Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information (SI)

An Efficient Synthesis of Weinreb Amides and Ketones via Palladium Nanoparticles on ZIF-8 Catalysed Carbonylative Coupling

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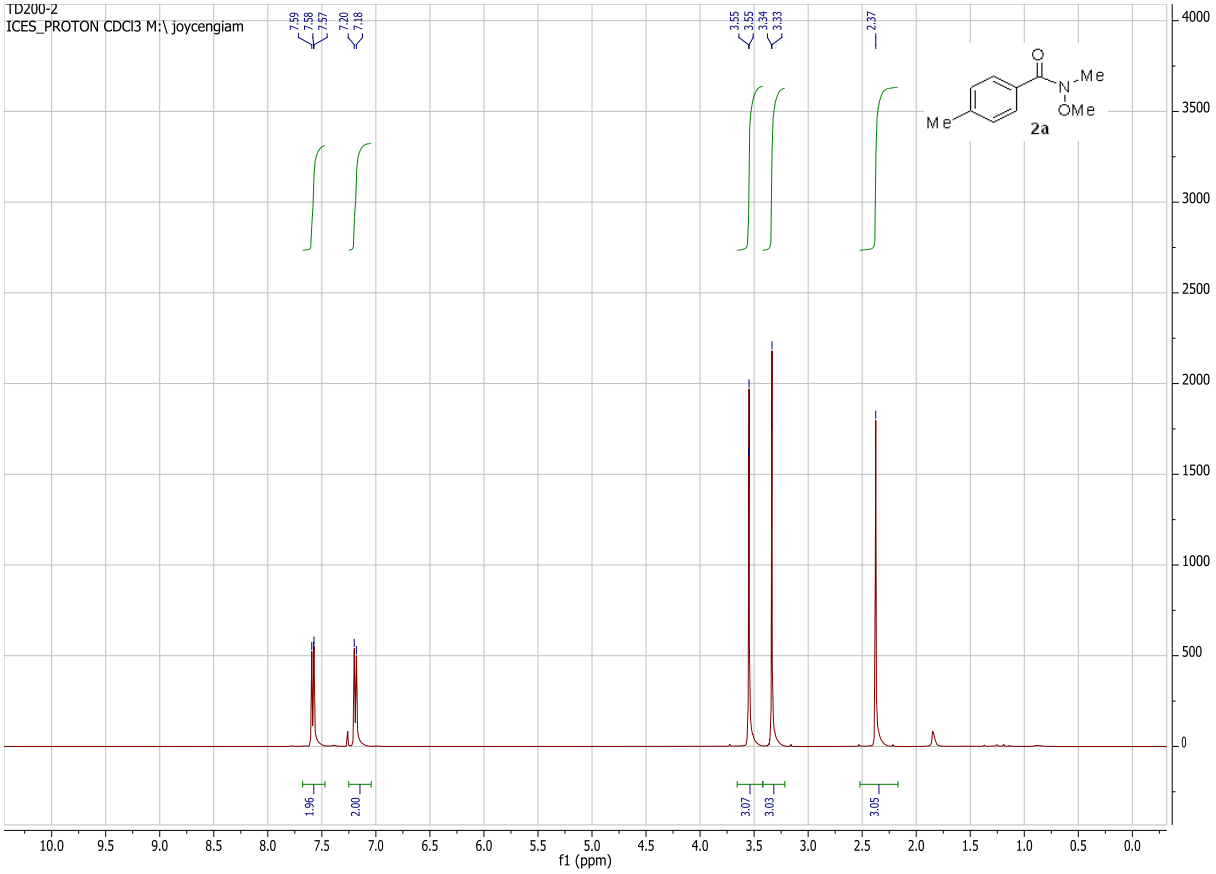
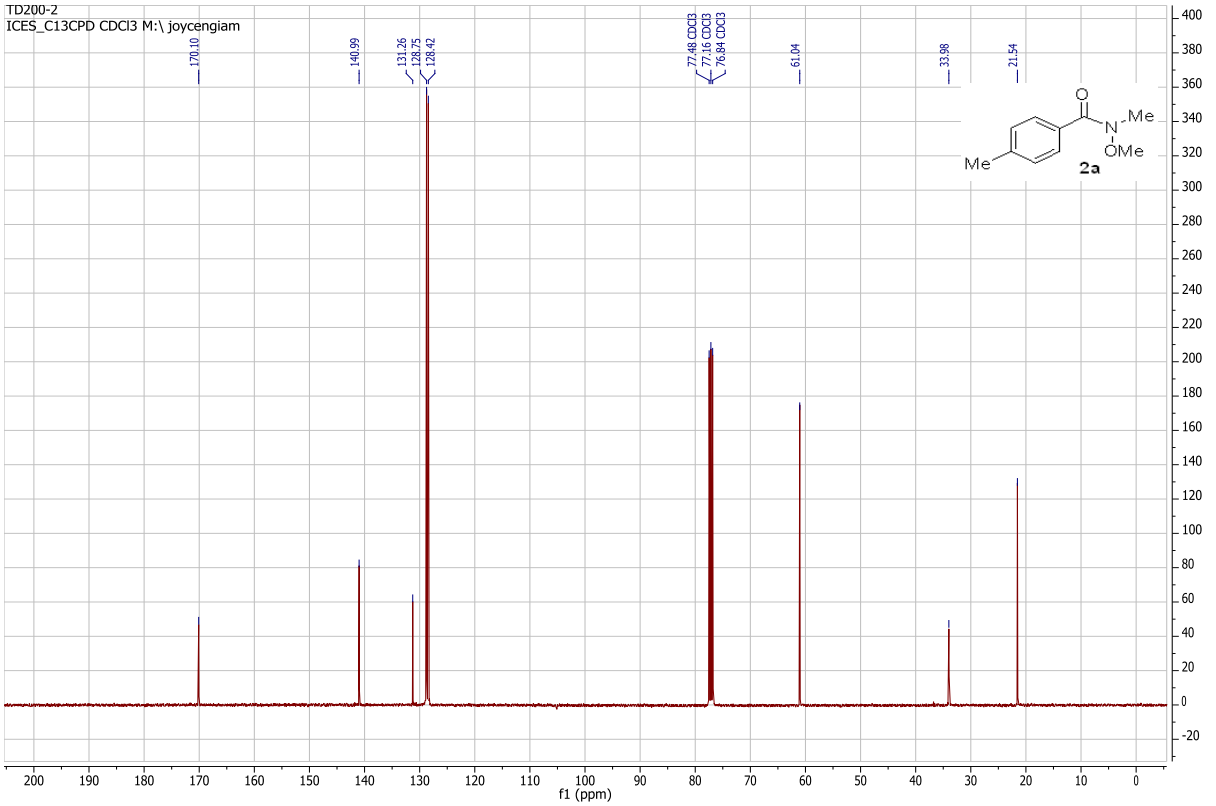
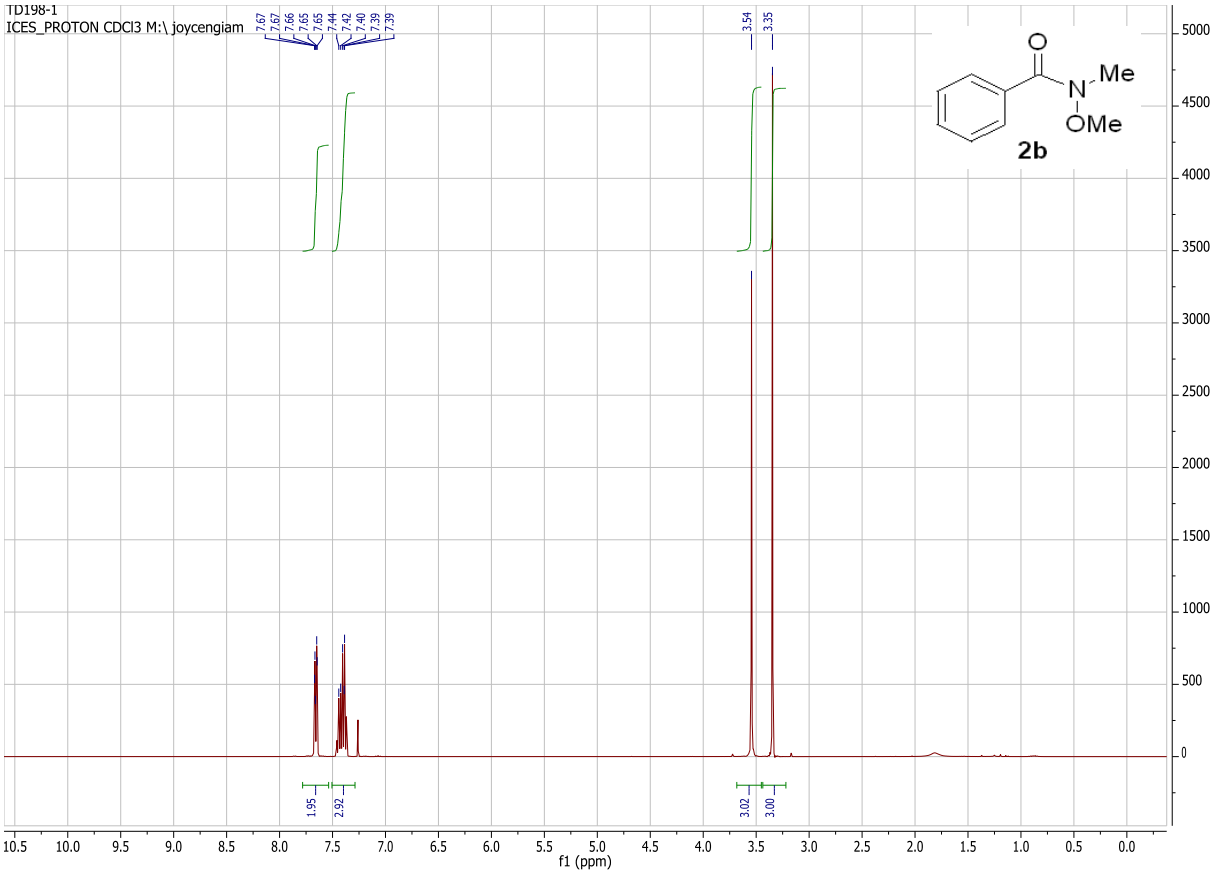
Contents

