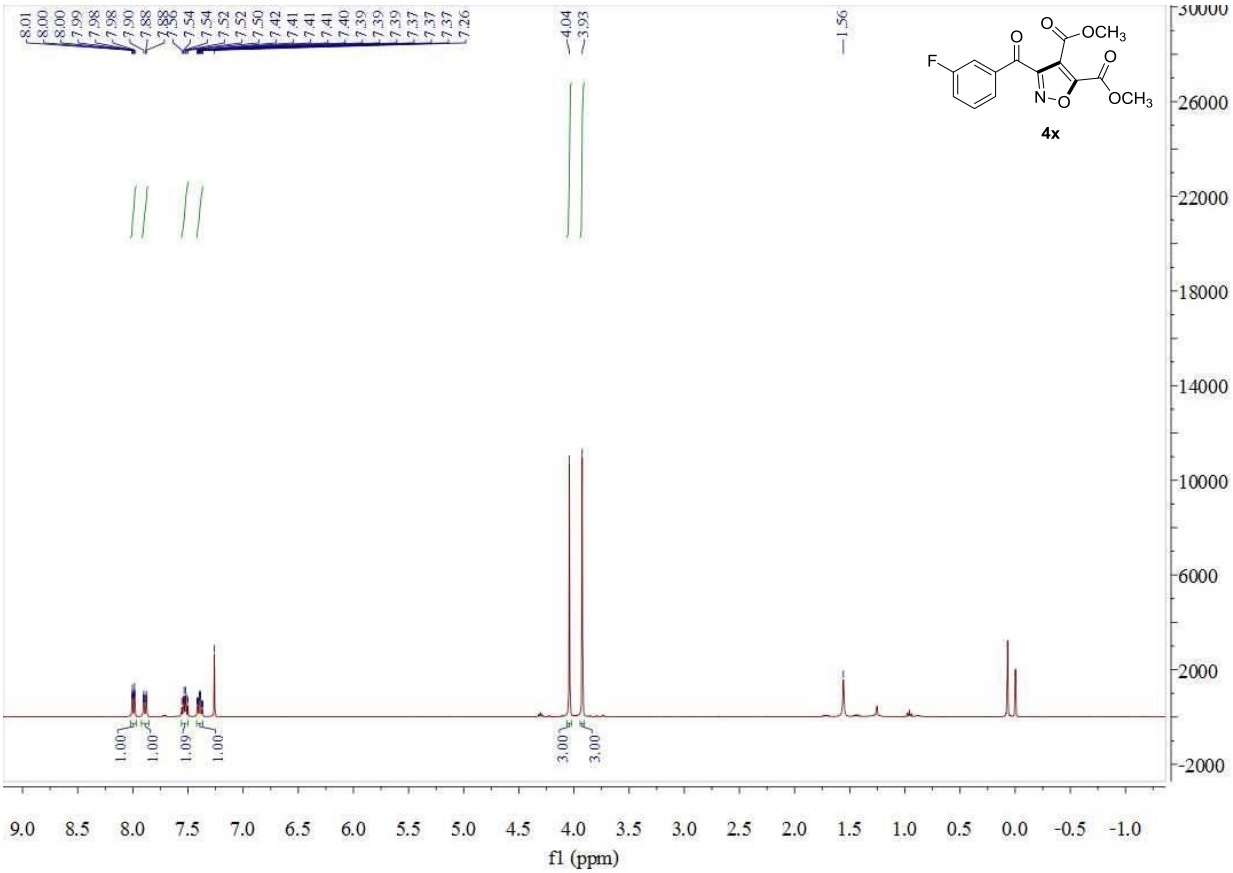

183.77
160.20
159.89
159.01
155.89
134.86
134.80
130.54
128.87
117.65
77.37
77.05
76.74
53.68
53.32
OCH3
OCH3
4u
f1 (ppm)

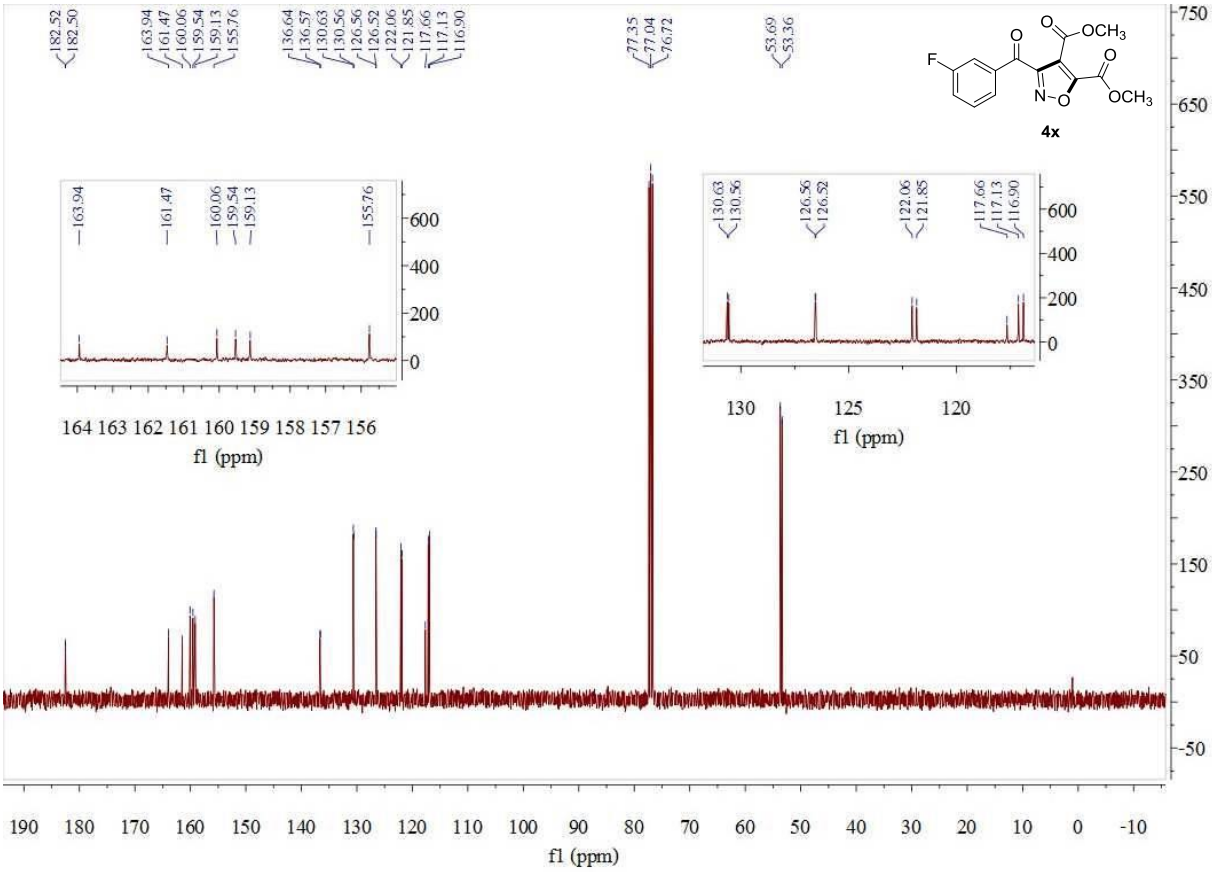

8.08
8.08
8.06
8.06
7.35
7.34
7.34
7.32
7.27
4.04
3.91
2.46
OCH3
H3C
4v
2.00
2.00
3.00
3.02
2.99
f1 (ppm)