A: ^{1}H and ^{13}C NMR Spectra of Weinreb amide products



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2.92
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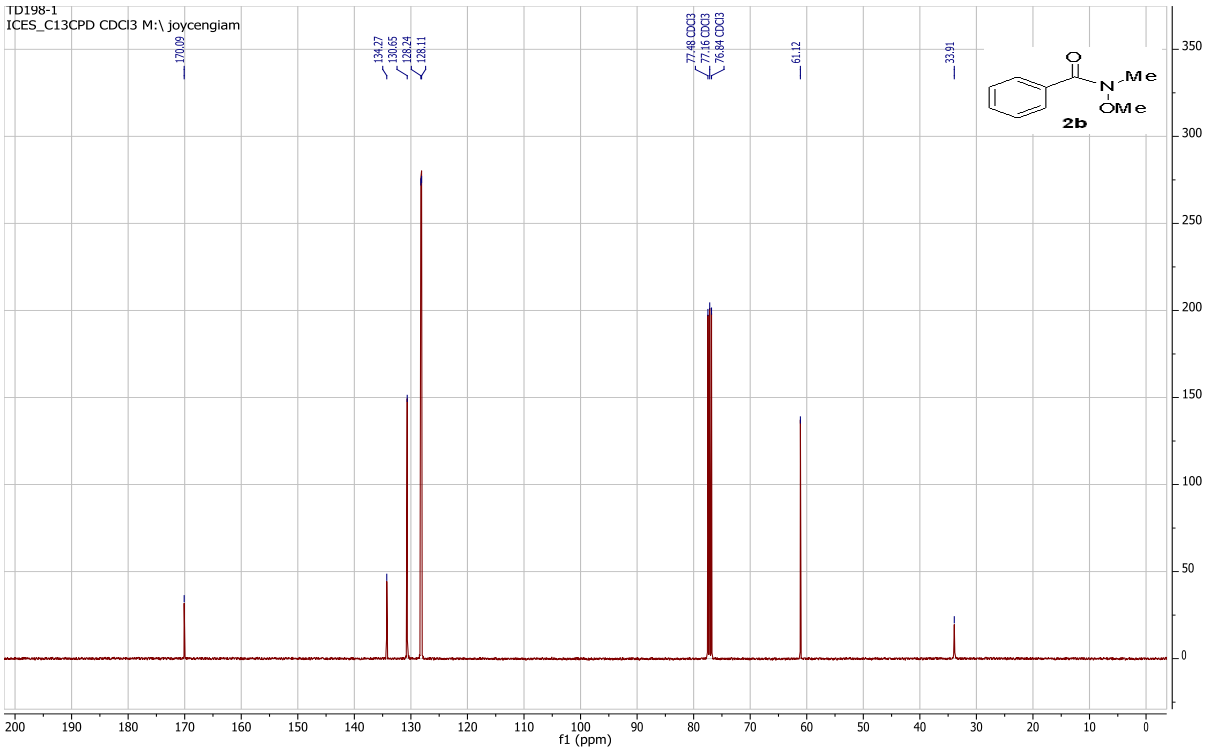
3.00
f1 (ppm)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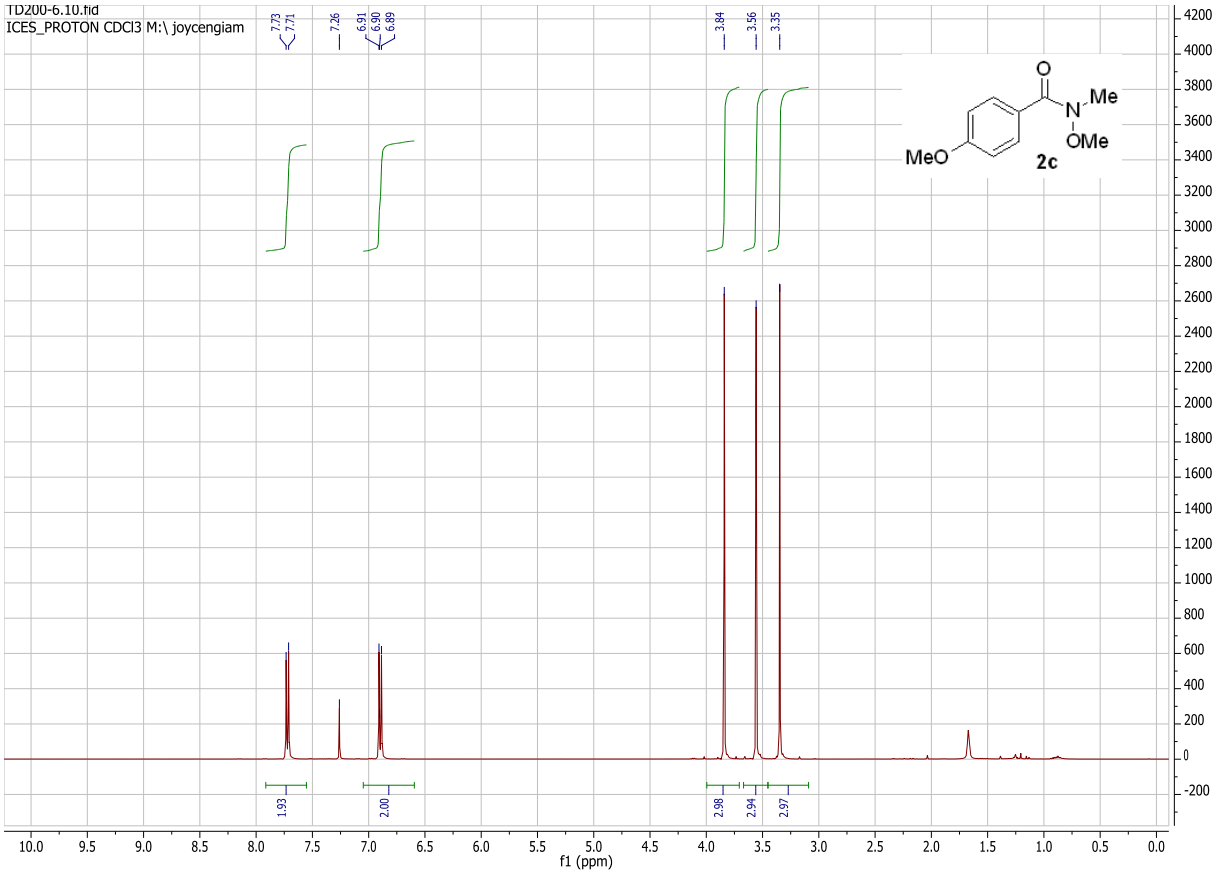
f1 (ppm)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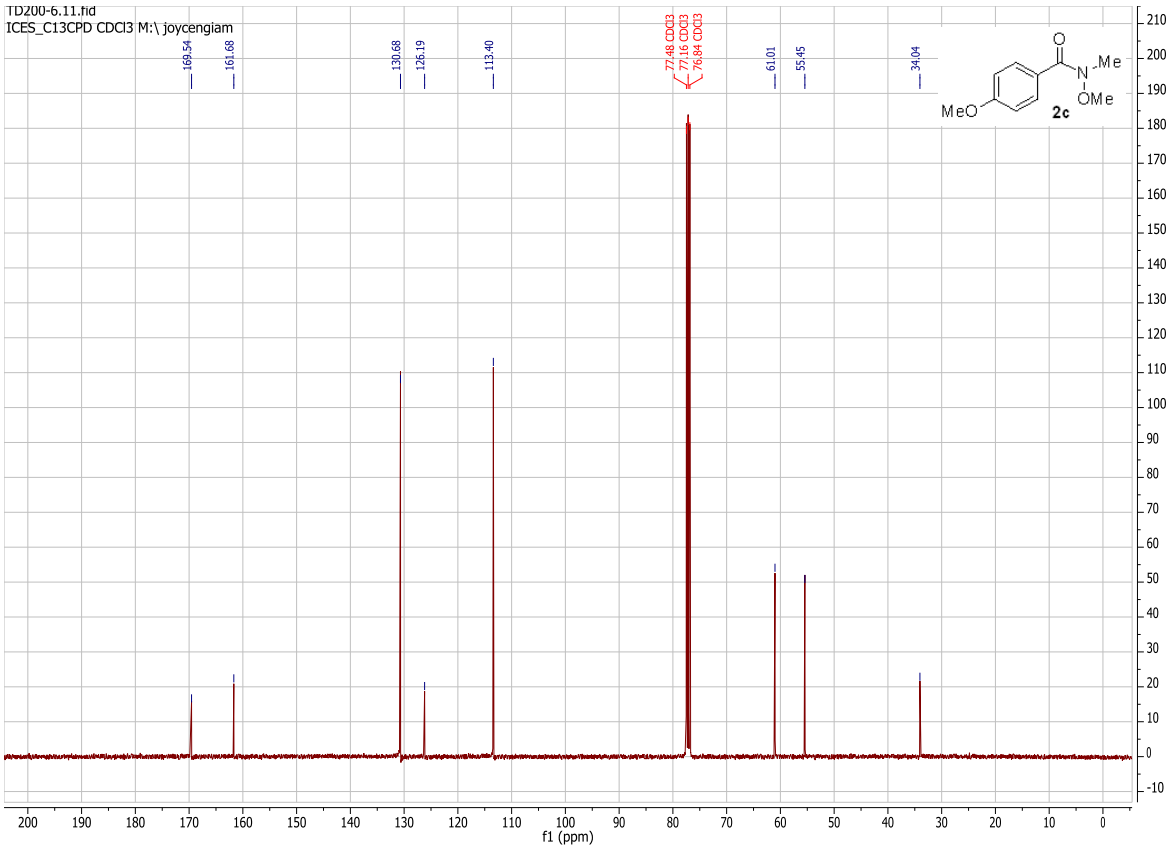
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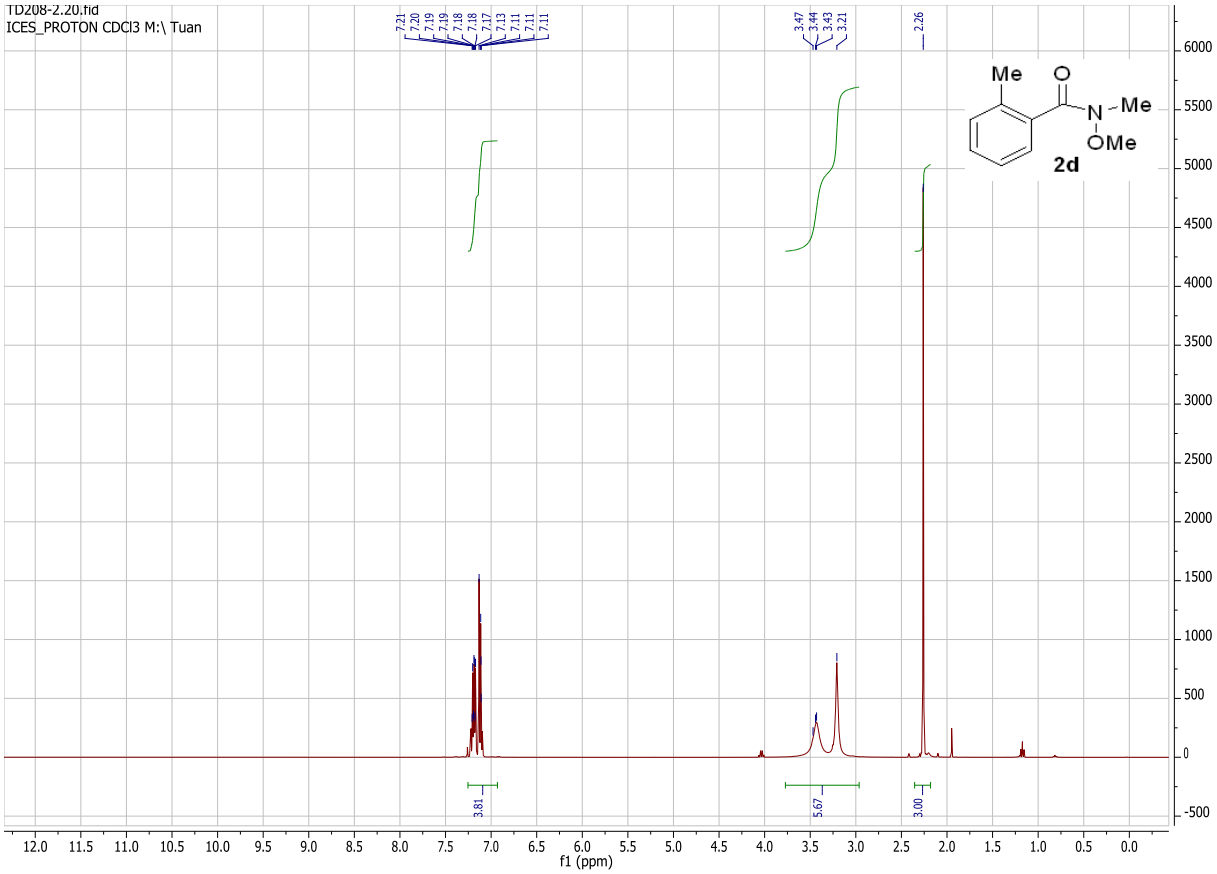
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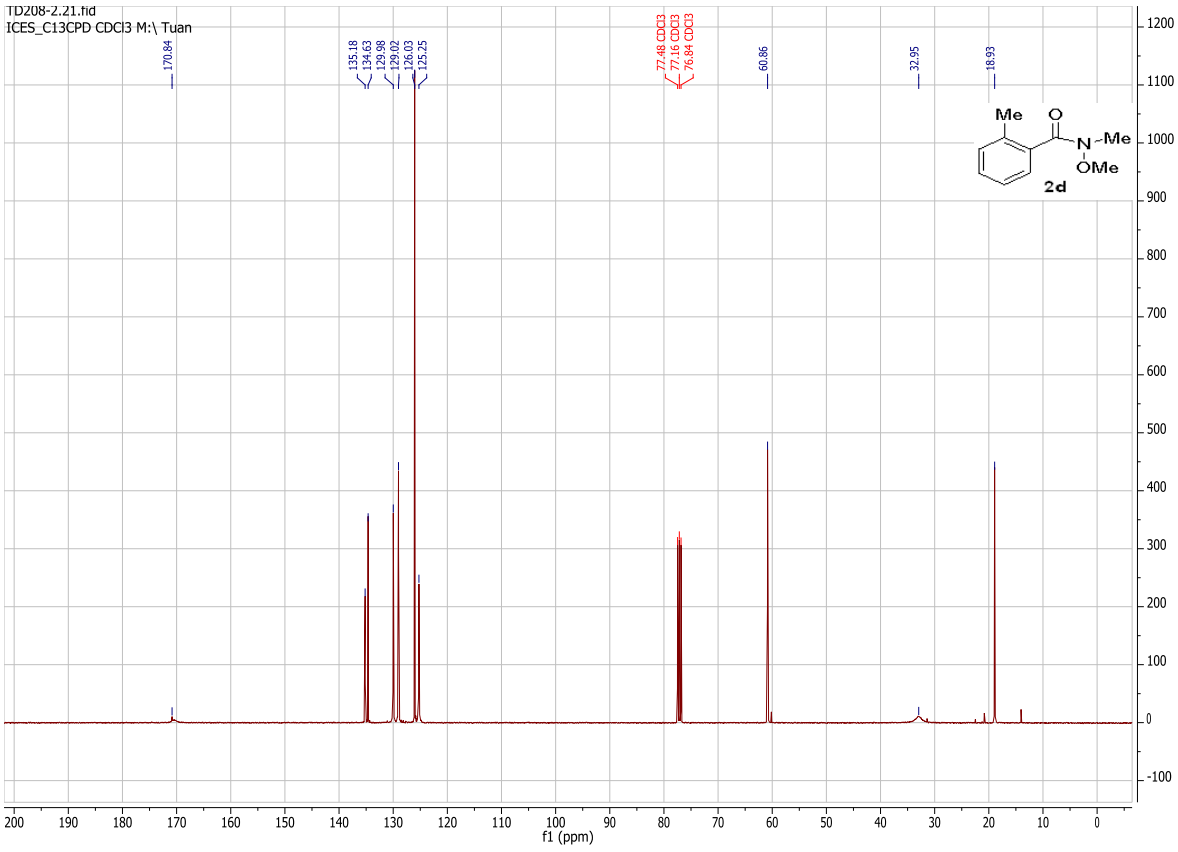
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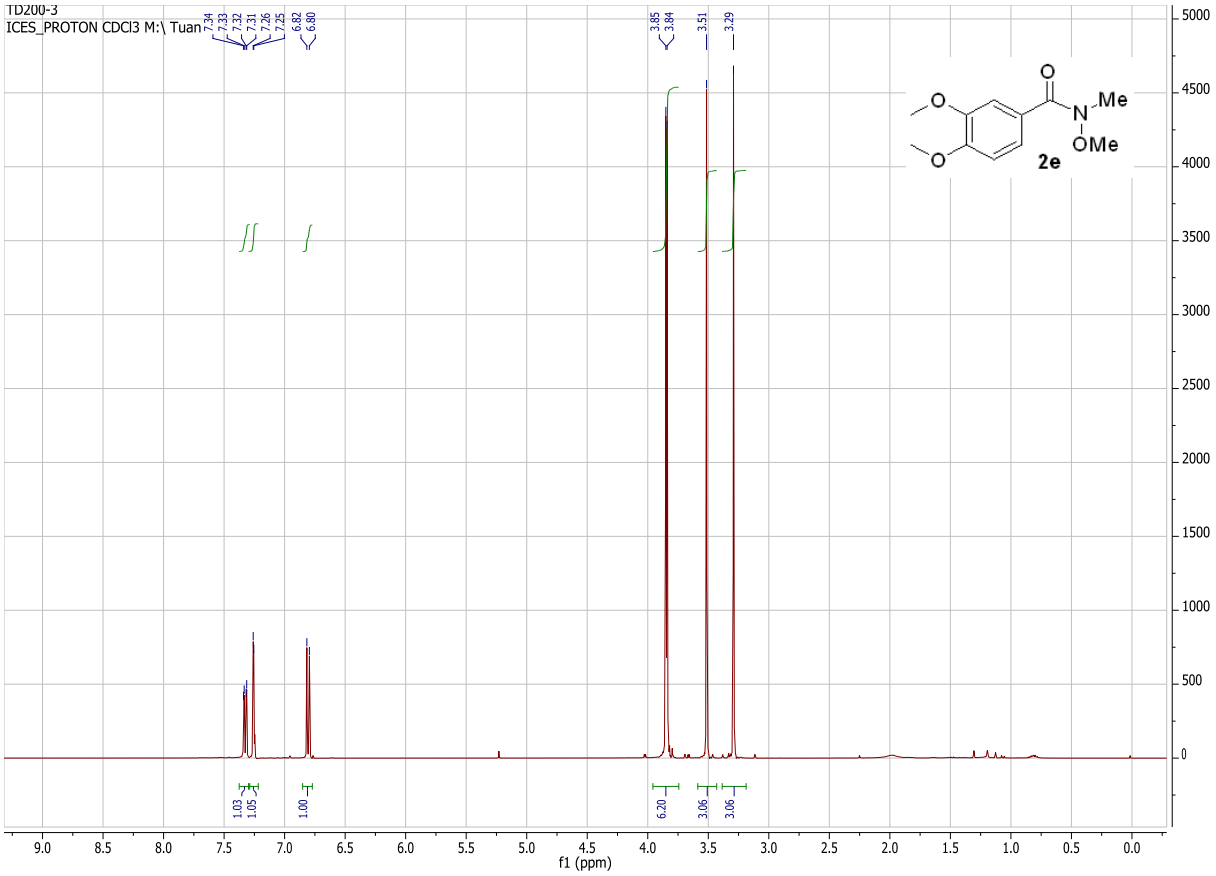
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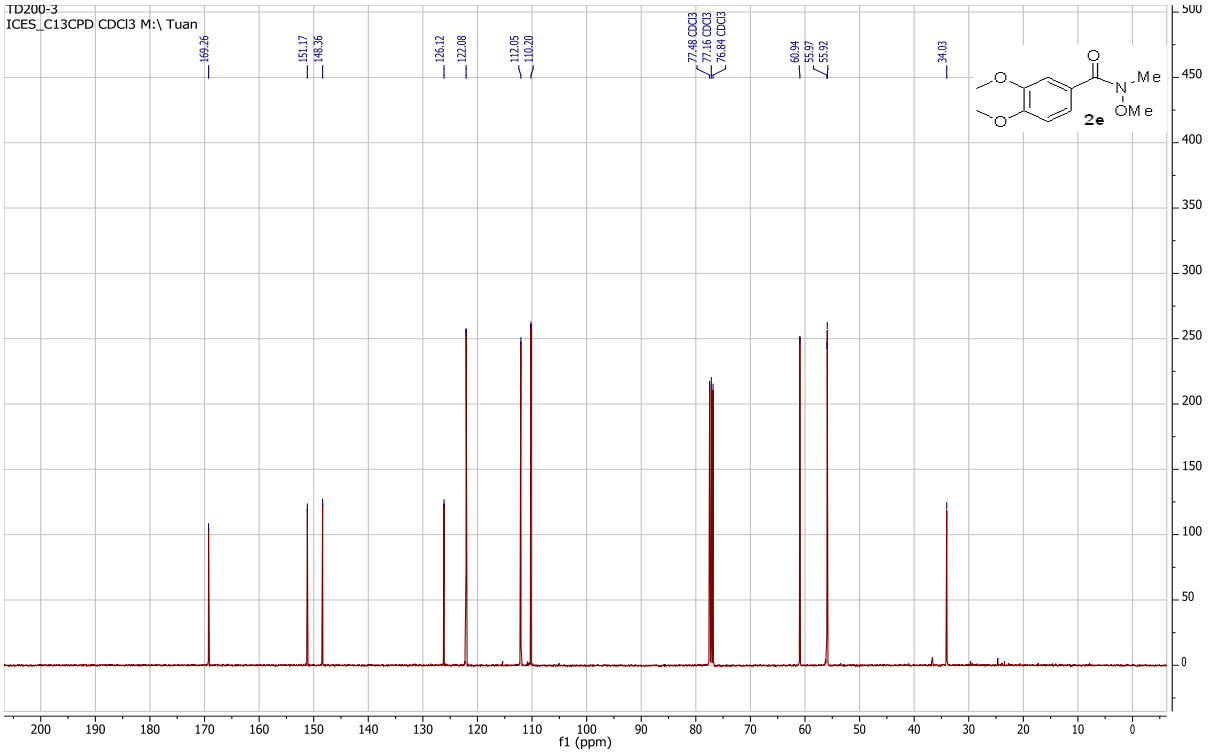
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f1 (ppm)
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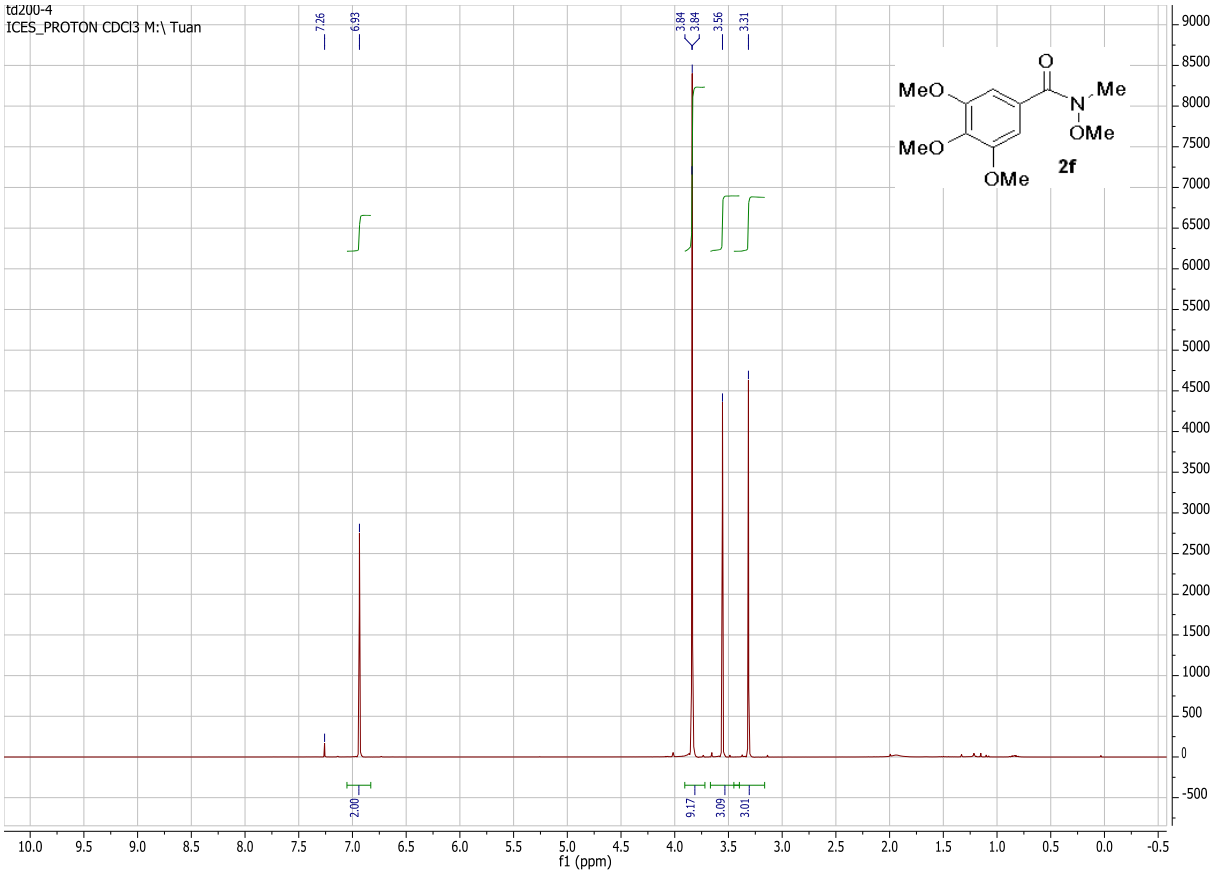
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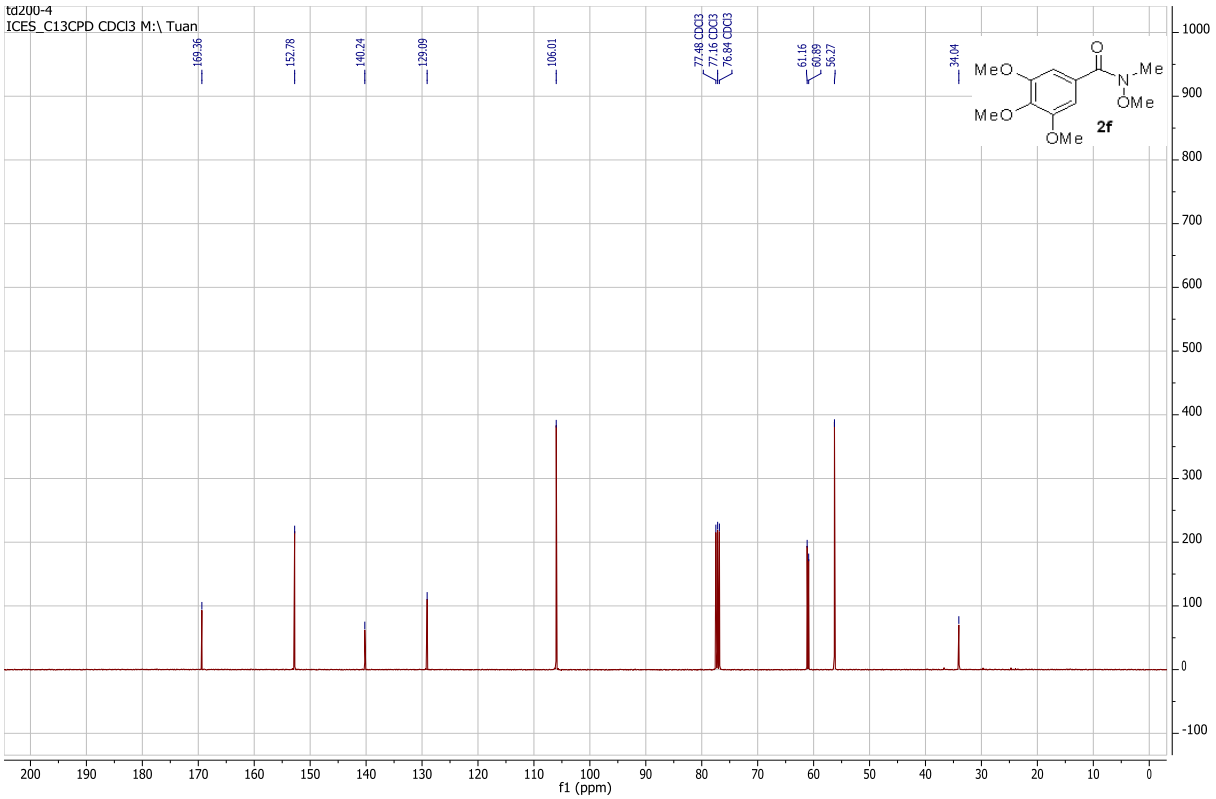
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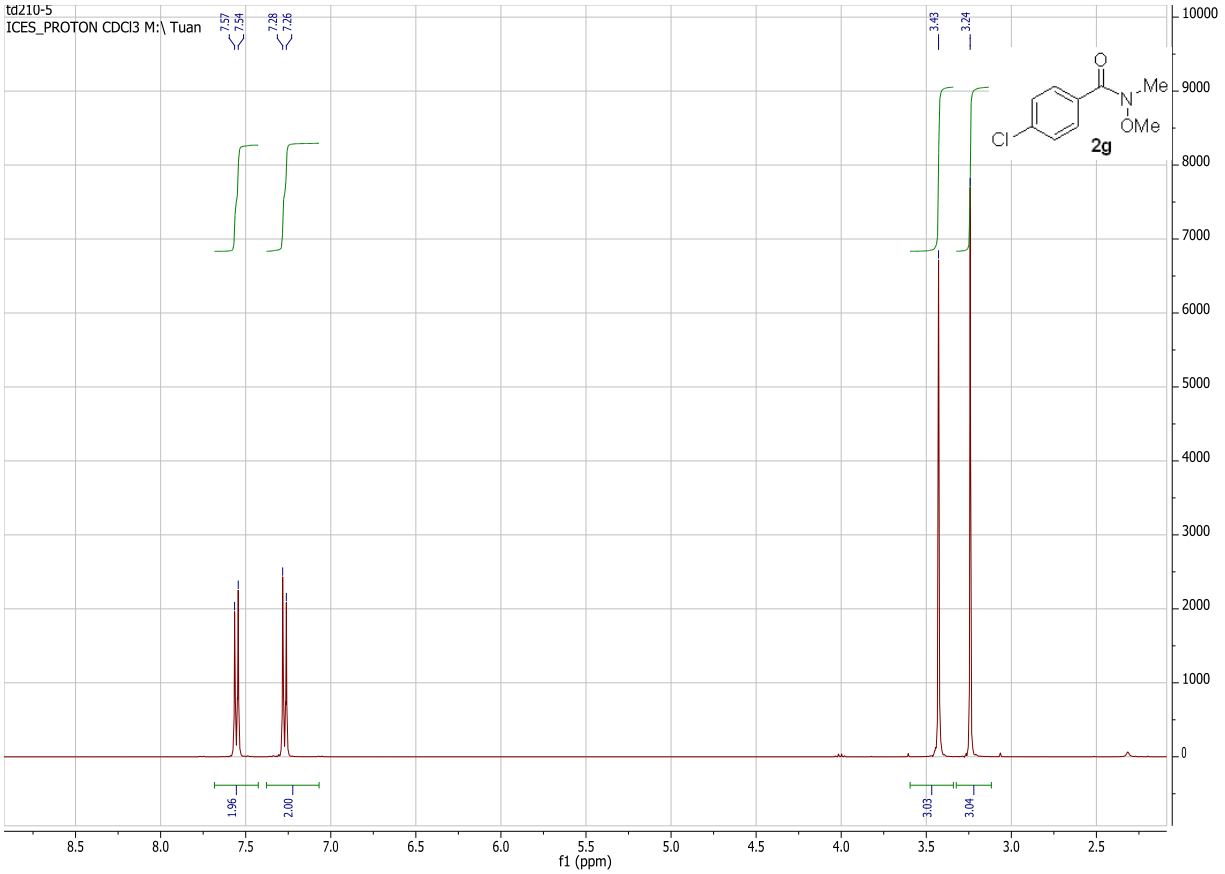
MeO
OMe
N
Me
OMe
O
2f
f1 (ppm)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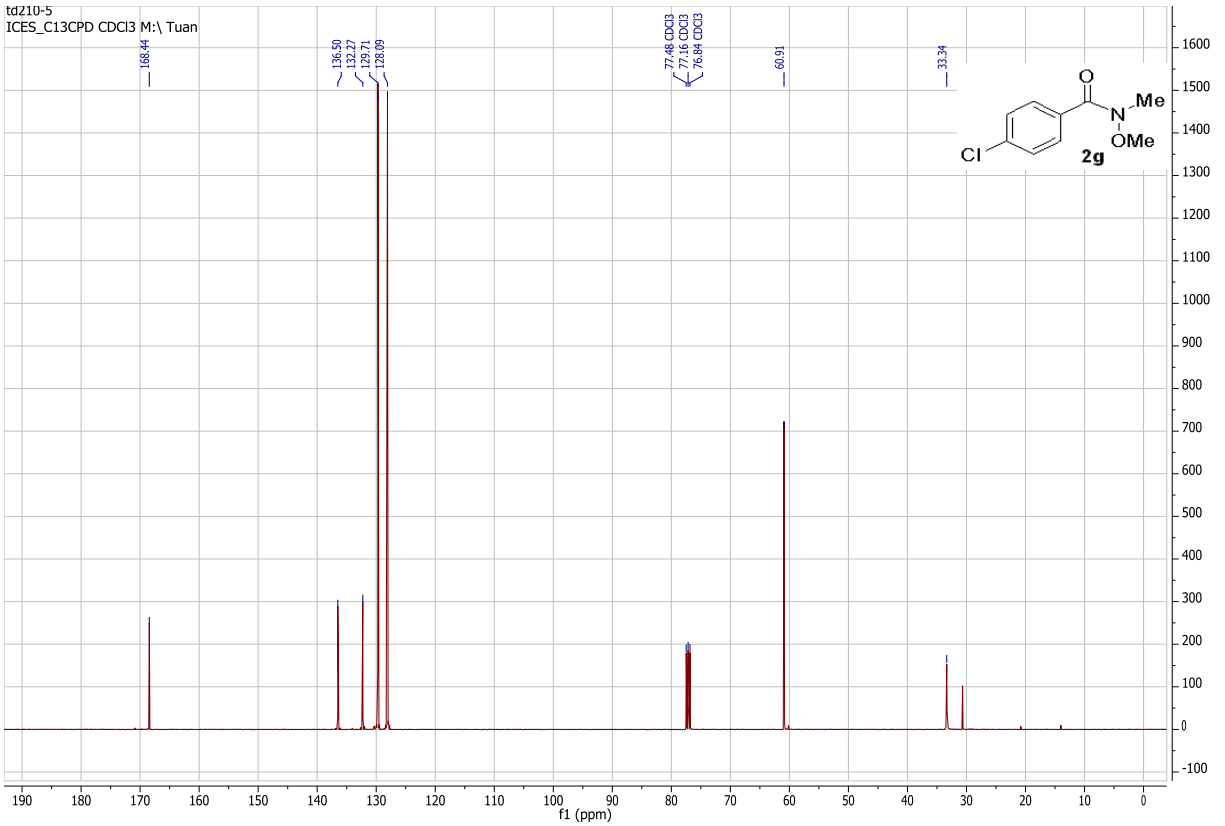
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Cl
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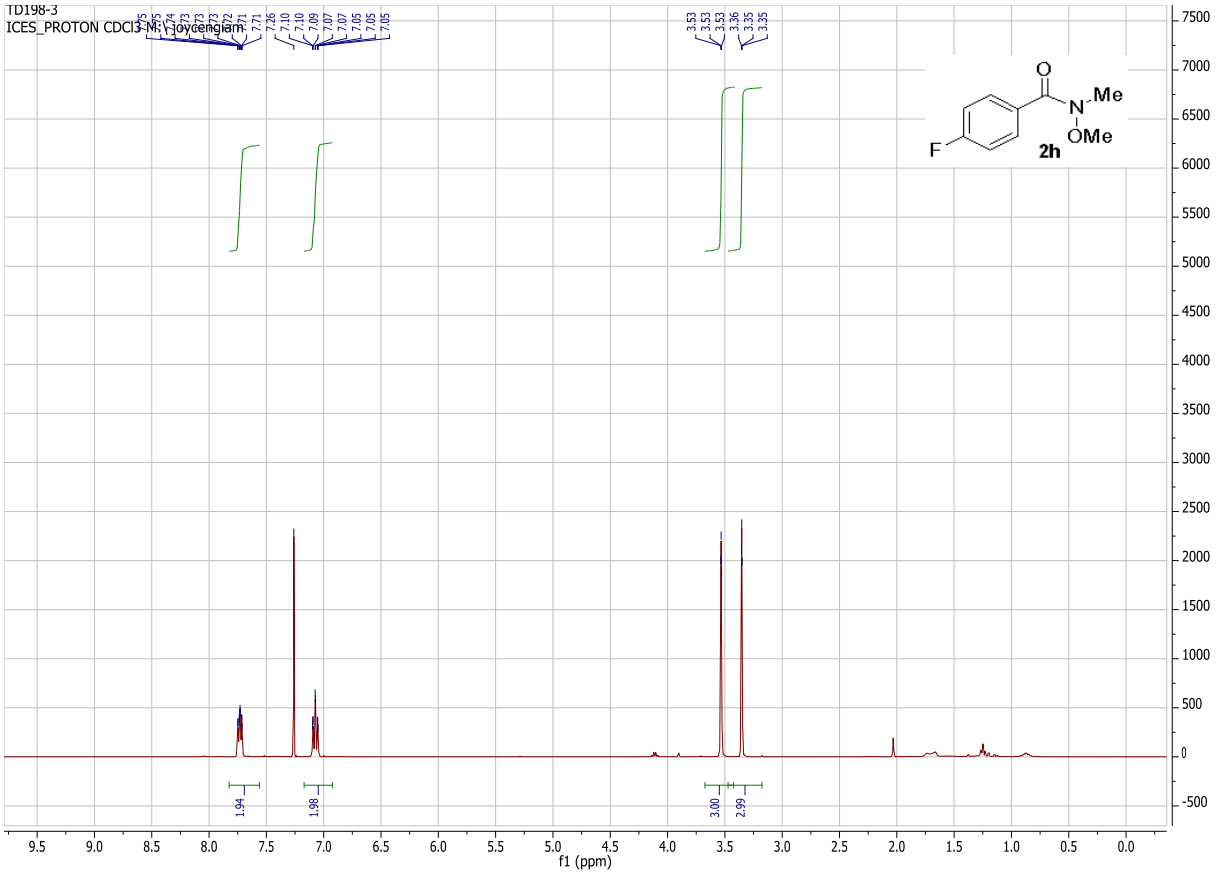
f1 (ppm)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OMe
F
2h