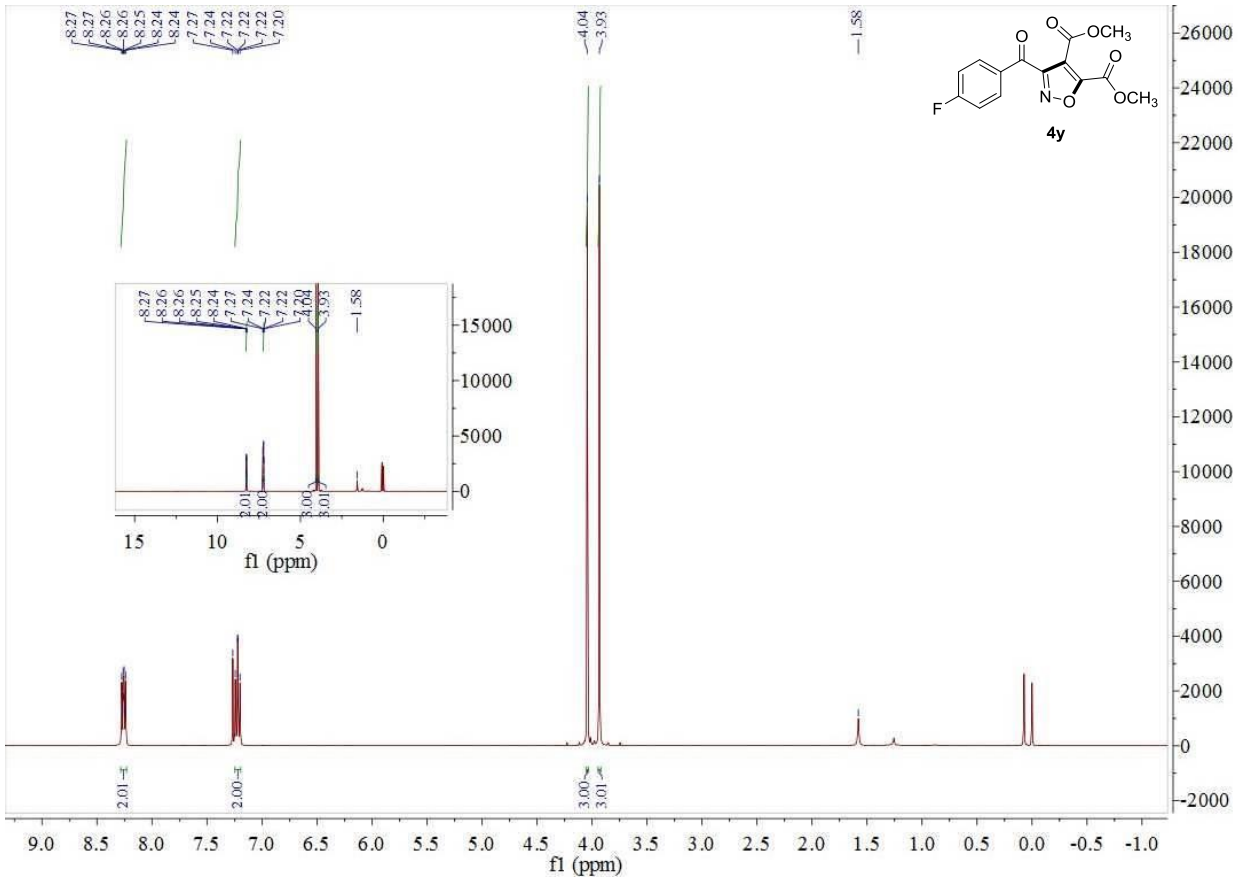

183.30
160.27
160.05
158.92
155.95
146.22
132.37
130.69
129.61
117.64
77.36
77.05
76.73
53.66
53.30
21.92
OCH3
H3C
4v
f1 (ppm)

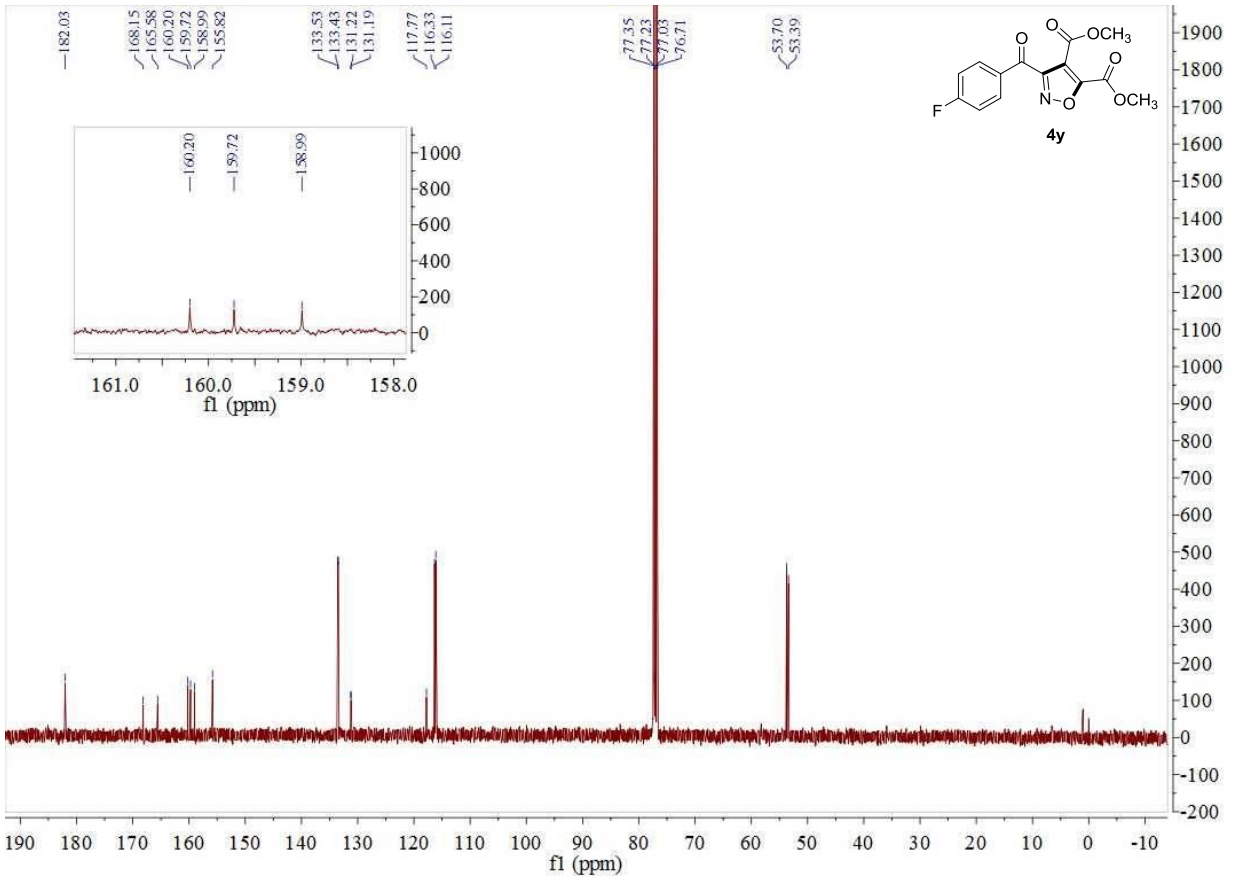

8.20
8.20
8.19
8.18
8.17
8.17
7.26
7.01
7.01
7.00
6.99
4.04
3.91
3.91
1.59
2.00
1.98
3.00
6.00
f1 (ppm)
4w