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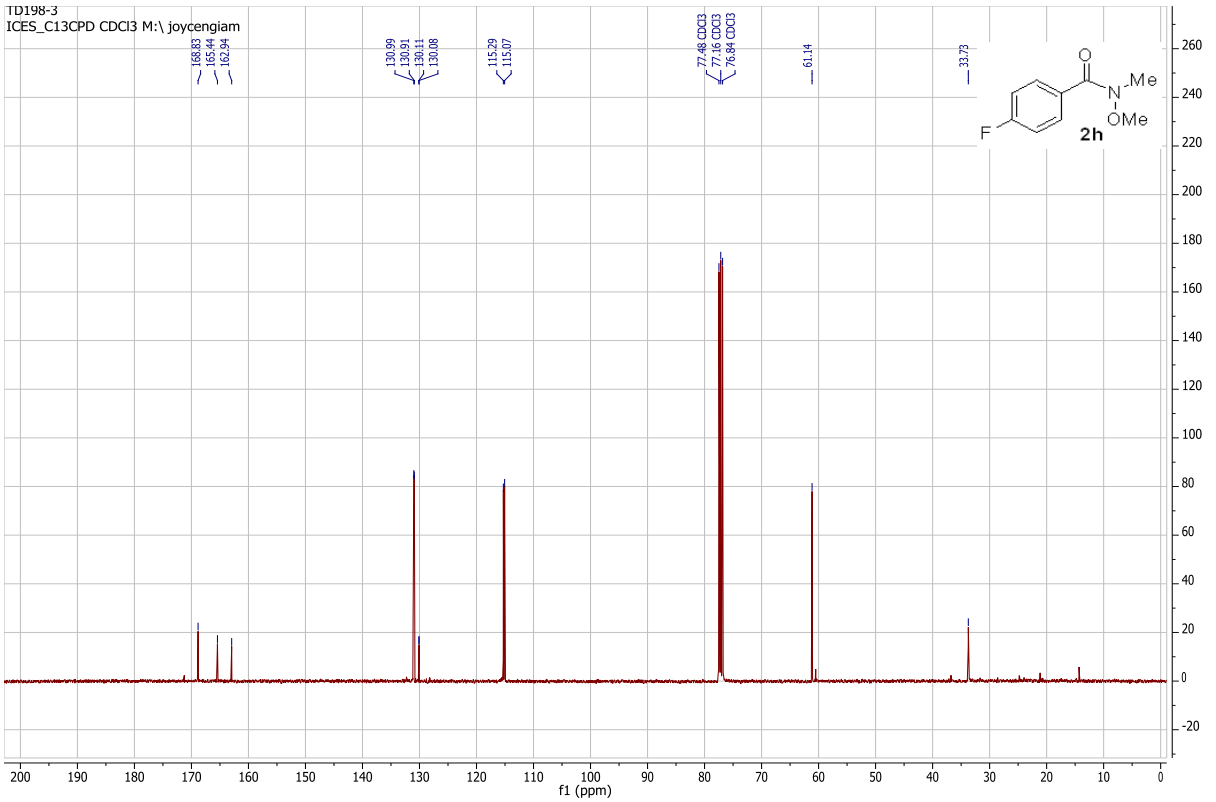
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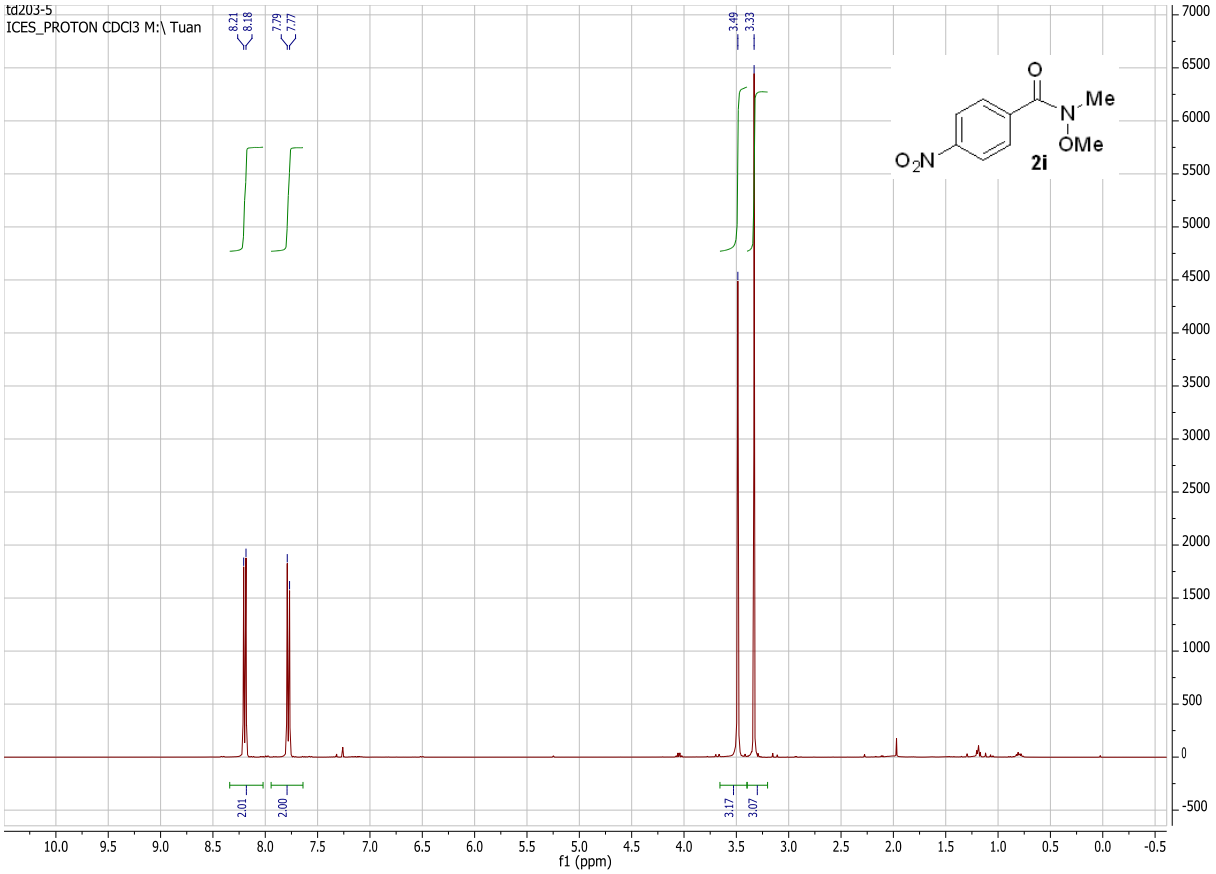
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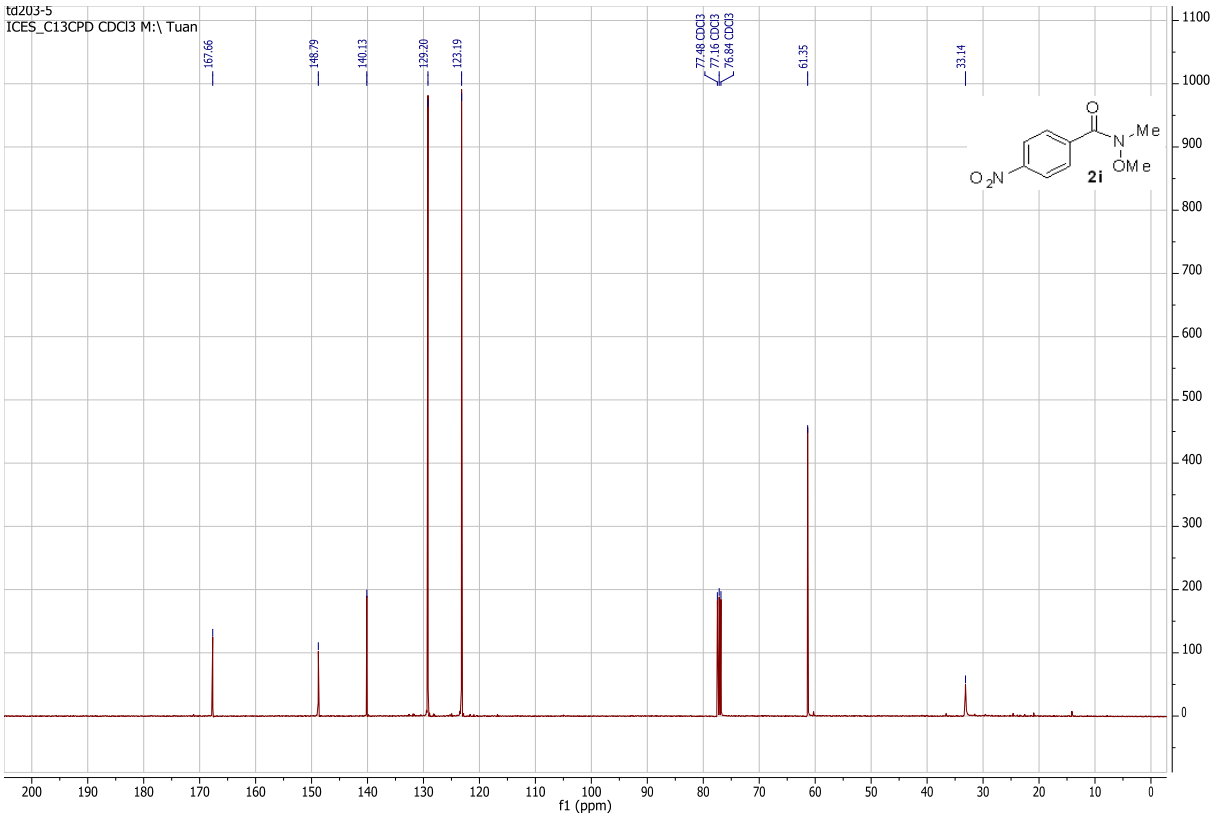
f1 (ppm)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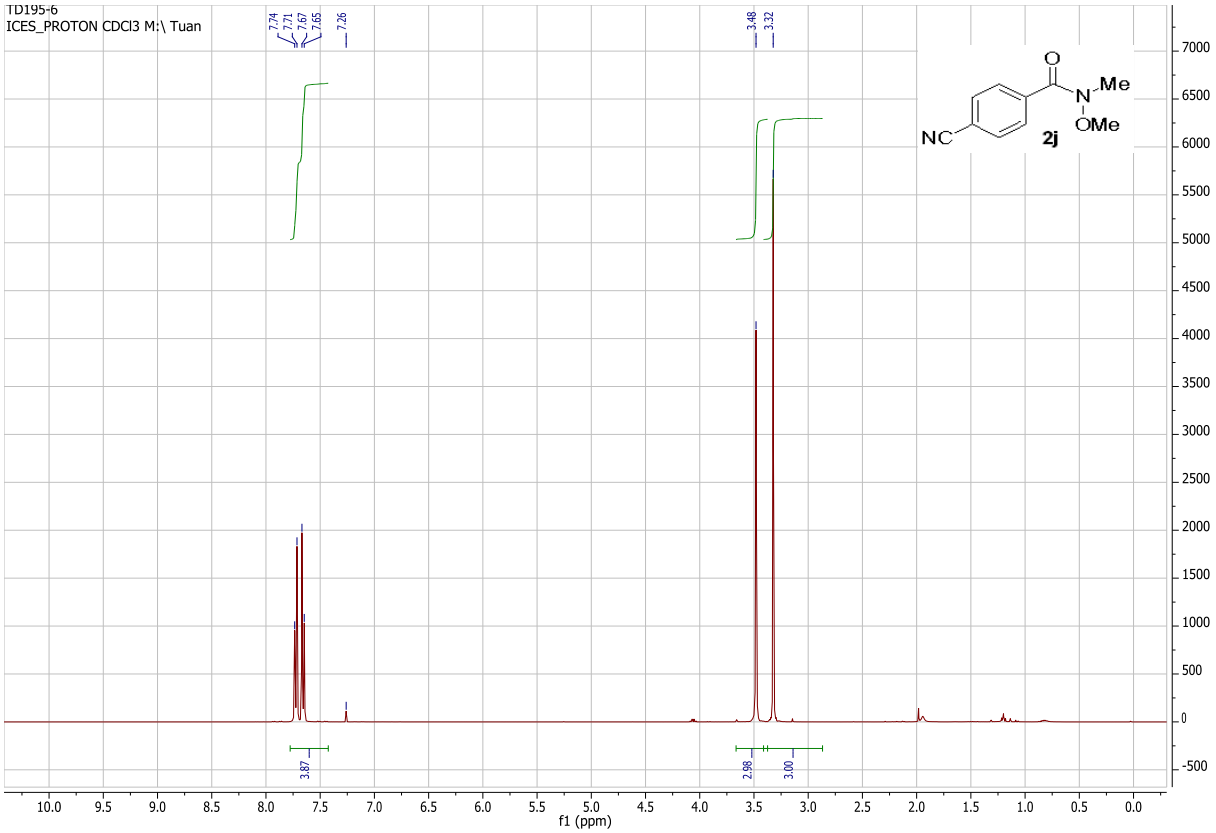
2i
f1 (ppm)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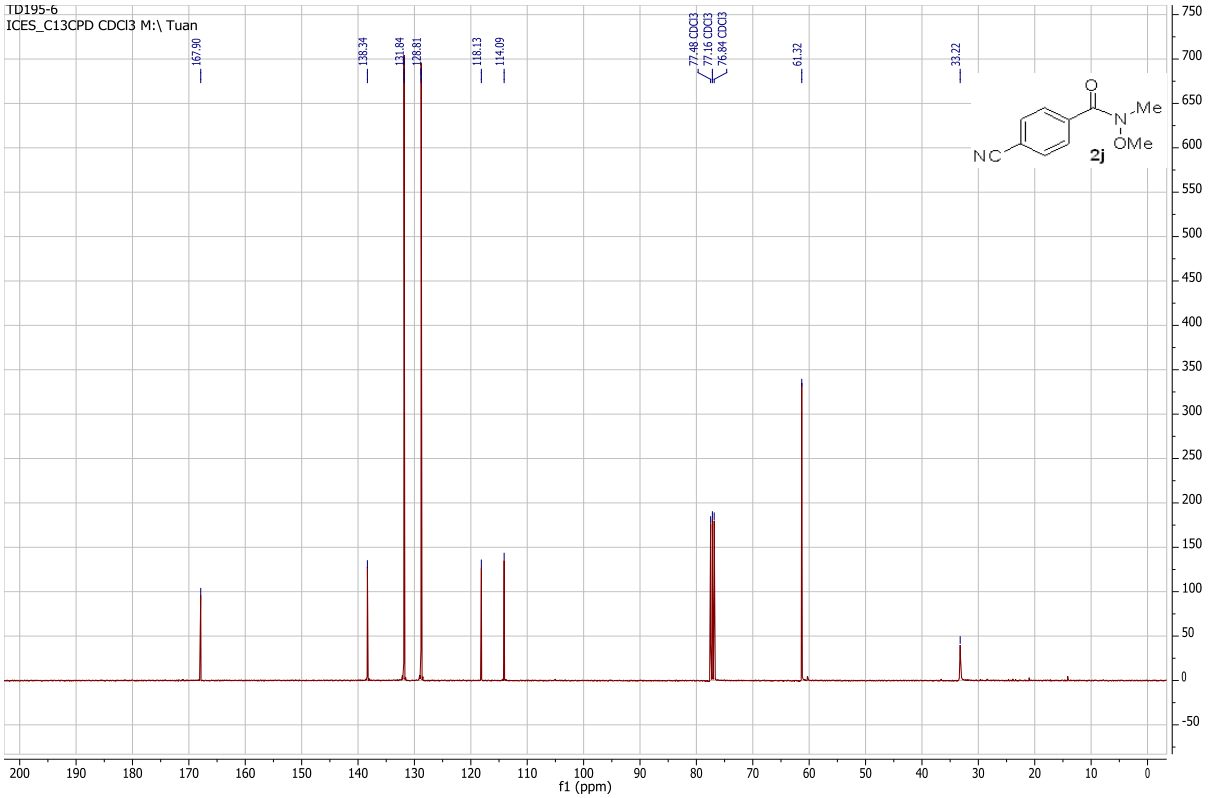
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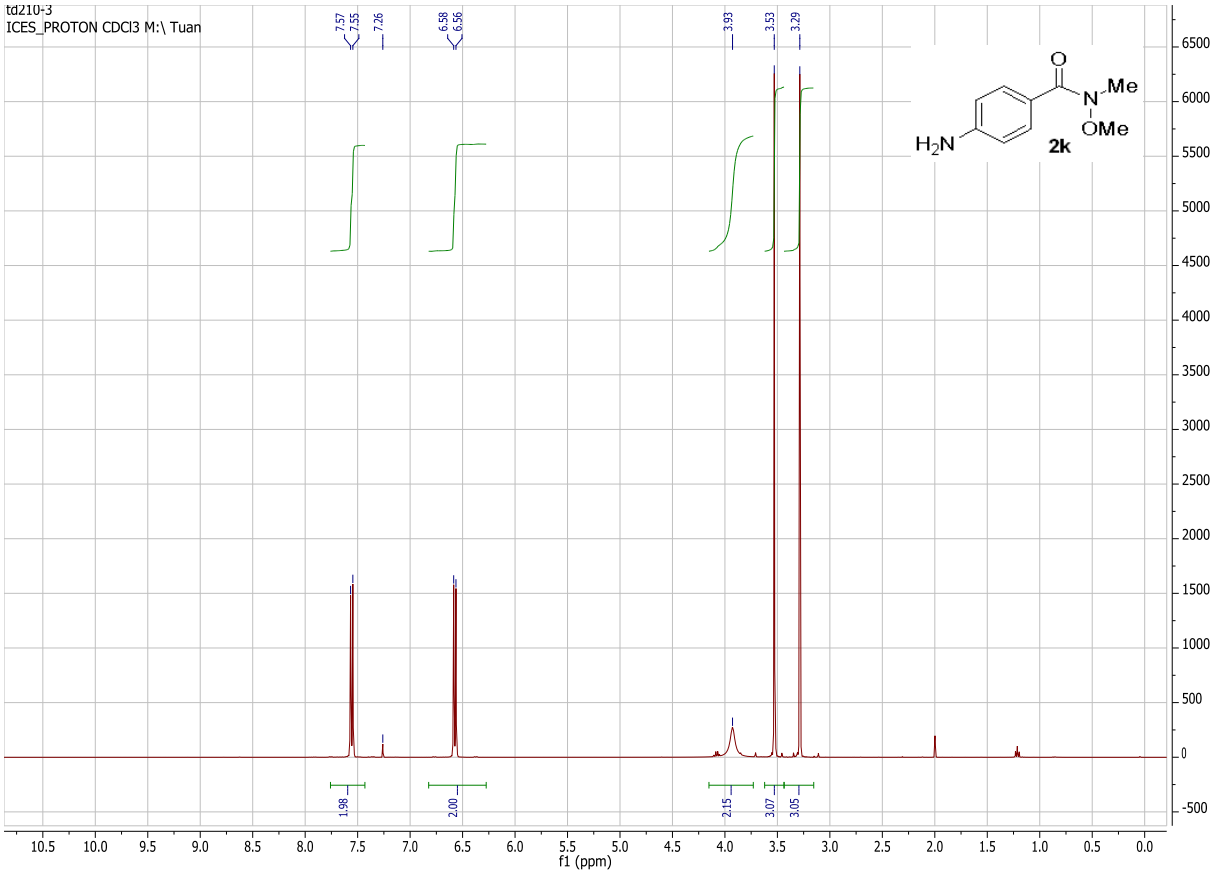
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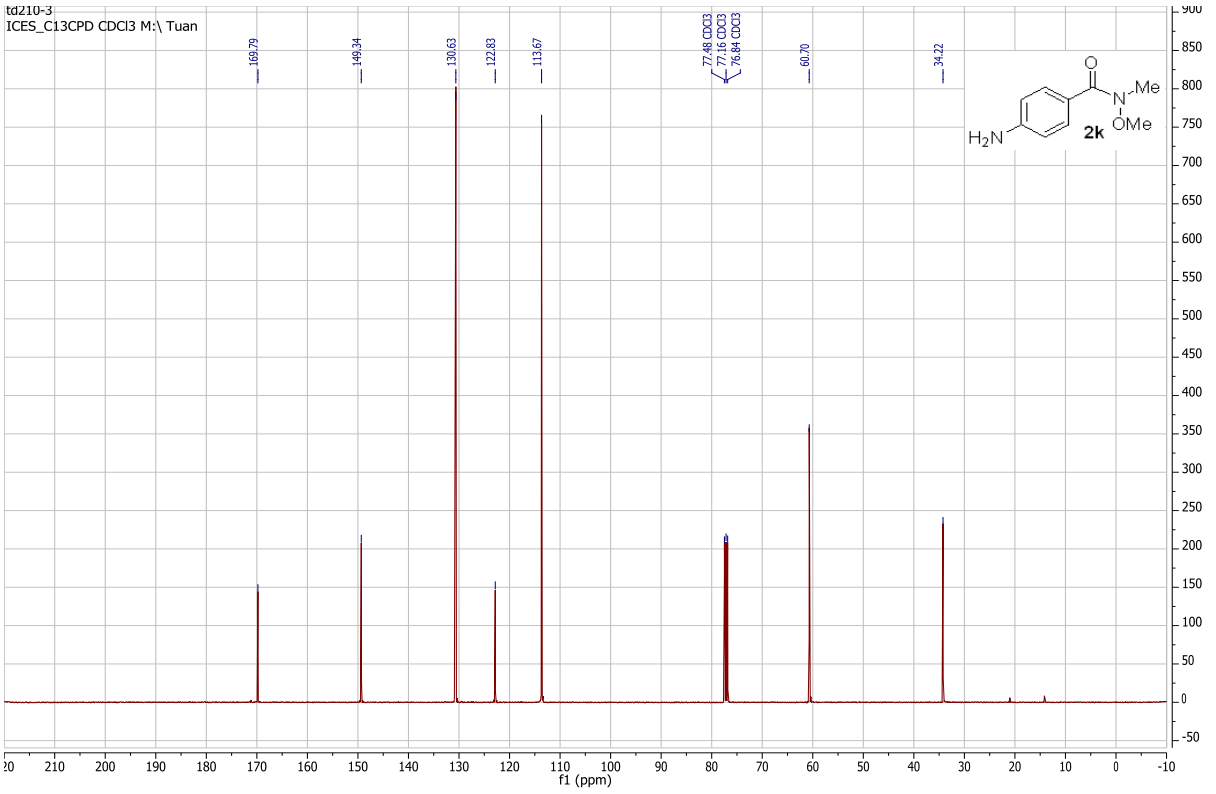
N
Me
OMe
H2N
2k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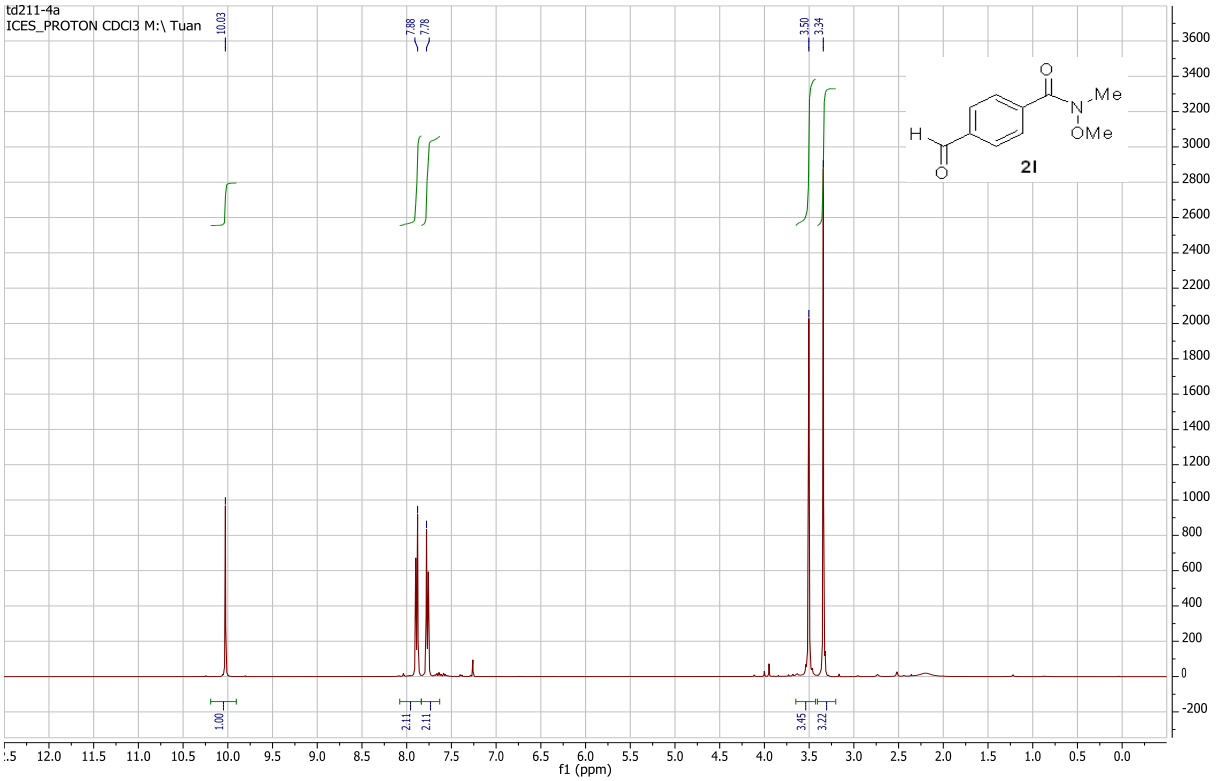
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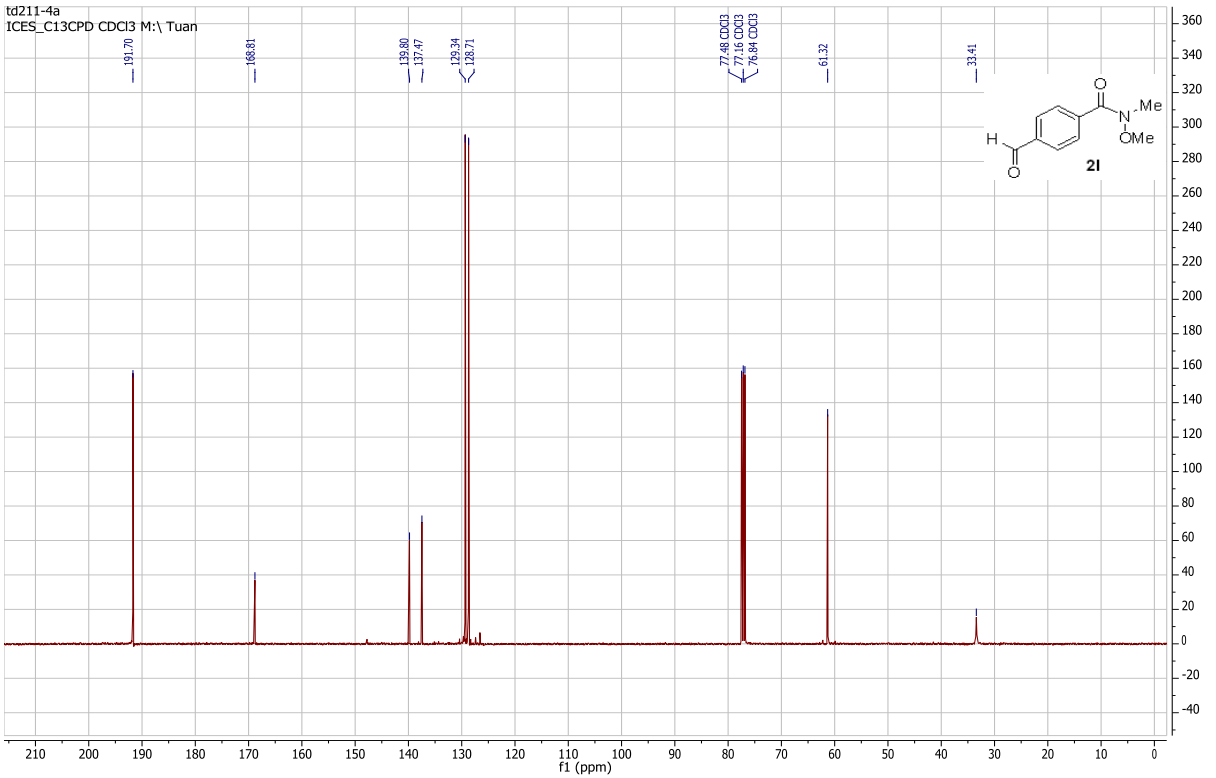
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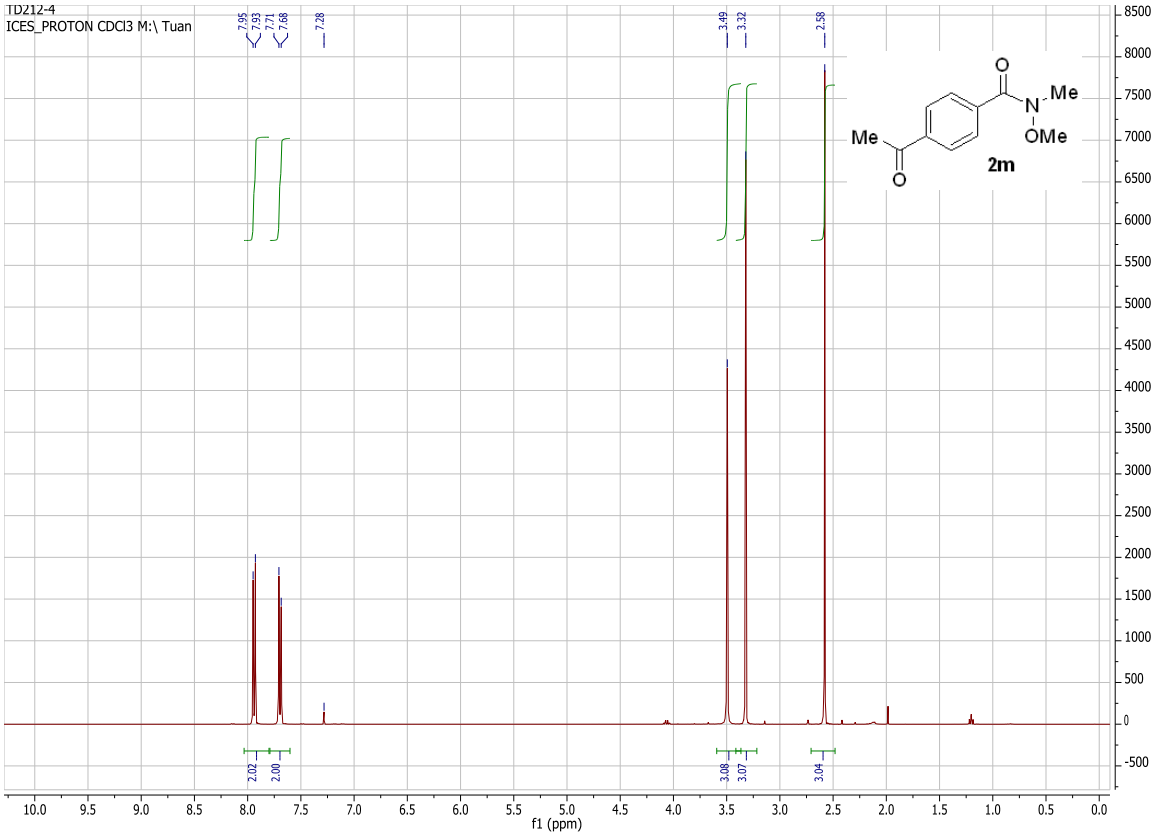
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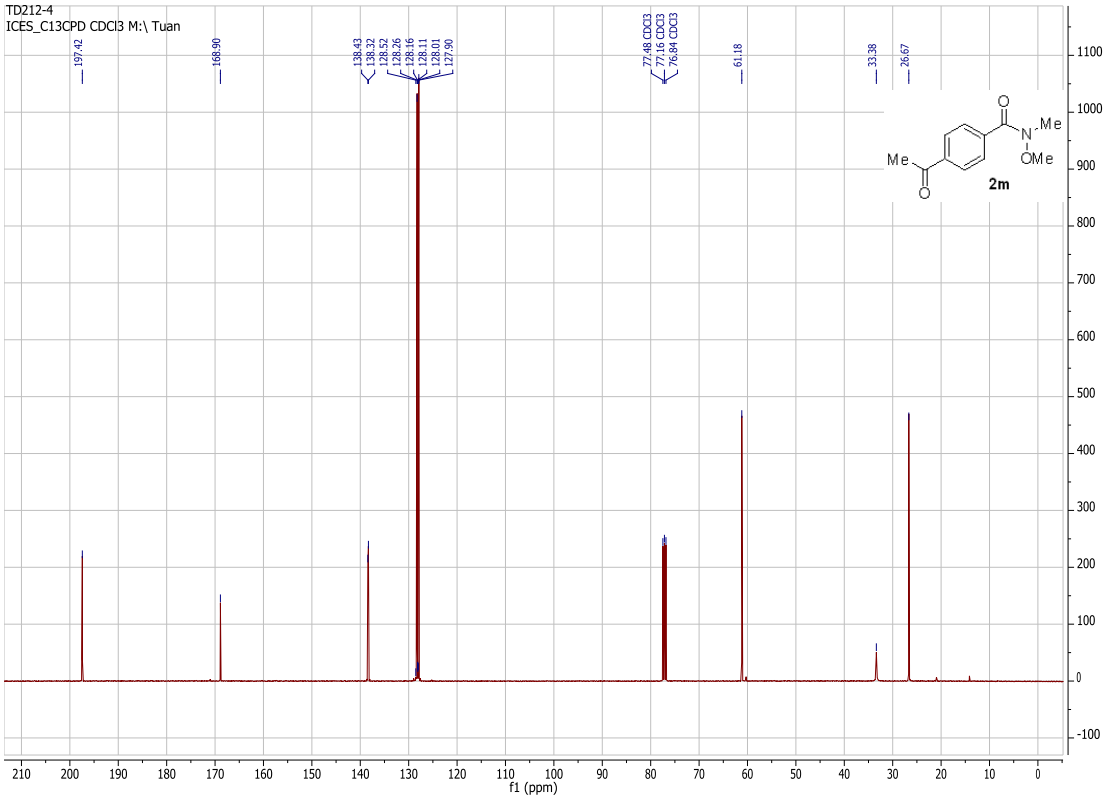
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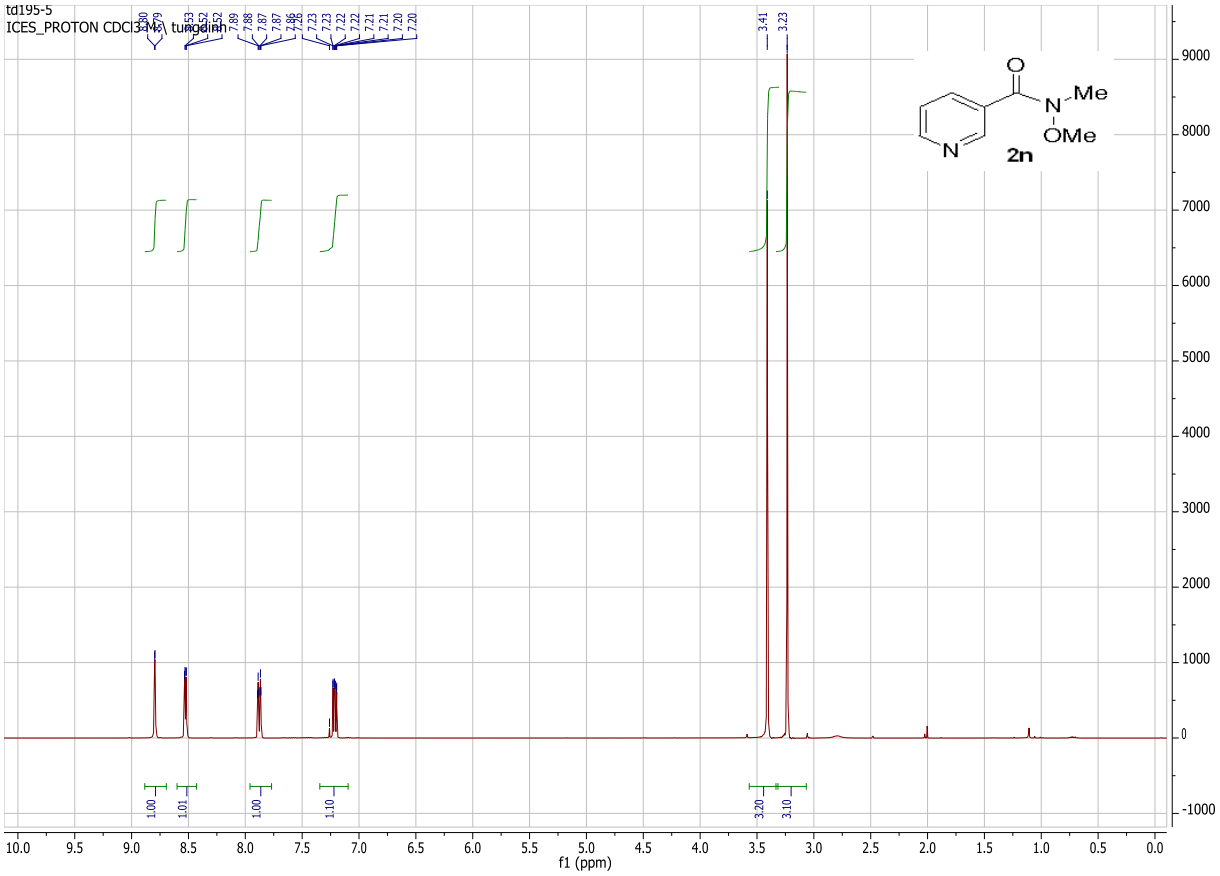
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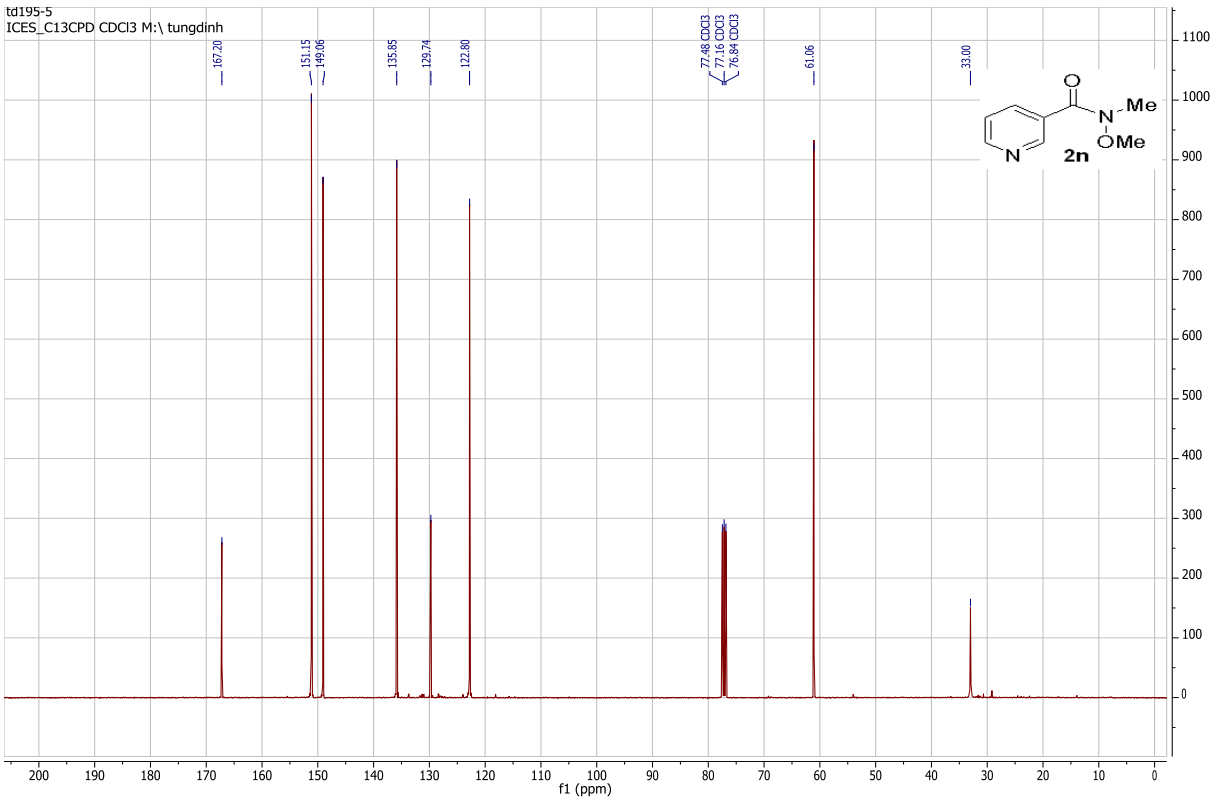
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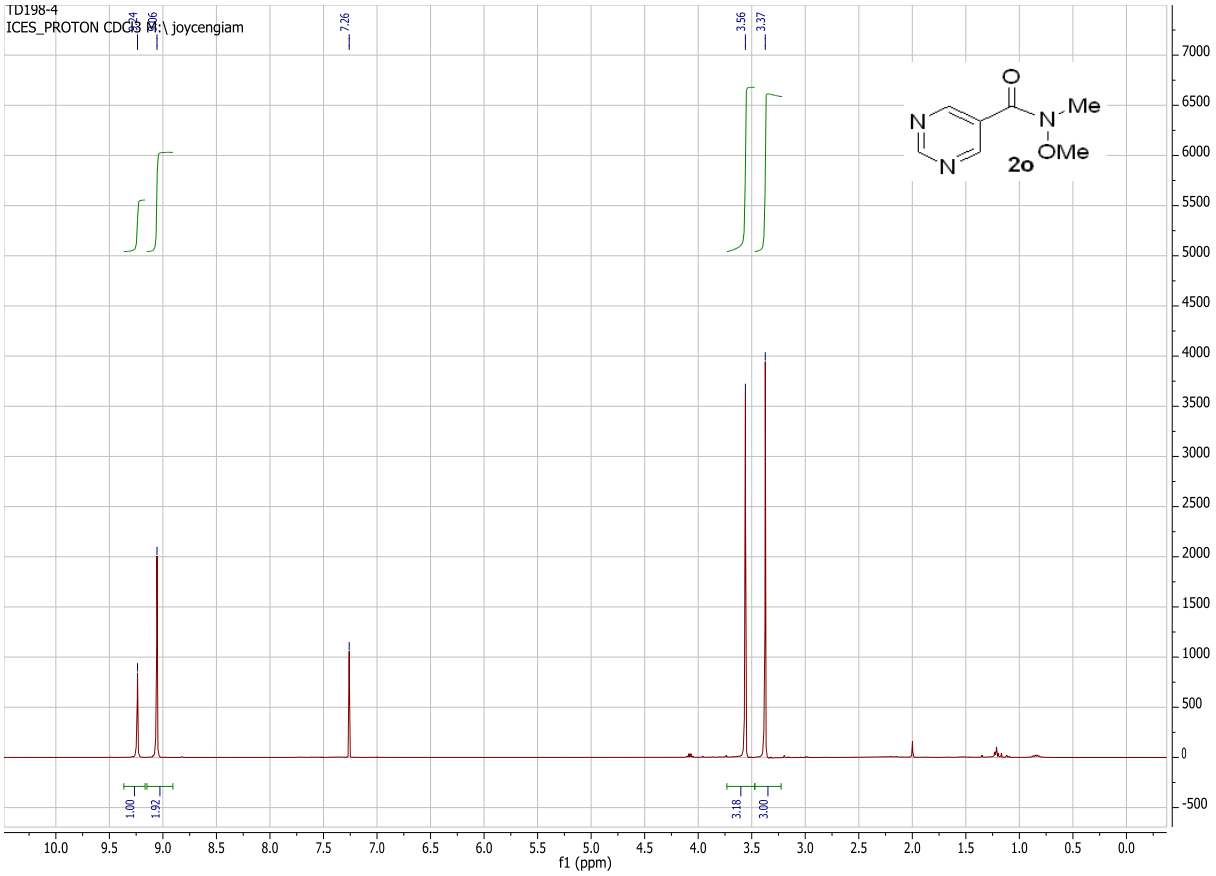
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3.00
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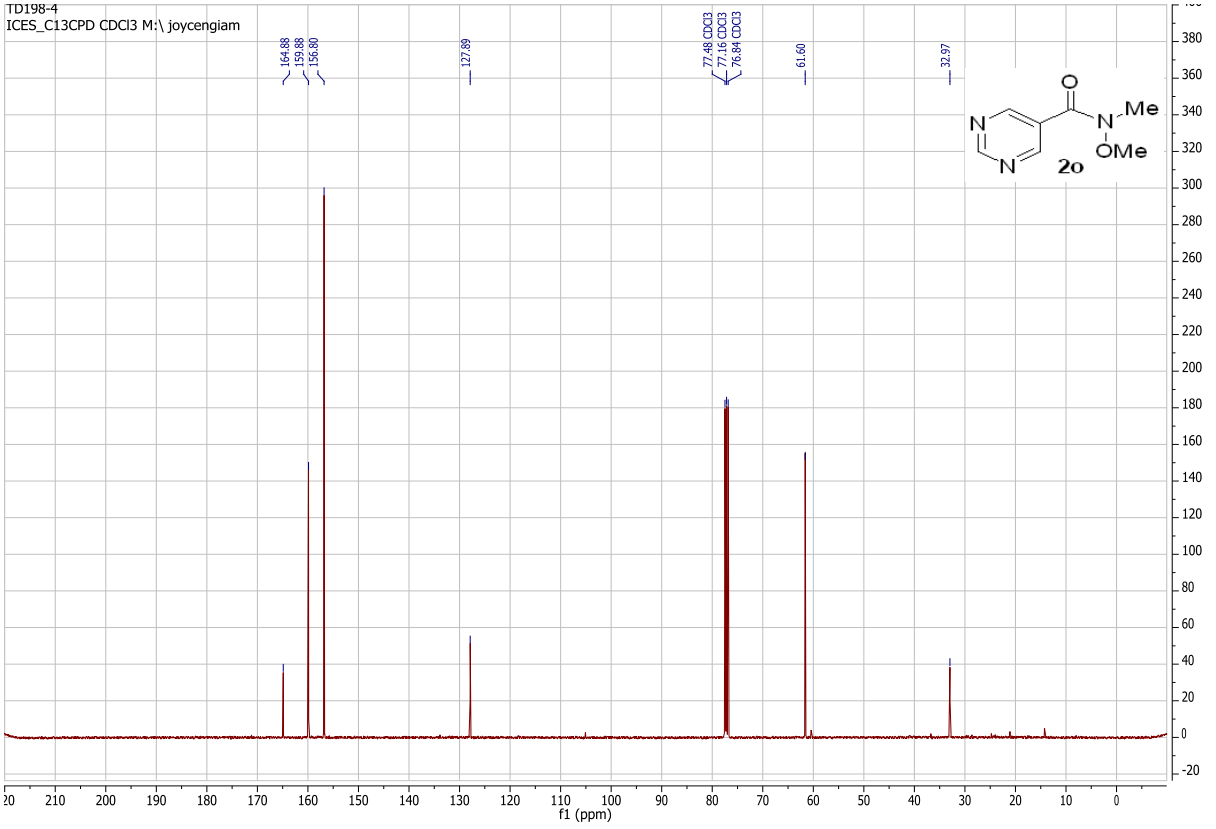
2o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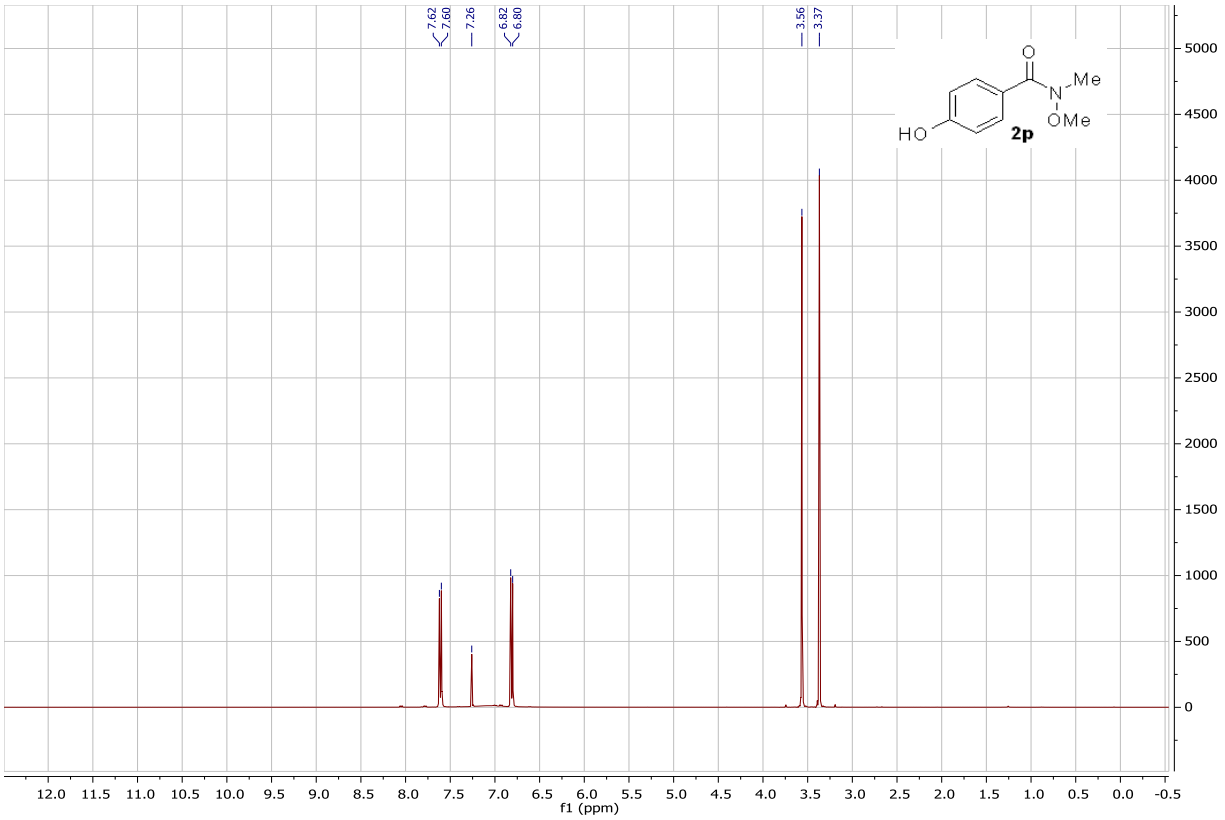