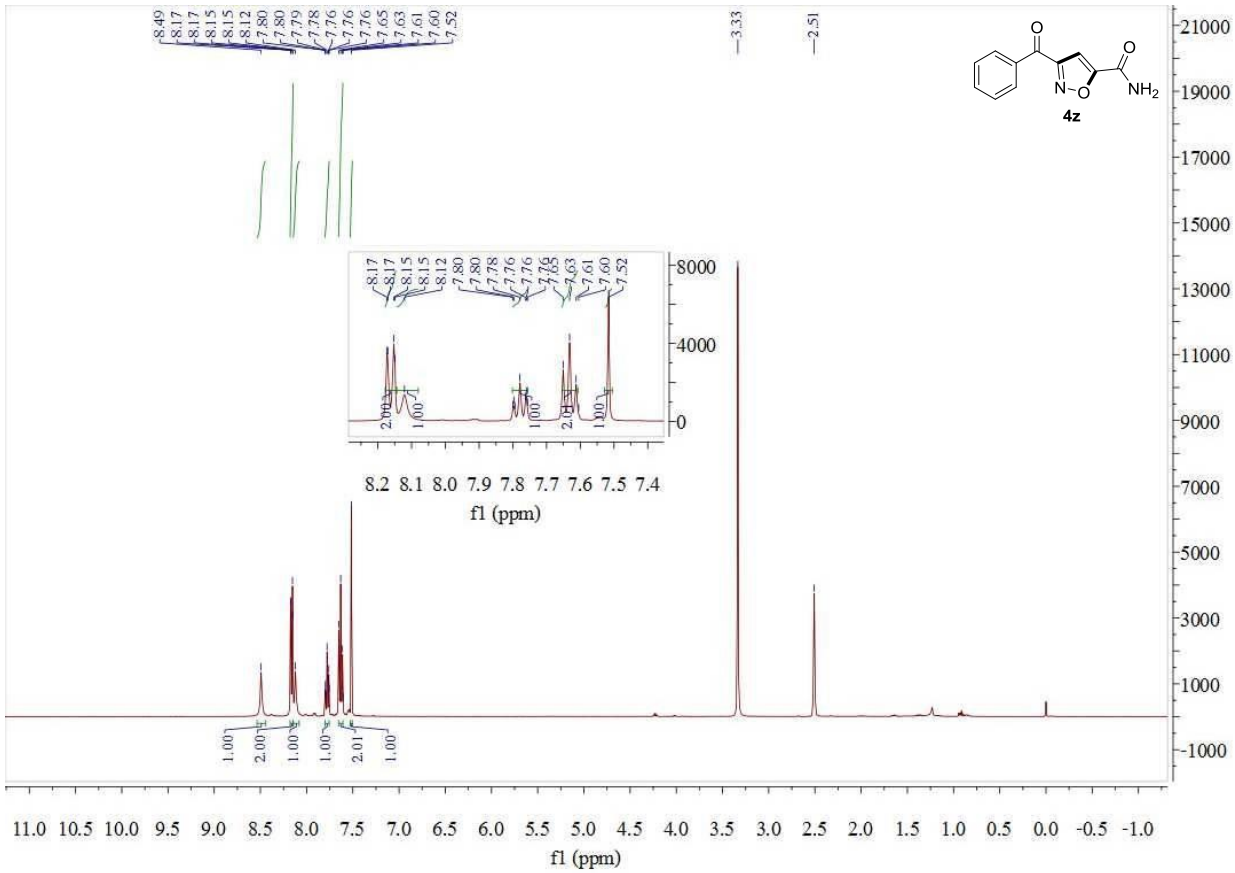

181.88
165.06
160.36
160.17
158.81
155.98
133.15
127.87
117.74
114.21
77.34
77.23
77.03
76.71
55.67
53.62
53.29
f1 (ppm)
4w

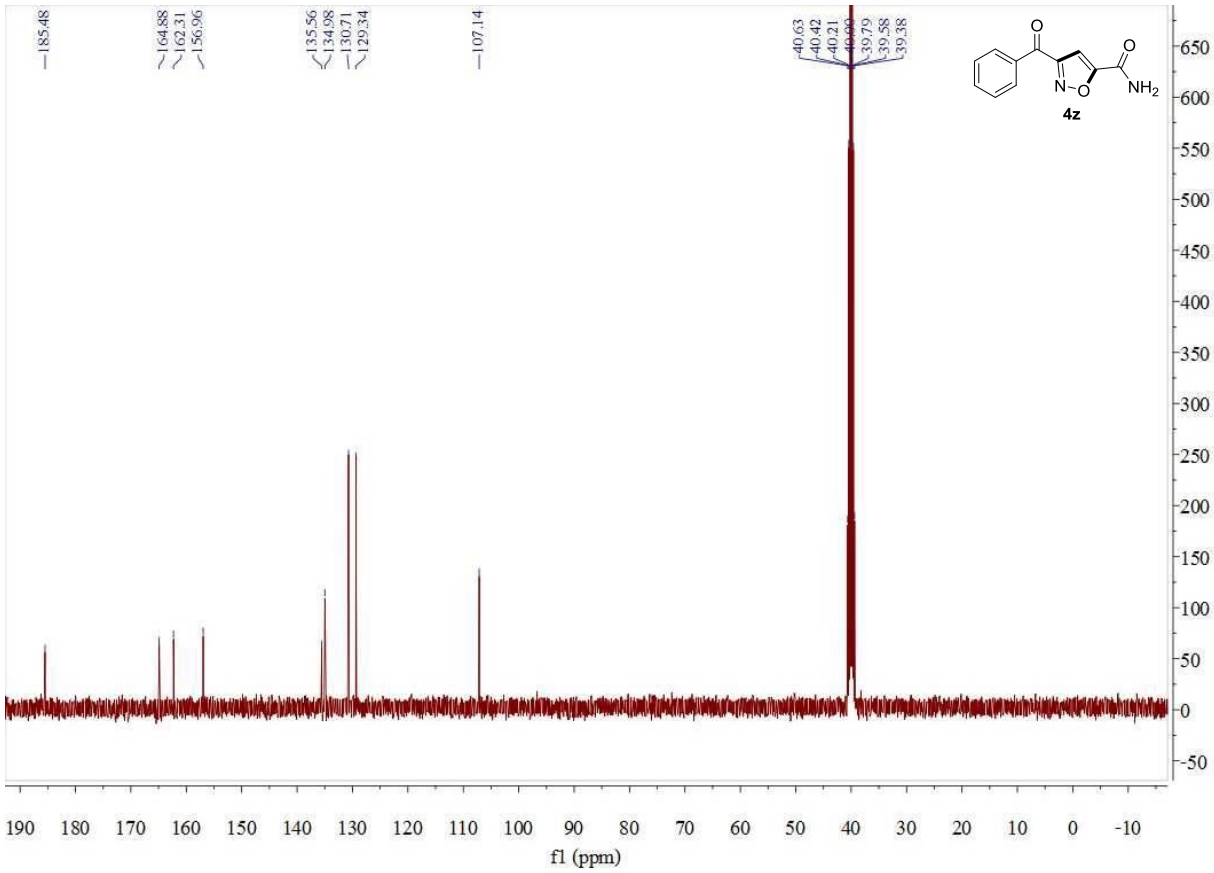

4x
f1 (ppm)

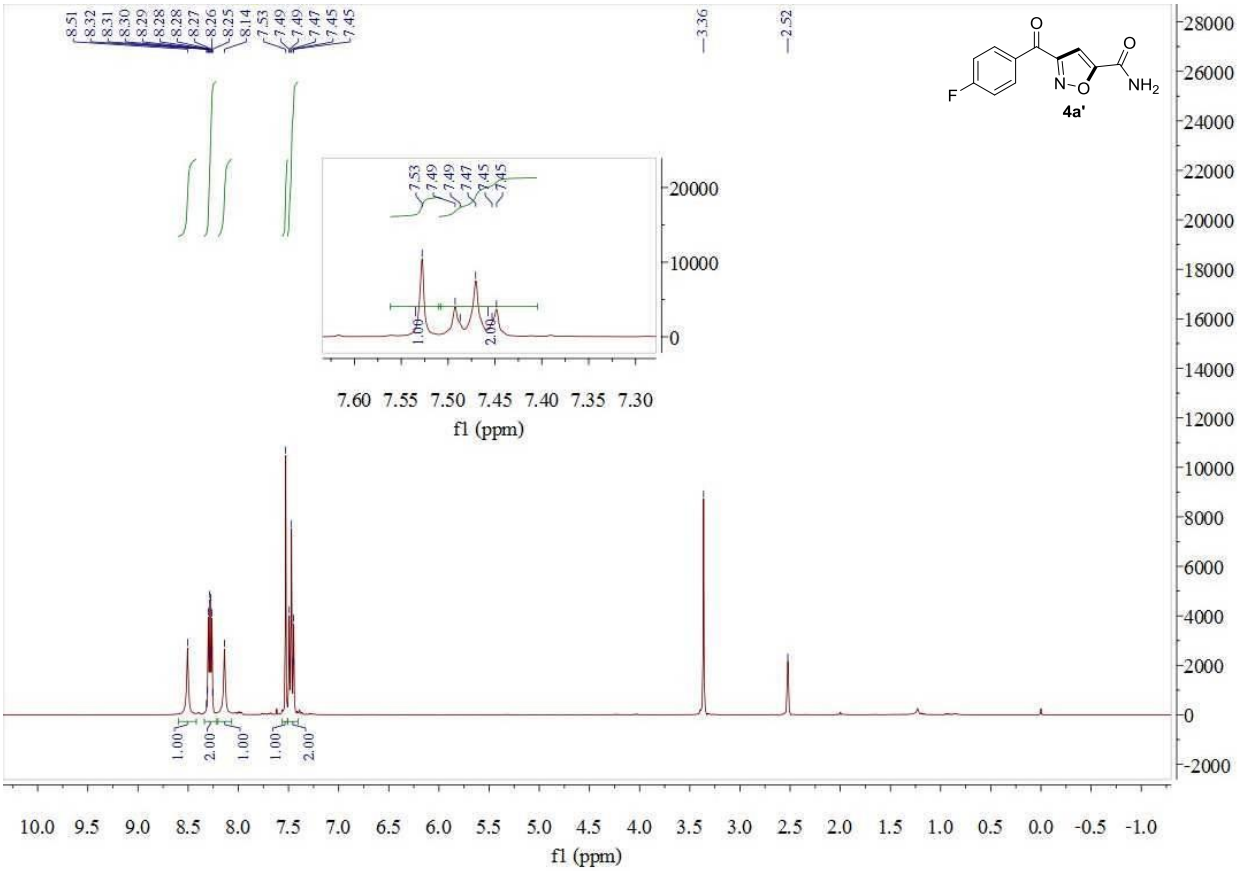

4x
f1 (ppm)

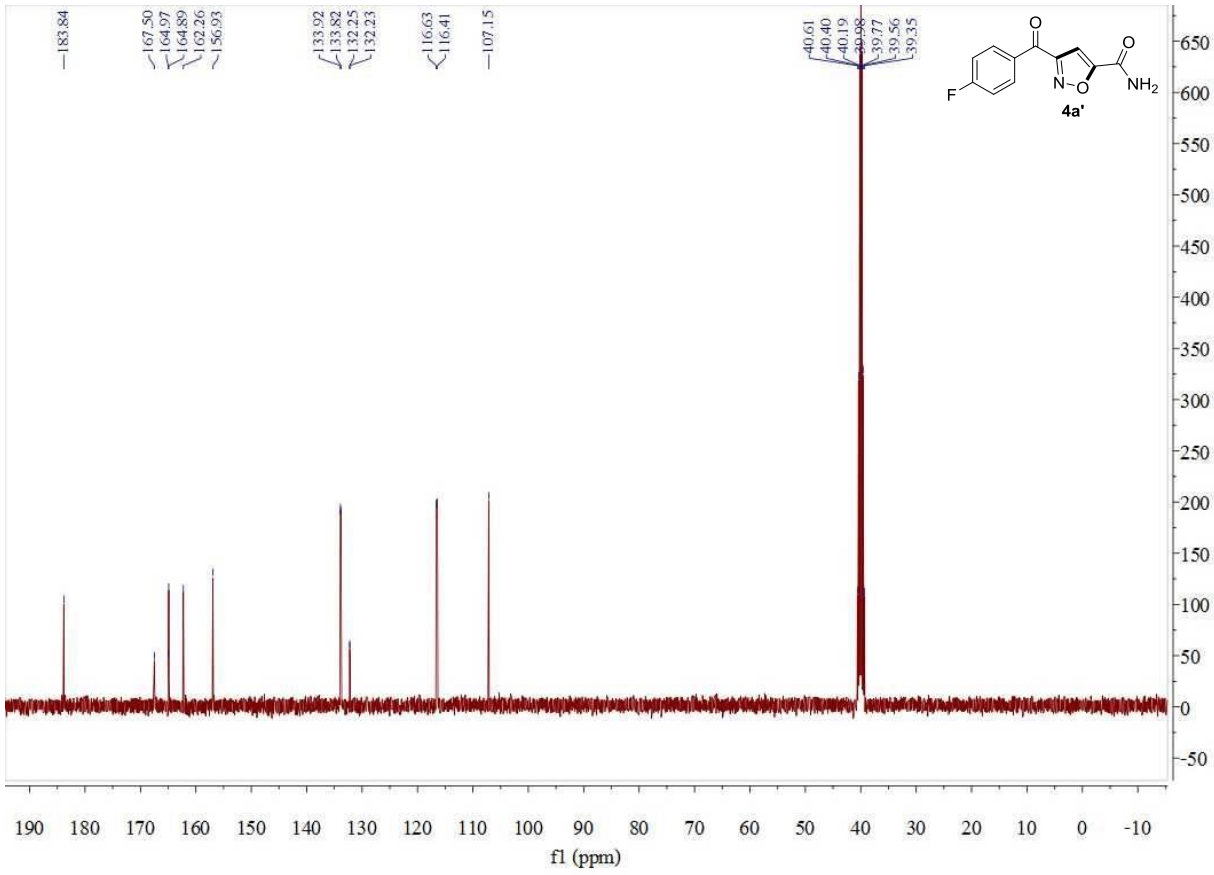

8.27
8.27
8.26
8.26
8.25
8.25
8.24
8.24
7.27
7.24
7.22
7.22
7.22
7.20
4.04
3.93
1.58
OCH3
OCH3
F
4y
2.01
2.00
3.00
3.01
f1 (ppm)

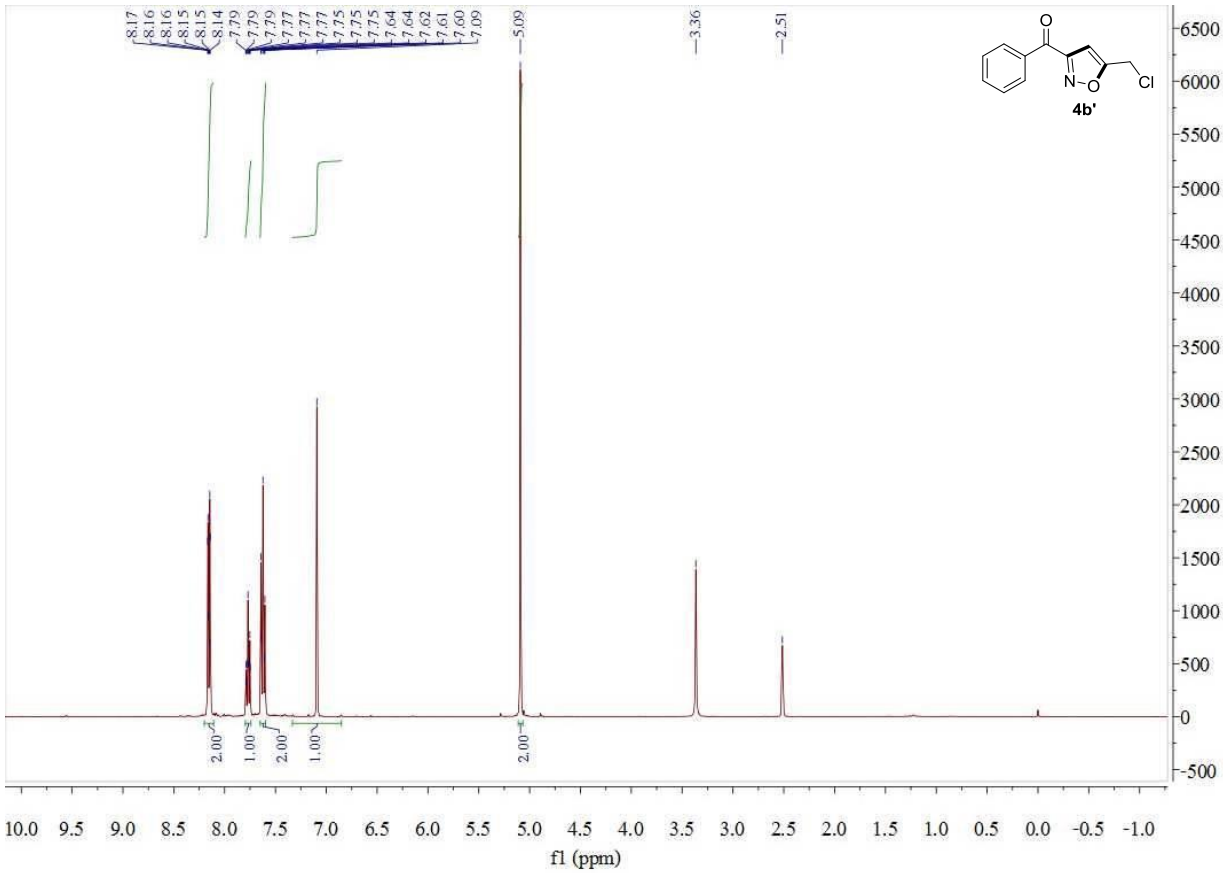

182.03
168.15
165.58
160.20
159.72
158.99
155.82
133.53
133.43
131.22
131.19
117.77
116.33
116.11
77.35
77.23
77.03
76.71
53.70
53.39
OCH3
OCH3
F
4y
160.20
159.72
158.99
f1 (ppm)