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OMe
HO
2p
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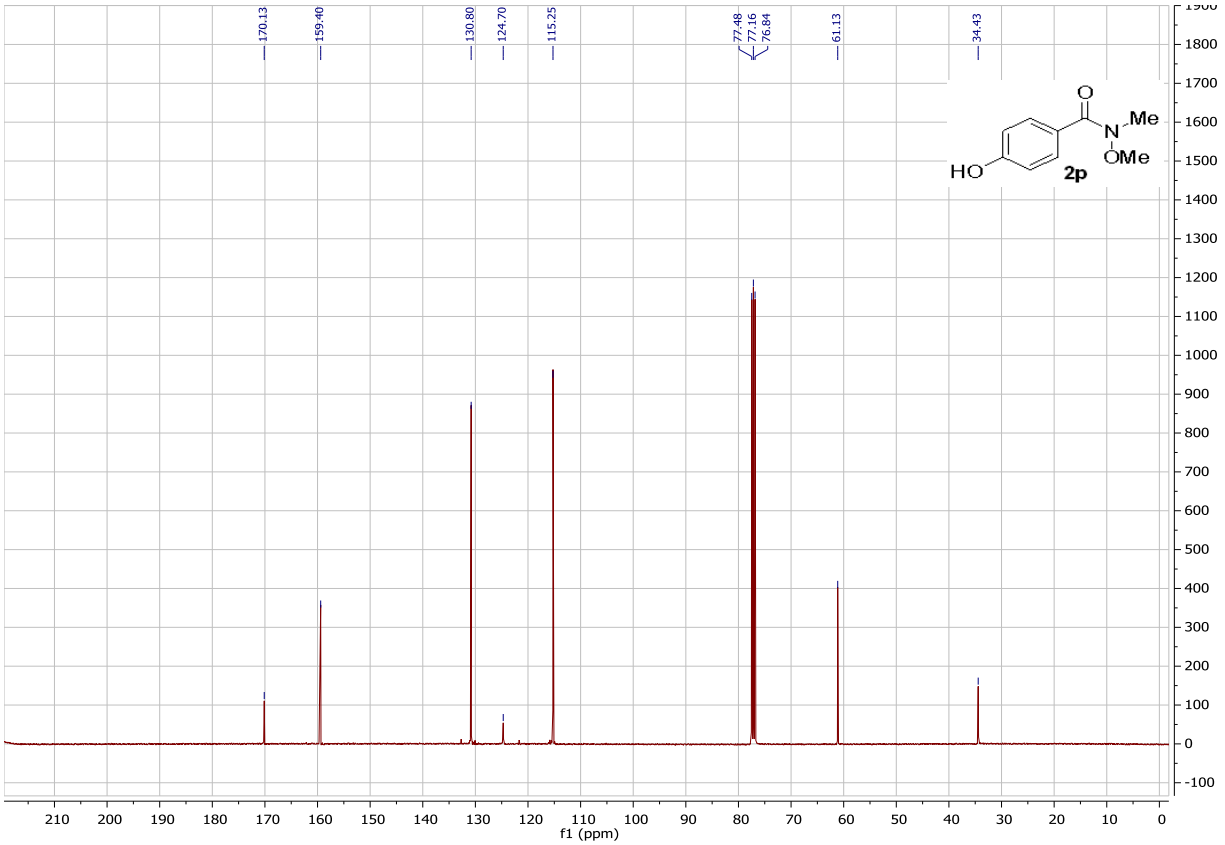
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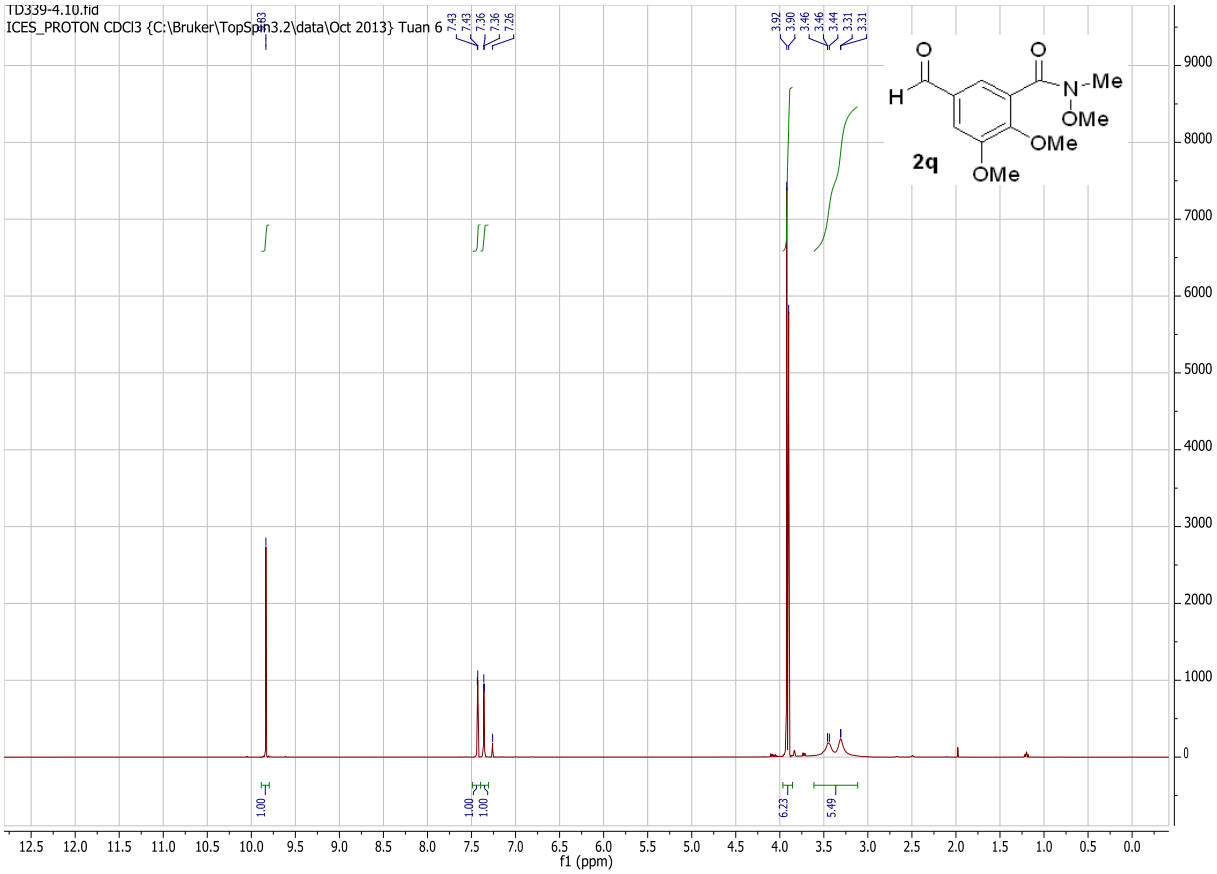
f1 (ppm)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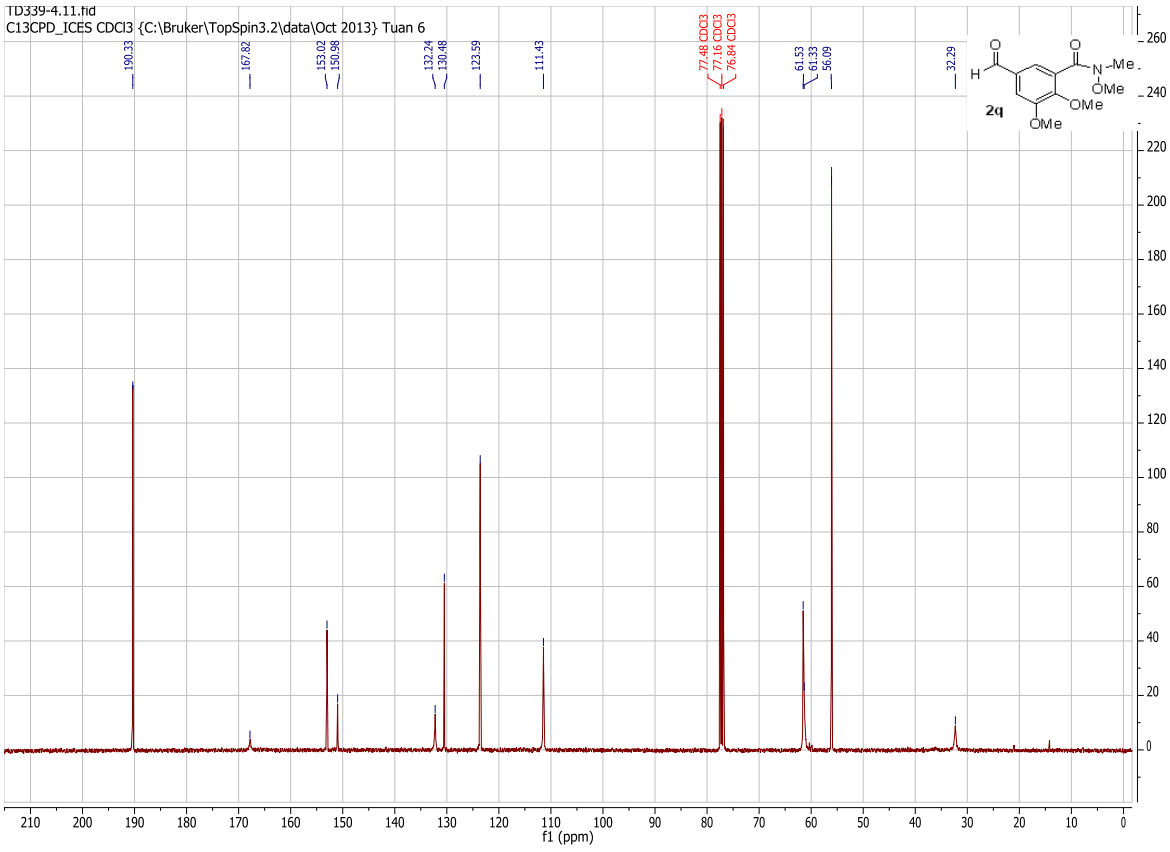
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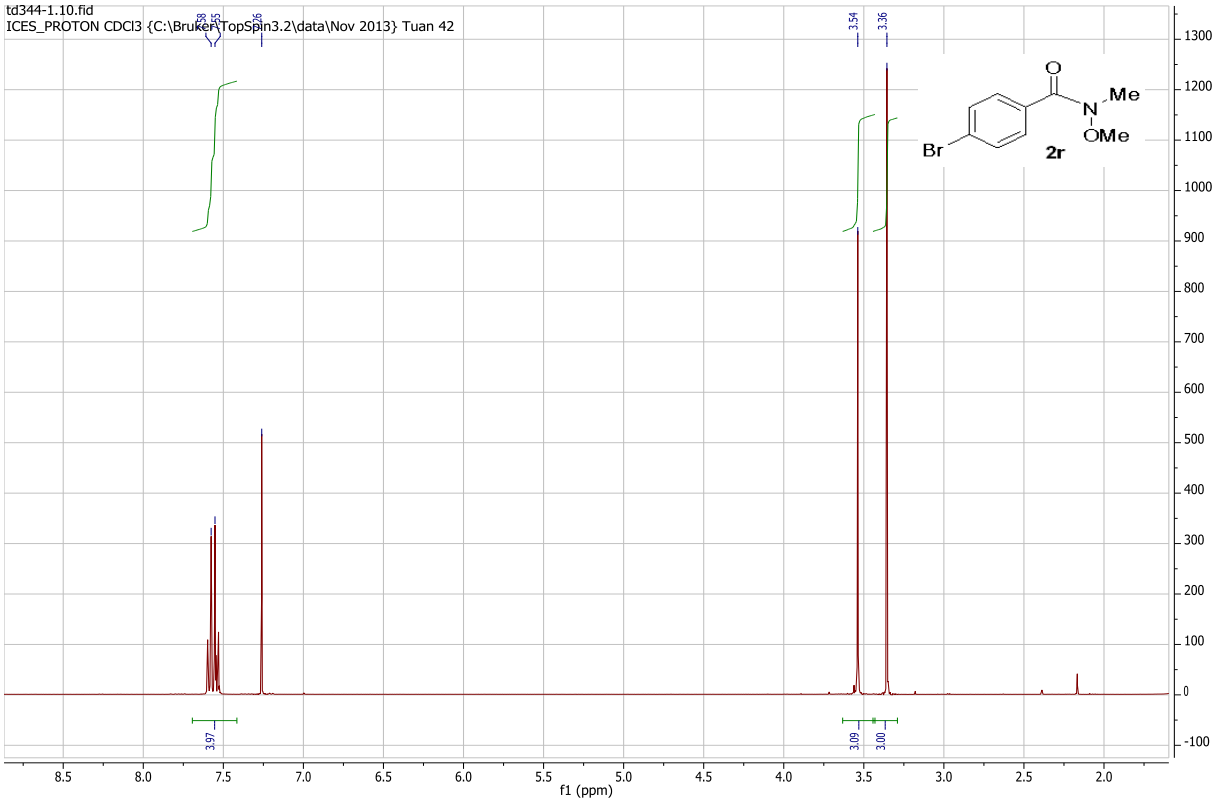
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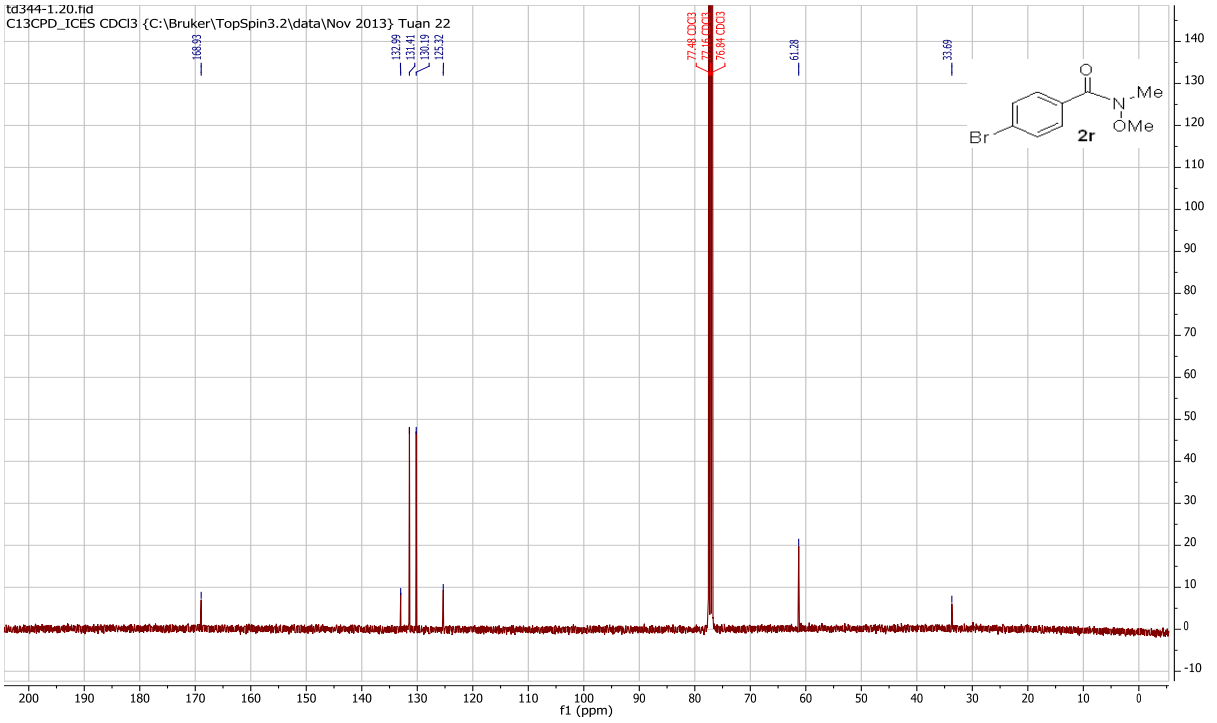
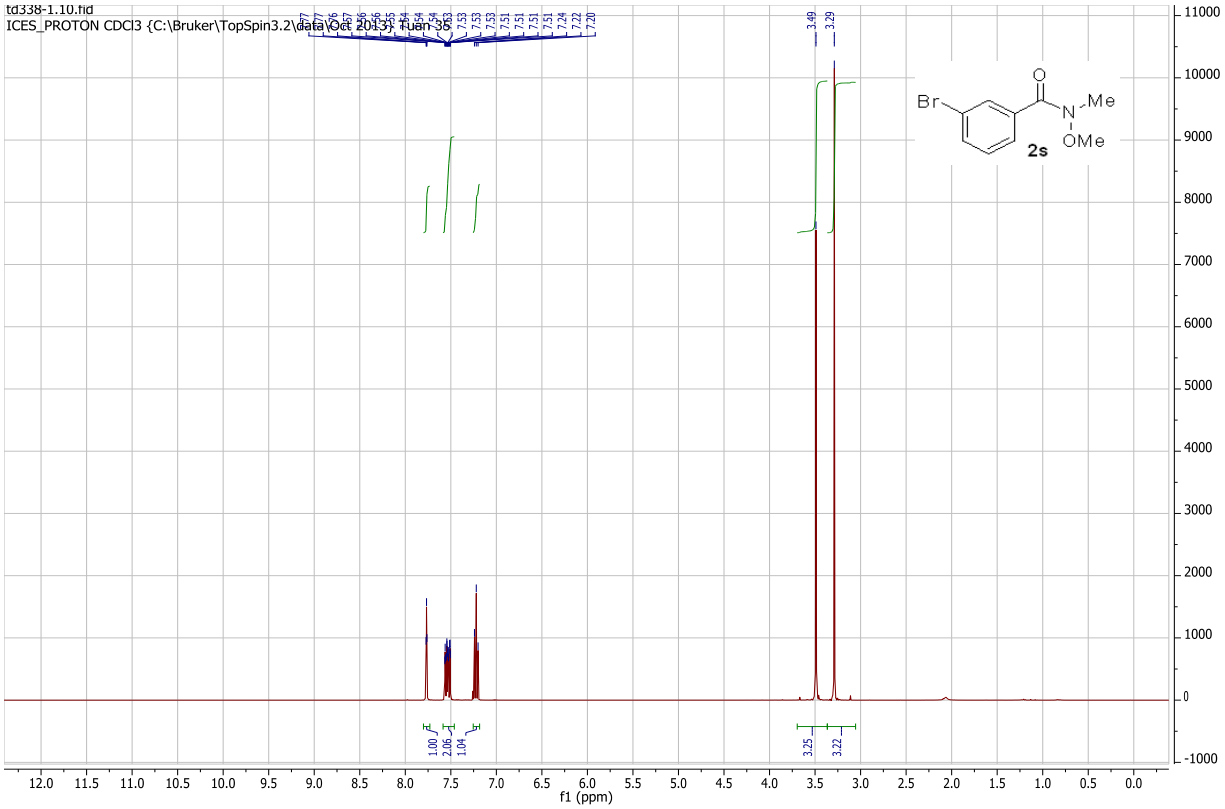
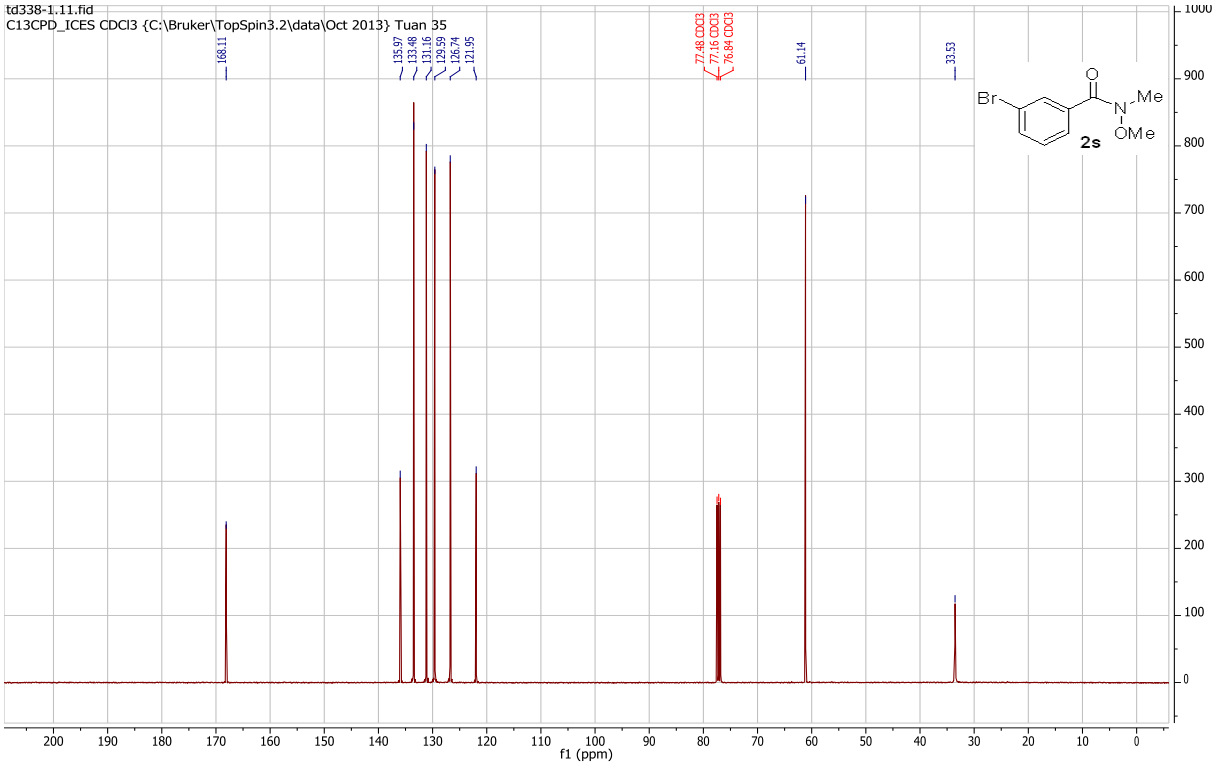
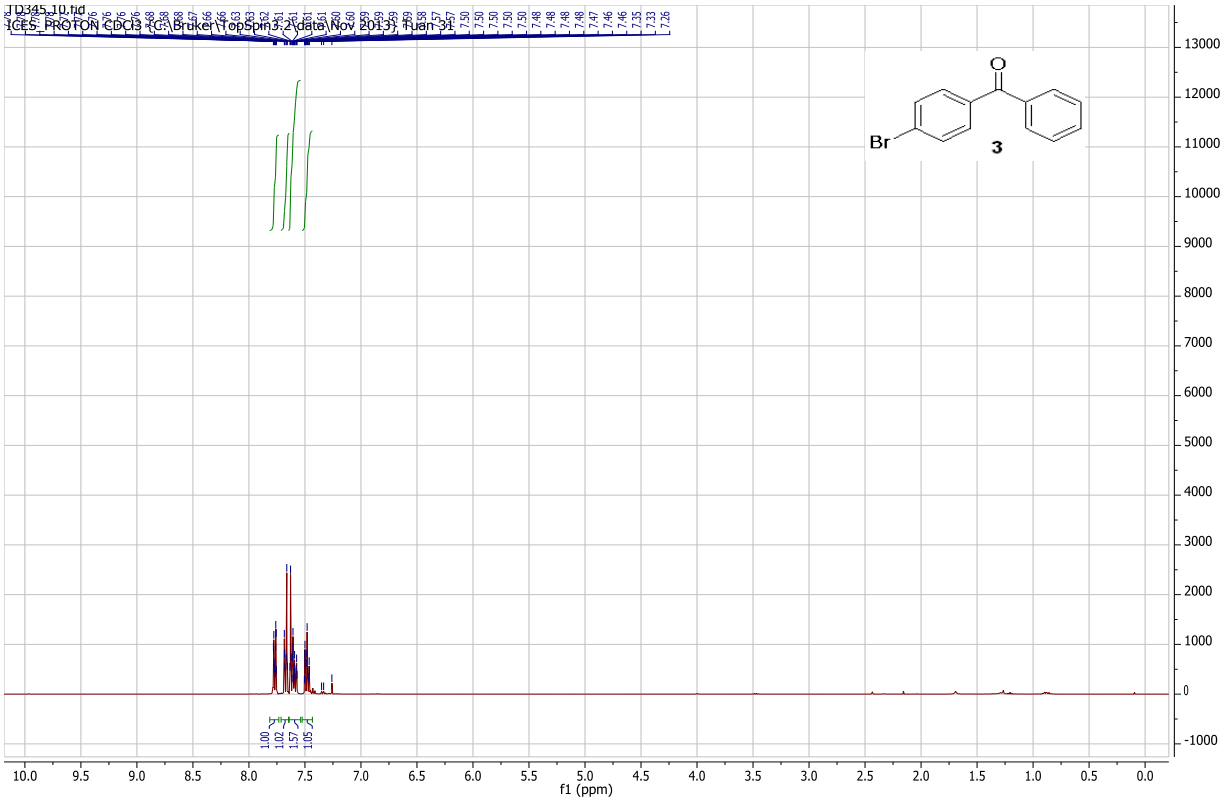
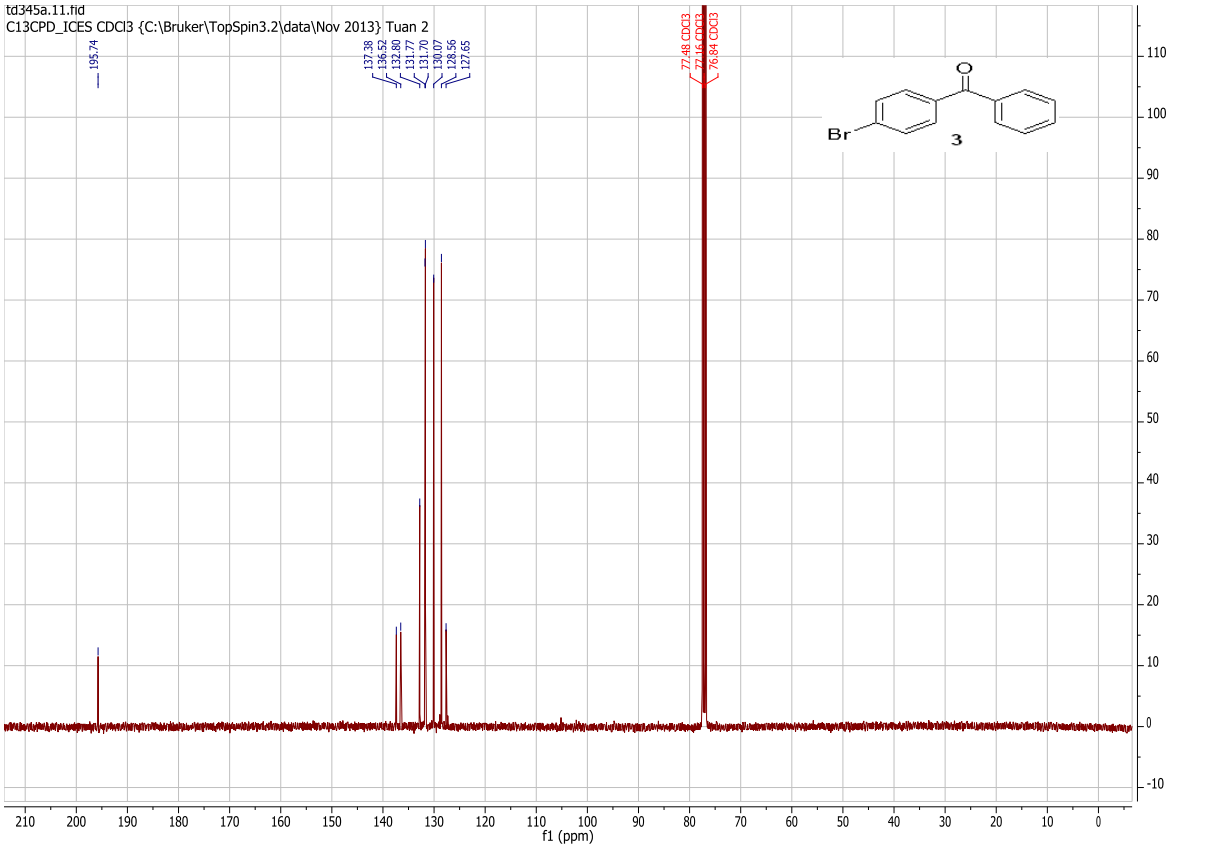
Me
OMe
Br
2r
f1 (ppm)