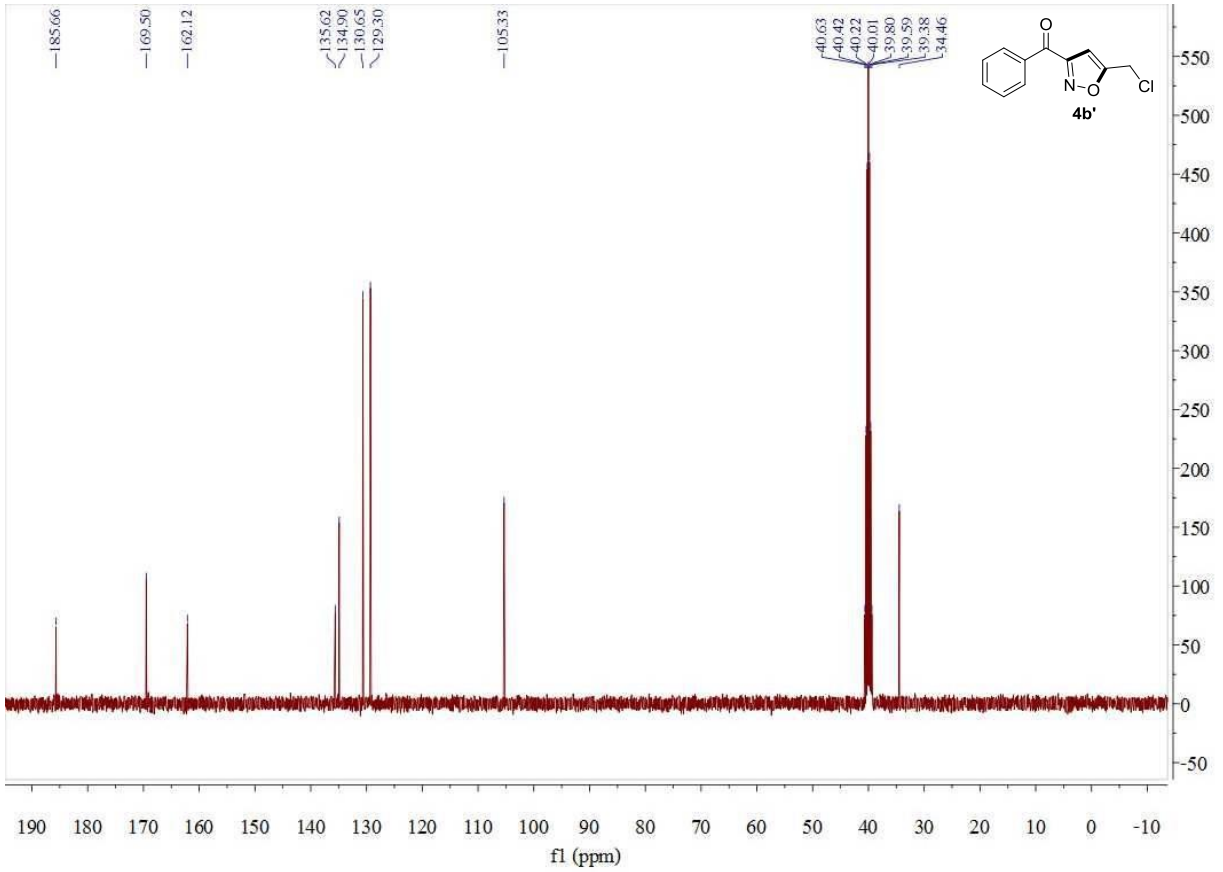

8.49
8.17
8.17
8.17
8.15
8.15
8.12
7.80
7.79
7.78
7.76
7.76
7.76
7.65
7.63
7.61
7.60
7.52
3.33
2.51
4z
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

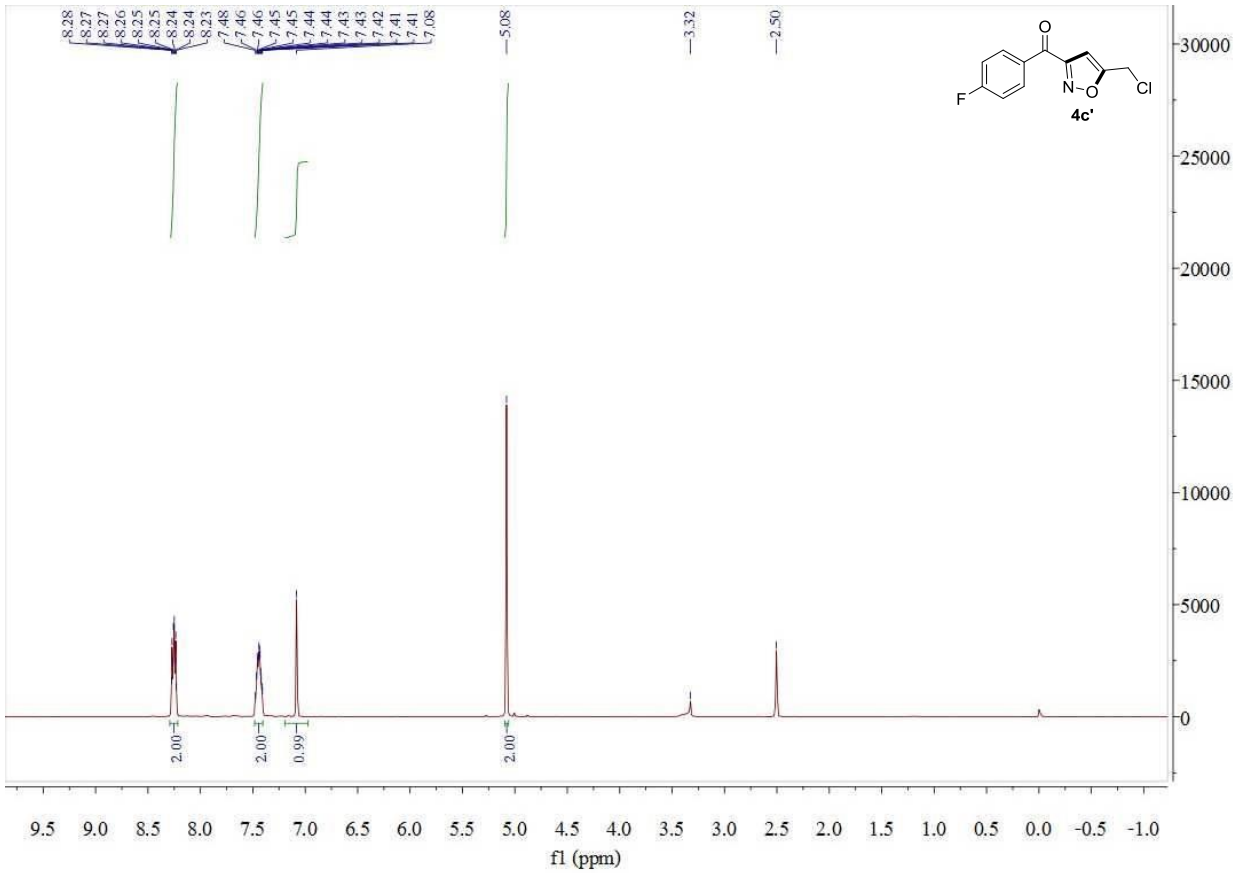

185.48
164.88
162.31
156.96
135.56
134.98
130.71
129.34
107.14
40.63
40.42
40.21
39.79
39.58
39.38
4z
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

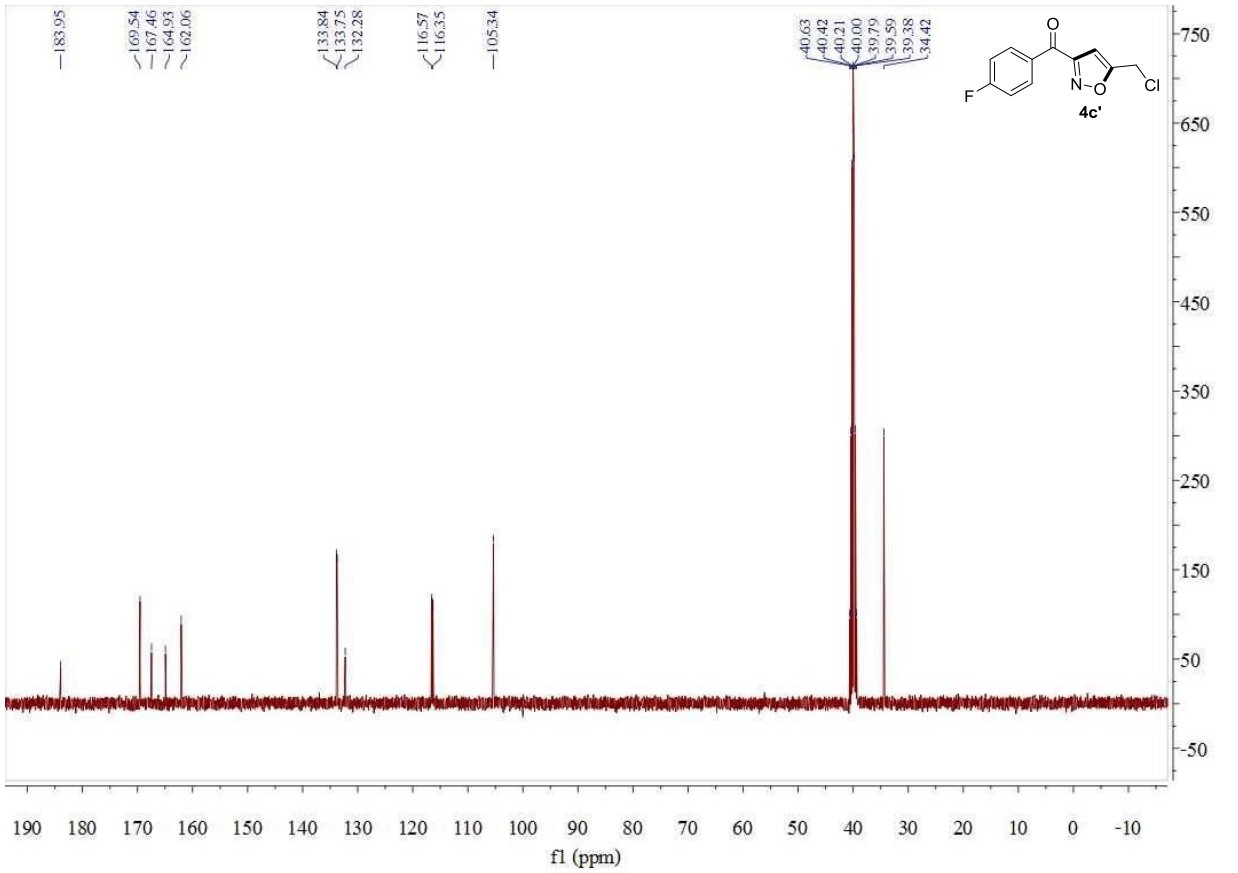

8.51
8.32
8.31
8.30
8.29
8.28
8.28
8.27
8.26
8.25
8.14
7.53
7.49
7.49
7.47
7.45
7.45
3.36
2.52
7.53
7.49
7.49
7.47
7.45
7.45
1.00
2.00
7.60 7.55 7.50 7.45 7.40 7.35 7.30
f1 (ppm)
1.00
2.00
1.00
1.00
2.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
4a'


183.84
167.50
164.97
164.89
162.26
156.93
133.92
133.82
132.25
132.23
116.63
116.41
107.15
40.61
40.40
40.19
39.98
39.77
39.56
39.35
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
4a'


8.17
8.16
8.16
8.15
8.15
8.14
7.79
7.79
7.79
7.79
7.77
7.77
7.77
7.77
7.75
7.75
7.75
7.64
7.64
7.62
7.61
7.60
7.09
5.09
3.36
2.51
2.00
1.00
2.00
1.00
2.00
O
N
O
Cl
4b'
fl (ppm)


185.66
169.50
162.12
135.62
134.90
130.65
129.30
105.33
40.63
40.42
40.22
40.01
39.80
39.59
39.38
34.46
O
N
O
Cl
4b'
fl (ppm)