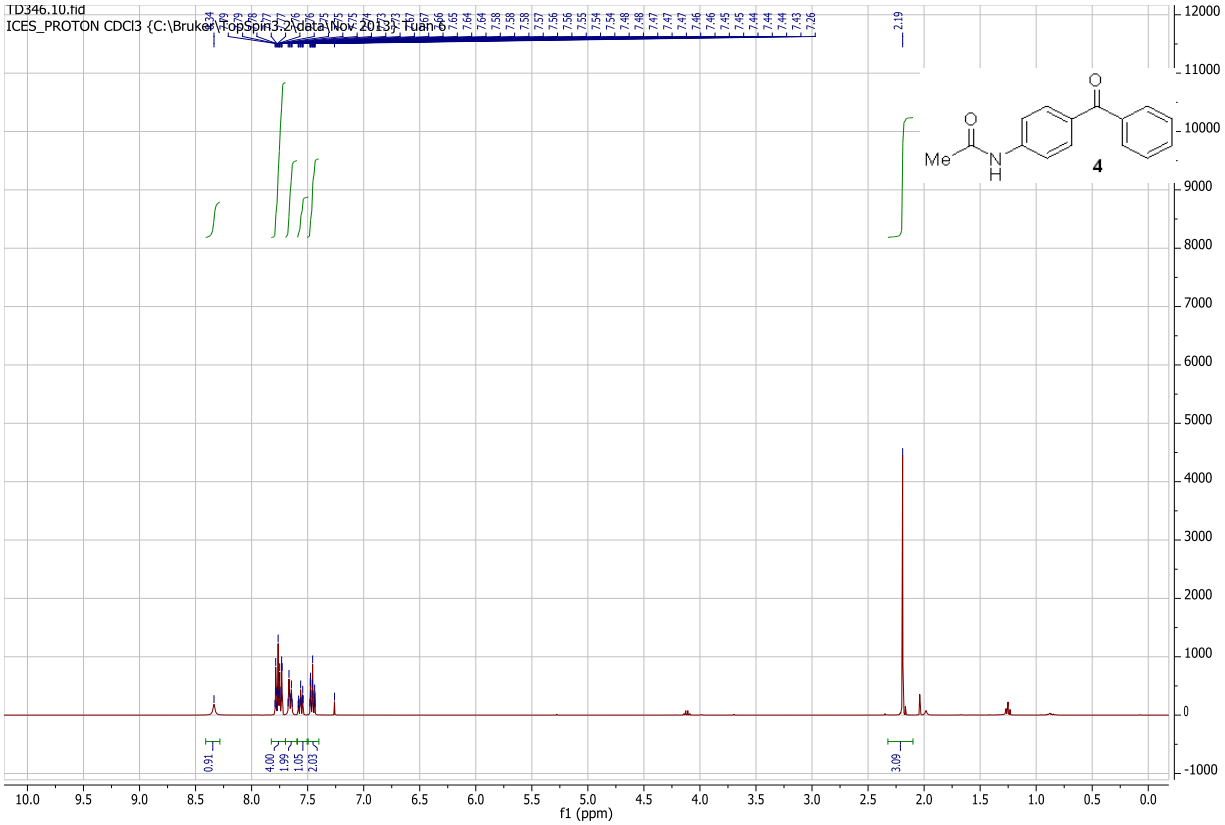

td344-1.20.fid
C13CPD_ICES CDCl3 {C:\Bruker\TopSpin3.2\data\Nov 2013} Tuan 22
168.93
132.99
131.41
130.19
125.32
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
61.28
33.69
O
N
Me
OMe
Br
2r
f1 (ppm)

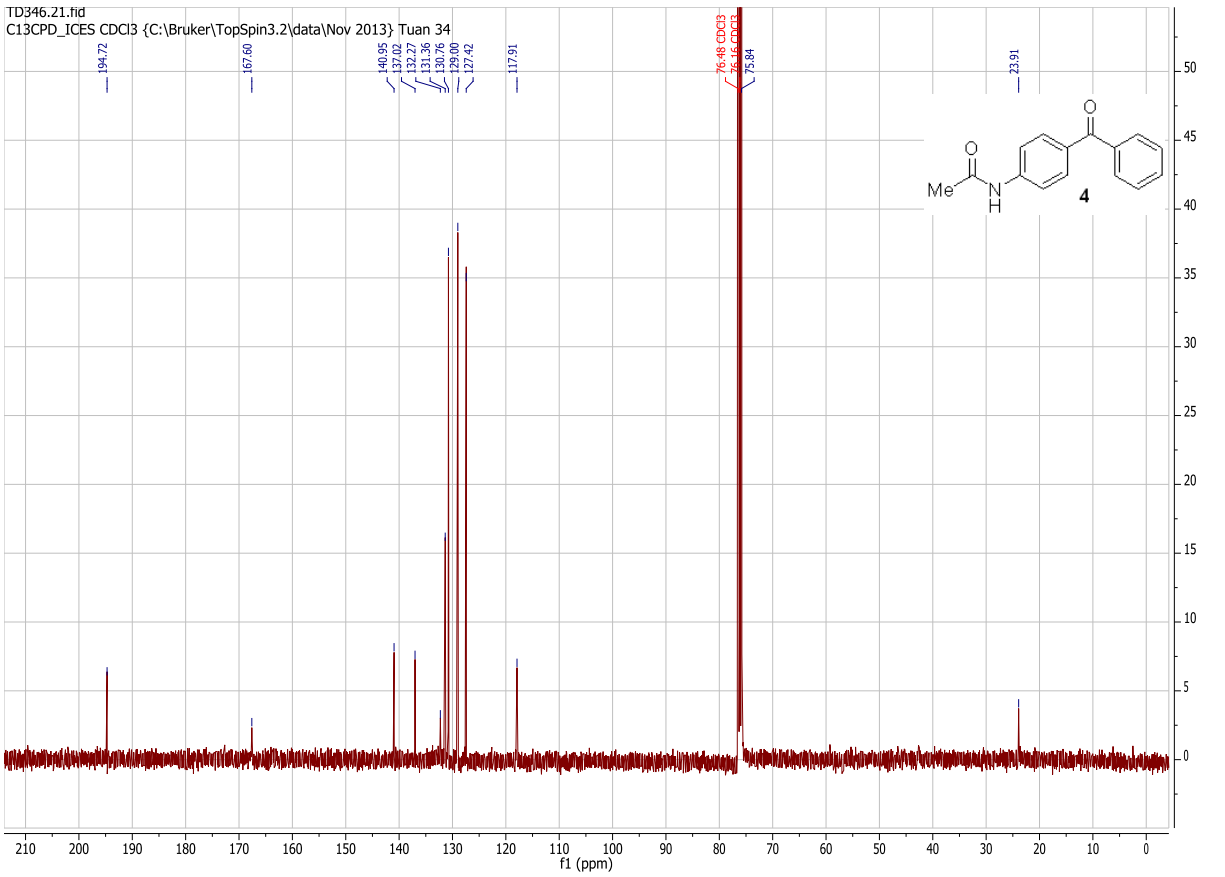

td338-1.10.fid
ICES_PROTON CDCl3 {C:\Bruker\TopSpin3.2\data\Oct 2013} Tuan 35
Br
O
N
Me
OMe
2s
f1 (ppm)