8.28
8.27
8.27
8.26
8.25
8.24
8.24
8.23
7.48
7.46
7.46
7.45
7.45
7.44
7.44
7.43
7.43
7.42
7.41
7.41
7.08
5.08
3.32
2.50
2.00
2.00
0.99
2.00
4c'
F
Cl
O
N
O
30000
25000
20000
15000
10000
5000
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 -0.5 -1.0
f1 (ppm)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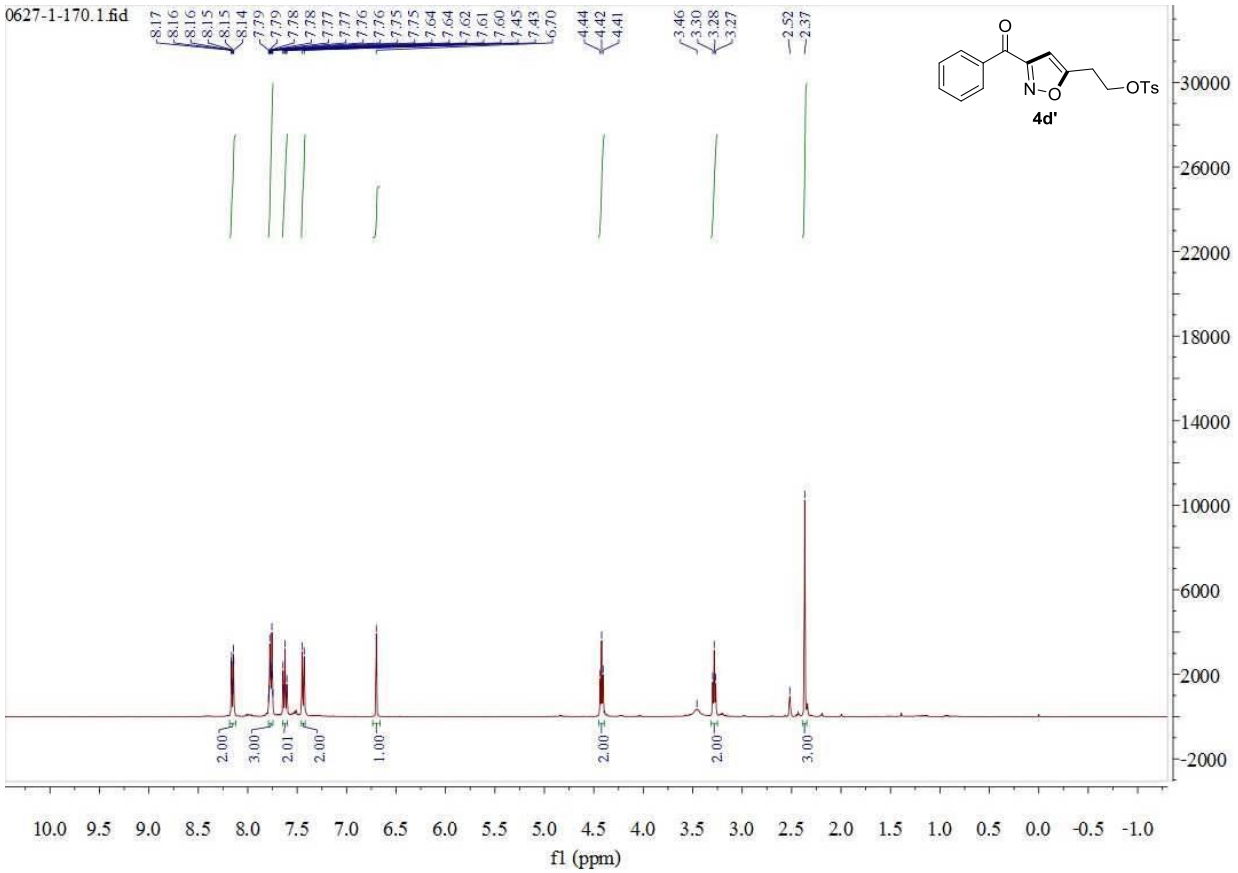
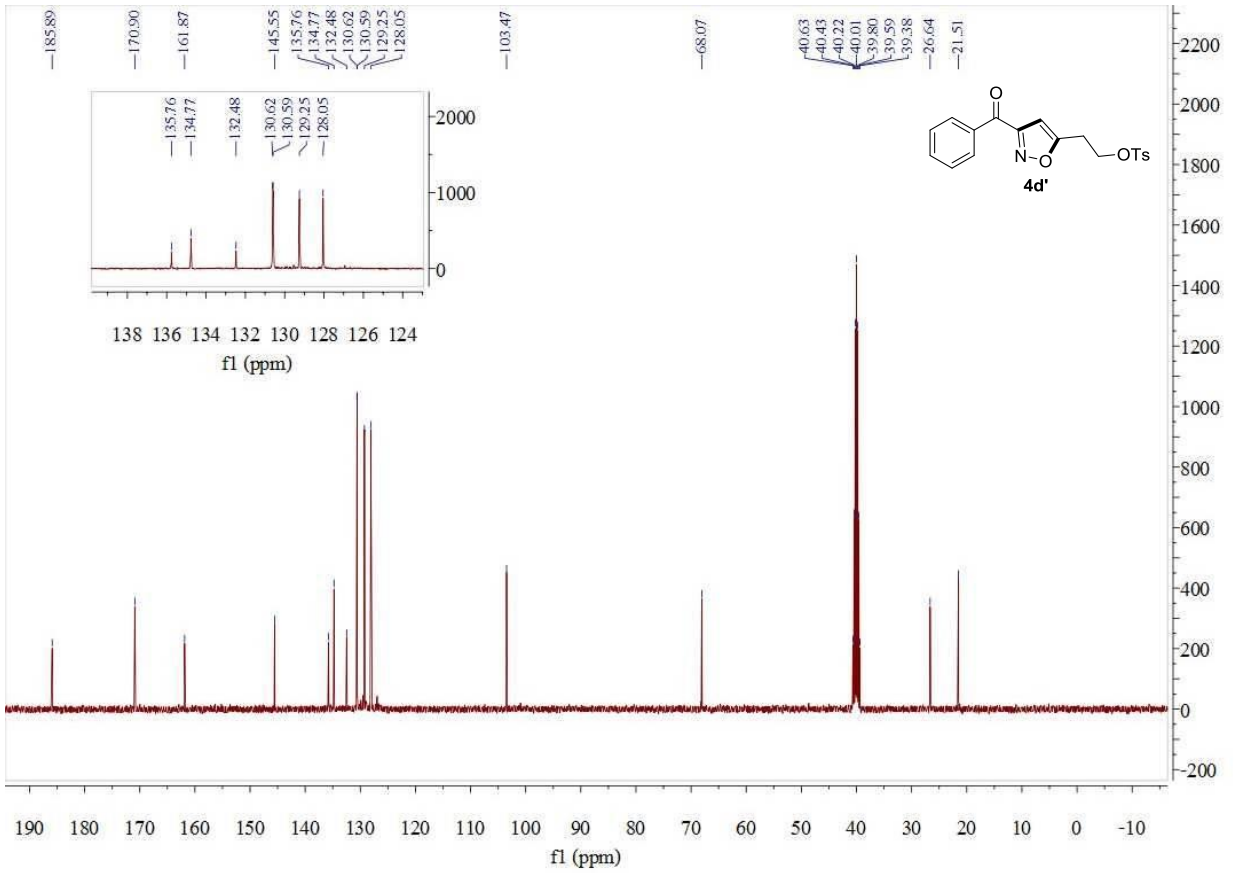
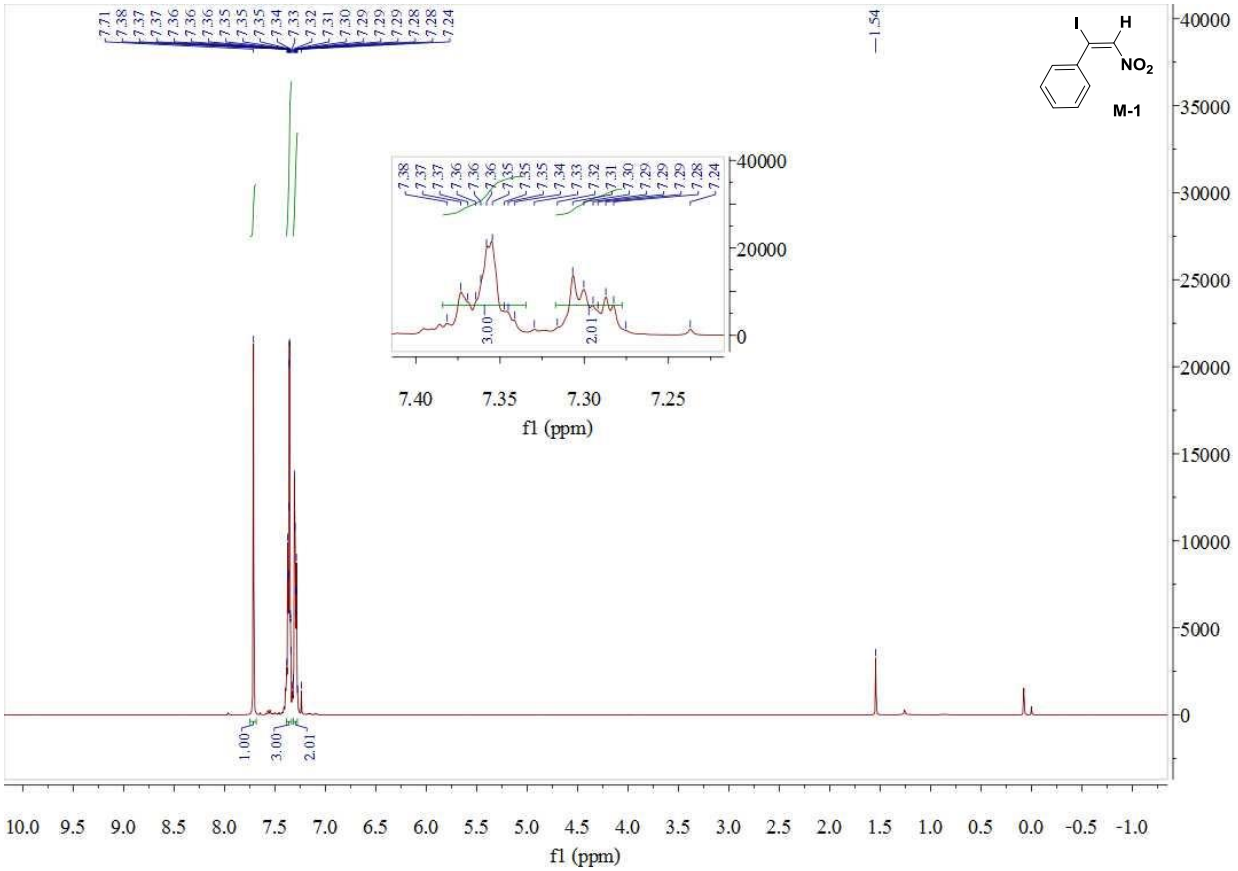
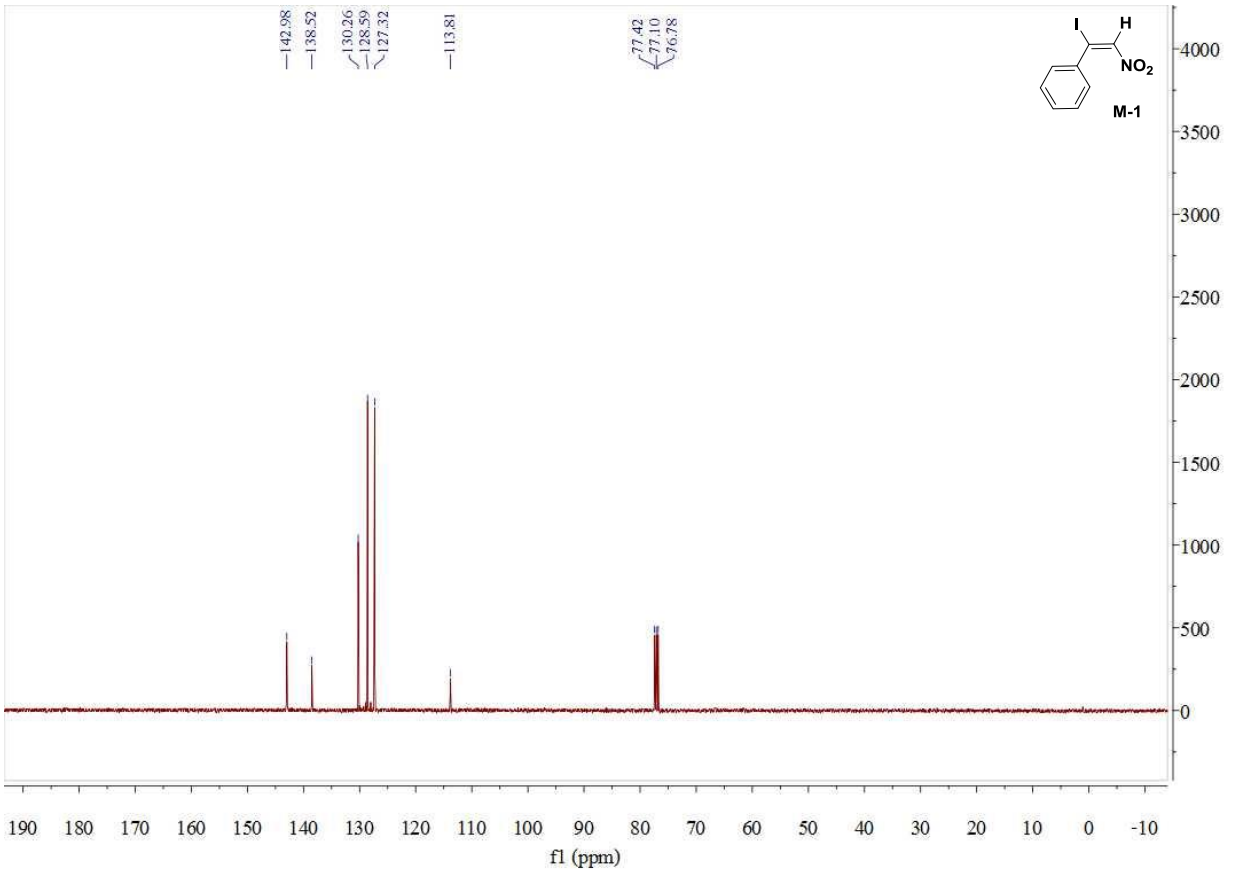
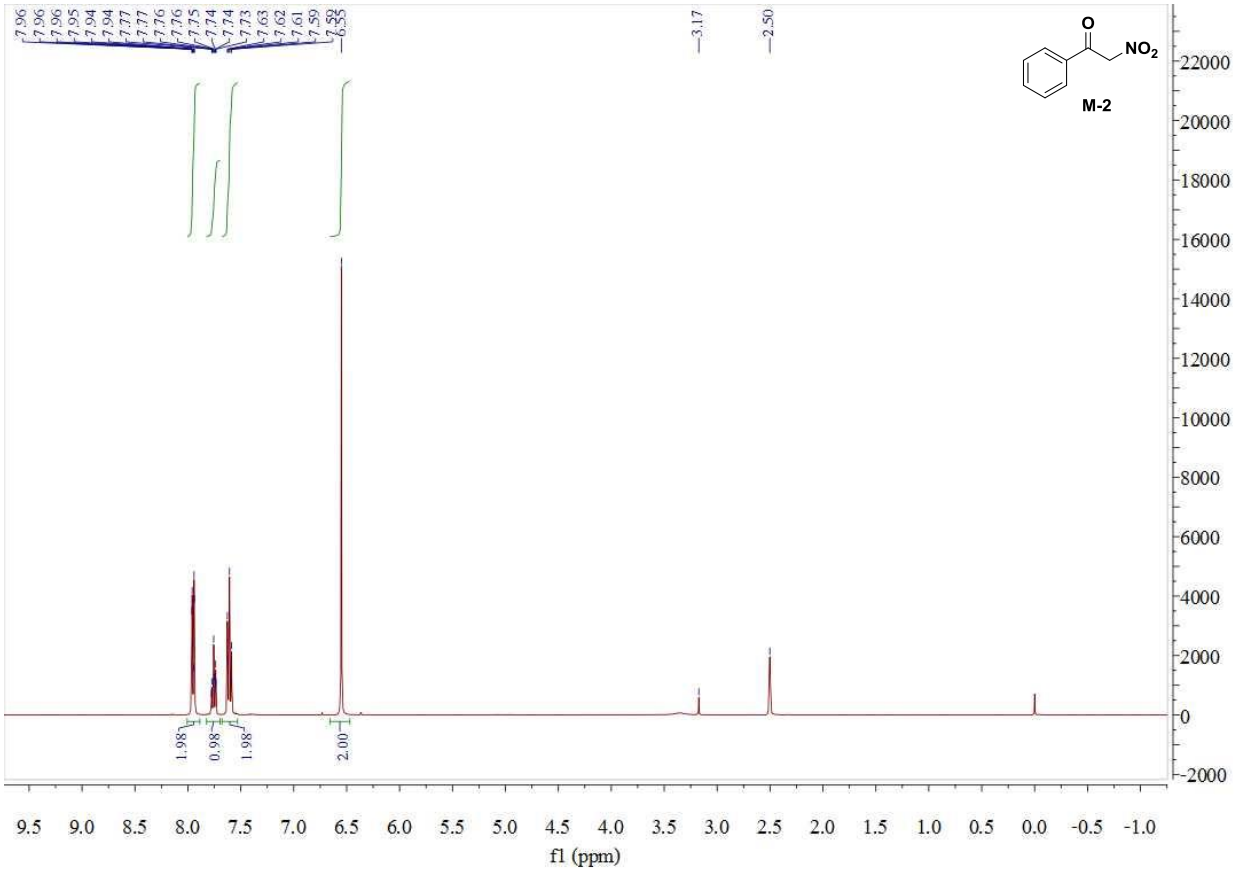
169.54
167.46
164.93
162.06
133.84
133.75
132.28
116.57
116.35
105.34
40.63
40.42
40.21
40.00
39.79
39.59
39.38
34.42
4c'
F
Cl
O
N
O
750
650
550
450
350
250
150
50
-50
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

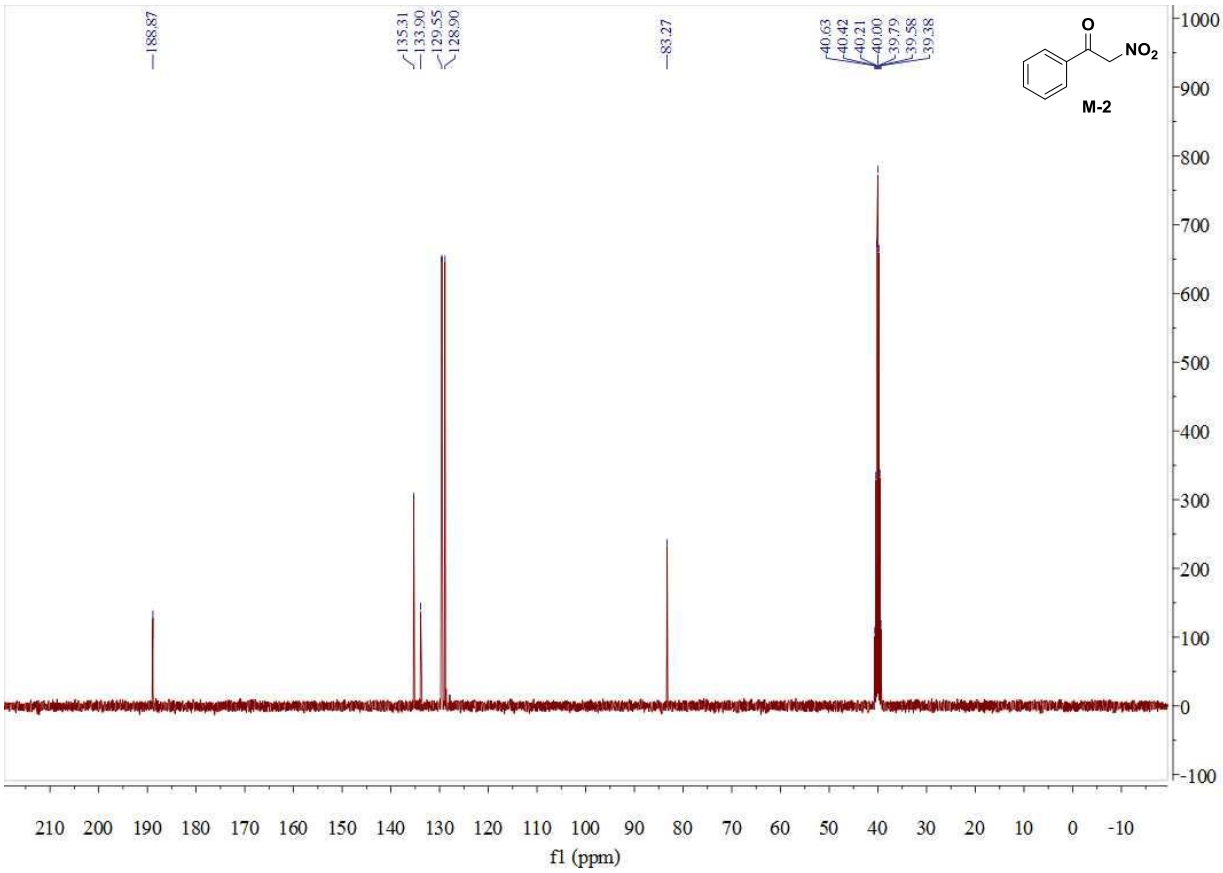

0627-1-170.1.fid
4d'
OTs
f1 (ppm)


4d'
OTs
f1 (ppm)


7.71
7.38
7.37
7.37
7.37
7.36
7.36
7.36
7.35
7.35
7.35
7.34
7.33
7.32
7.31
7.30
7.29
7.29
7.29
7.28
7.28
7.24
1.54
I
H
NO2
M-1
1.00
3.00
2.01
7.40
7.35
7.30
7.25
f1 (ppm)
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
-0.5
-1.0
f1 (ppm)


142.98
138.52
130.26
128.59
127.32
113.81
77.42
77.10
76.78
I
H
NO2
M-1
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.96
7.96
7.96
7.95
7.94
7.94
7.77
7.77
7.76
7.76
7.75
7.74
7.74
7.73
7.63
7.62
7.61
7.59
6.55
3.17
2.50
1.98
0.98
1.98
2.00
O
NO2
M-2
f1 (ppm)


188.87
135.31
133.90
129.55
128.90
83.27
40.63
40.42
40.21
40.00
39.79
39.58
39.38
O
NO2
M-2
f1 (ppm)