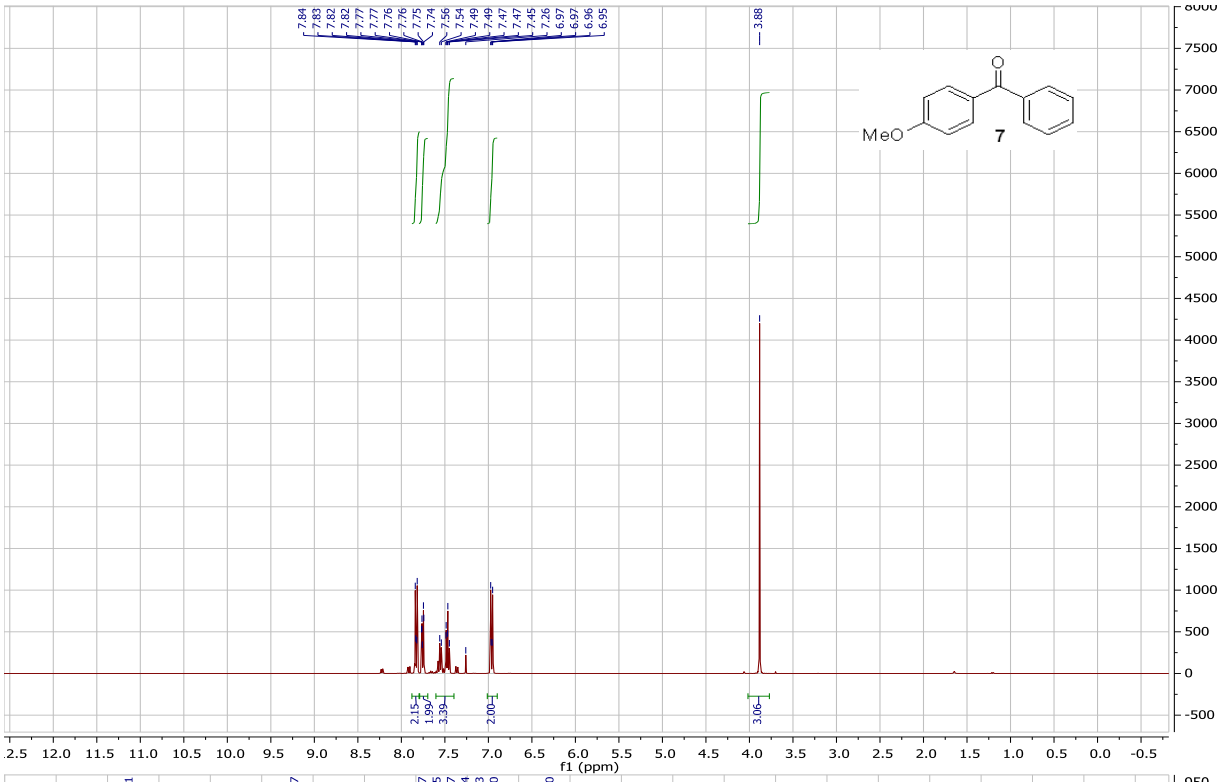

td338-1.11.fid
C13CPD_ICES CDCl3 {C:\Bruker\TopSpin3.2\data\Oct 2013} Tuan 35
168.11
135.97
133.48
131.16
129.59
126.74
121.95
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
61.14
33.53
Br
O
N
Me
OMe
2s
f1 (ppm)

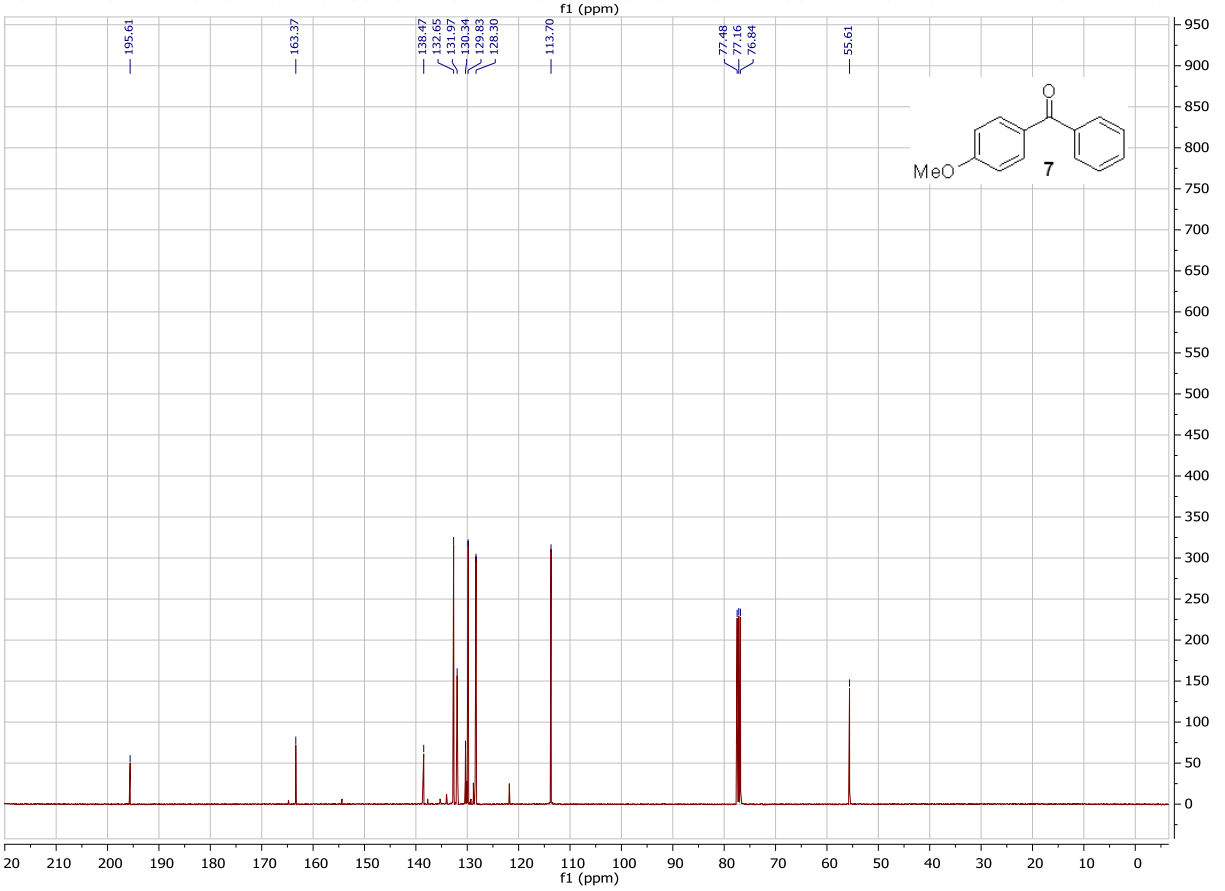

TD345.10.fid
1CES_PROTON CDCl3 {C:\Bruker\TopSpin3.2\data\Nov 2013} Tuan 31
Br
O
3
f1 (ppm)

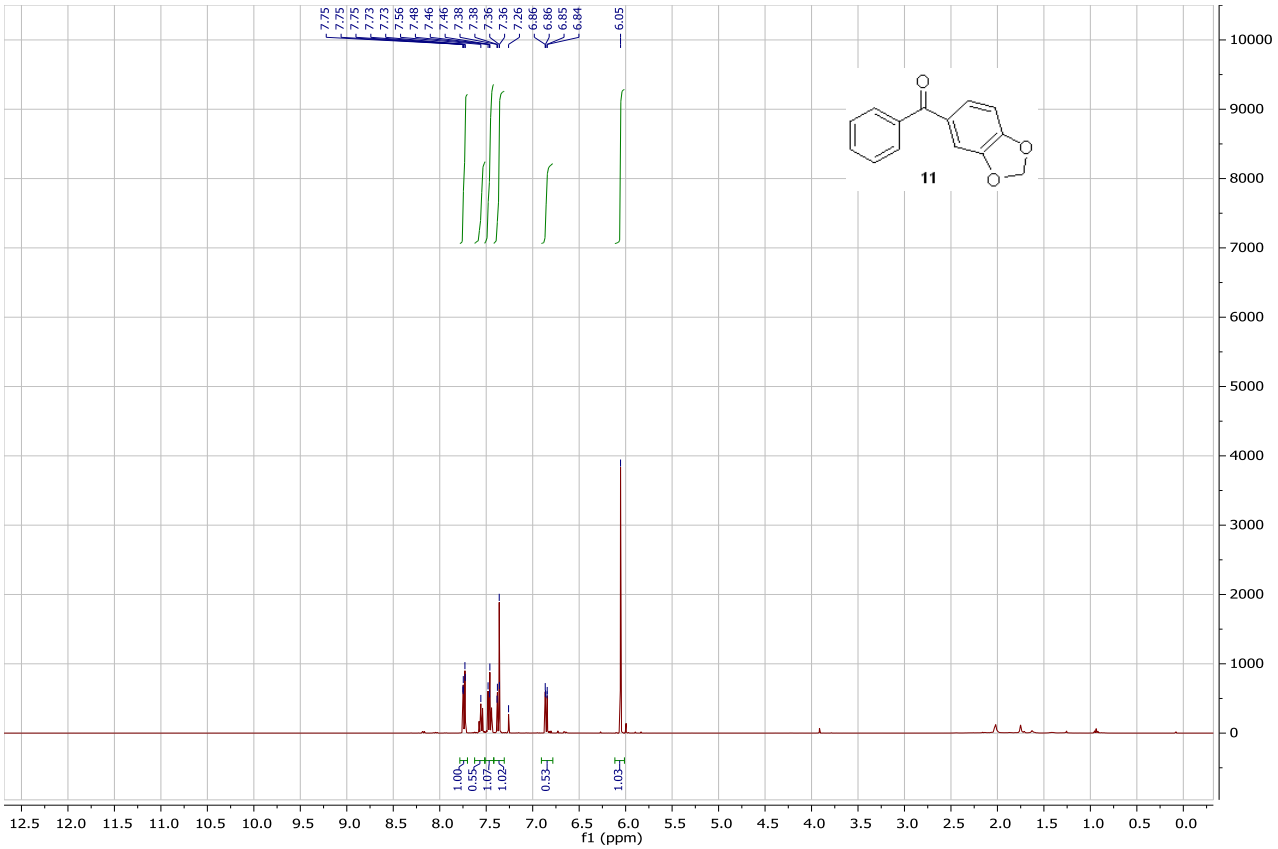

td345a.11.fid
C13CPD_ICES CDCl3 {C:\Bruker\TopSpin3.2\data\Nov 2013} Tuan 2
195.74
137.38
136.52
132.80
131.77
131.70
130.07
128.56
127.65
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
Br
O
3
f1 (ppm)

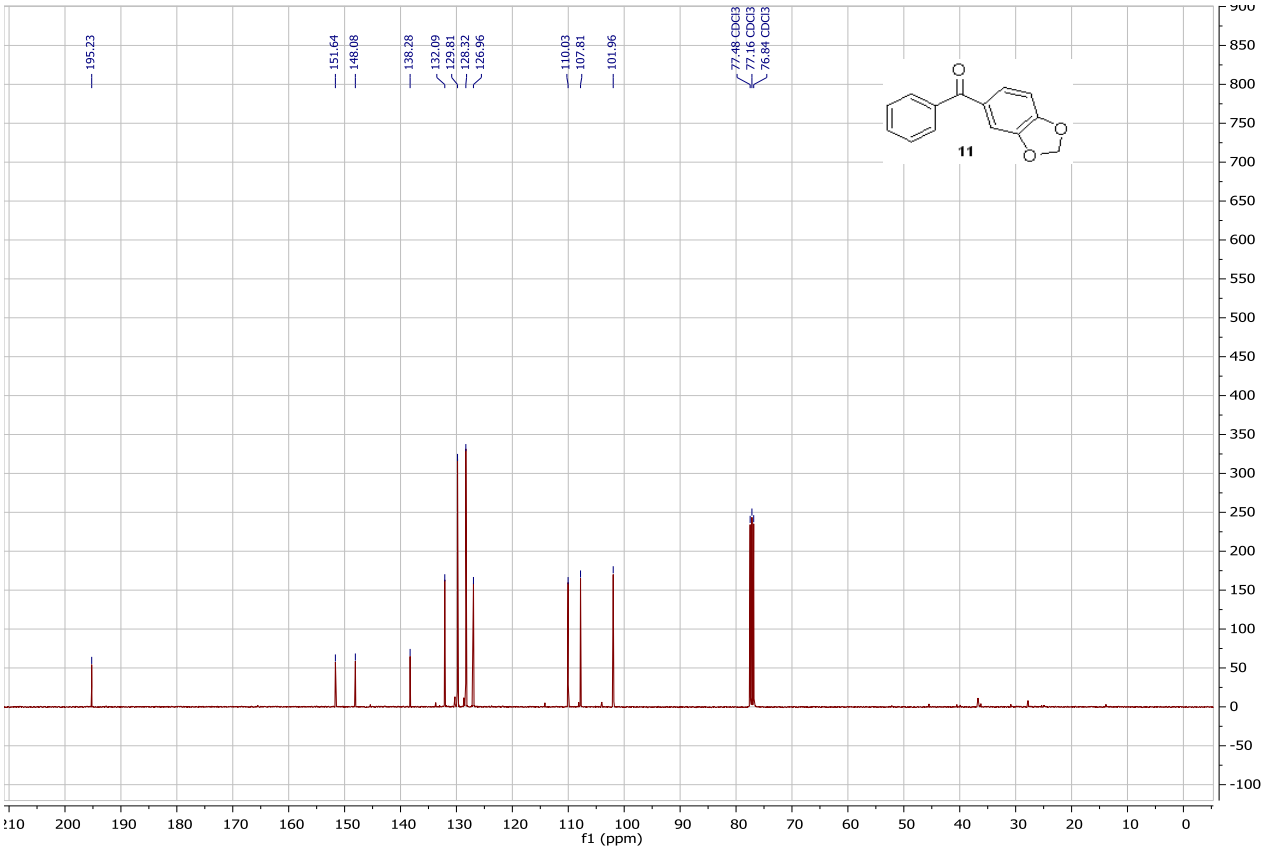

TD346.10.fid
ICES_PROTON CDCl3 {C:\Bruker\TopSpin3.2\data\Nov 2013} Tuan 6
2.19
Me
4
0.91
4.00
1.99
1.05
2.03
3.09
f1 (ppm)


TD346.21.fid
C13CPD_ICES CDCl3 {C:\Bruker\TopSpin3.2\data\Nov 2013} Tuan 34
194.72
167.60
140.95
137.02
132.27
131.36
130.76
129.00
127.42
117.91
76.48 CDCl3
76.16 CDCl3
75.84
23.91
Me
4
f1 (ppm)


7.84
7.83
7.82
7.82
7.77
7.77
7.76
7.76
7.75
7.74
7.56
7.54
7.49
7.49
7.47
7.47
7.45
7.26
6.97
6.97
6.96
6.95
3.88
MeO
7
2.15
1.99
3.39
2.00
3.06
f1 (ppm)


195.61
163.37
138.47
132.65
131.97
130.34
129.83
128.30
113.70
77.48
77.16
76.84
55.61
MeO
7
f1 (ppm)


7.75
7.75
7.75
7.73
7.73
7.56
7.48
7.46
7.46
7.38
7.38
7.36
7.36
7.26
6.86
6.86
6.85
6.84
6.05
11
1.00
0.55
1.07
1.02
0.53
1.03
f1 (ppm)


195.23
151.64
148.08
138.28
132.09
129.81
128.32
126.96
110.03
107.81
101.96
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
11
f1 (ppm)

B: Recycles and Pd-leaching studies

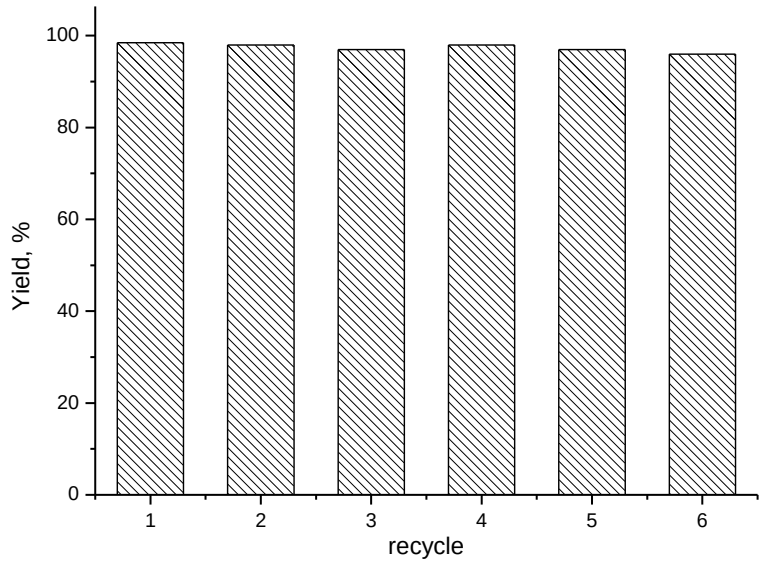
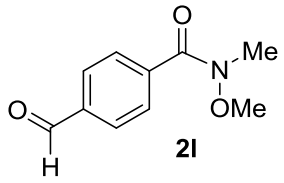


Figure 1. Recycle experiments for the synthesis of Weinreb amides from *p*-iodotoluene. Conditions: 0.5 mmol *p*-iodotoluene, 1.5 mmol *N,O*-dimethylhydroxyamine hydrochloride, 24 mg 1wt%Pd/ZIF-8, 2.5 equiv K_2CO_3, 2 mL toluene, 105 ^oC, 3 bar CO, 12h.

Pd leaching measurements by ICP AES:

Entry	Pd leaching (ppm)
Run 1	1.72
Run 2	1.38
Run 3	1.54
Run 4	2.15
Run 5	1.29
Run 6	1.62

C: HRMS (ESI TOF)



2l

Qualitative Compound Report

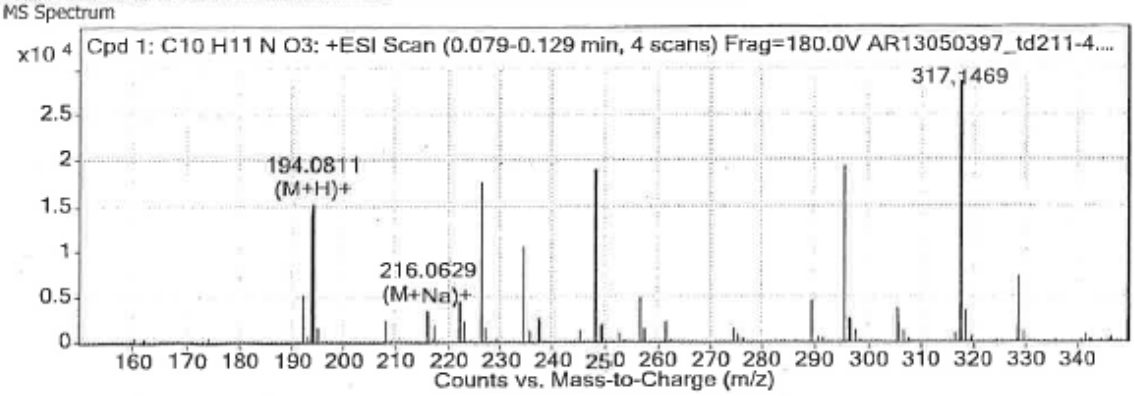
Data File	AR13050397_td211-4.d	**Sample Name**	AR13050397_td211-4
Sample Type	Sample	**Position**	Vial 22
Instrument Name	ESI TOF	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Test.m	**Comment**	

Compound Table

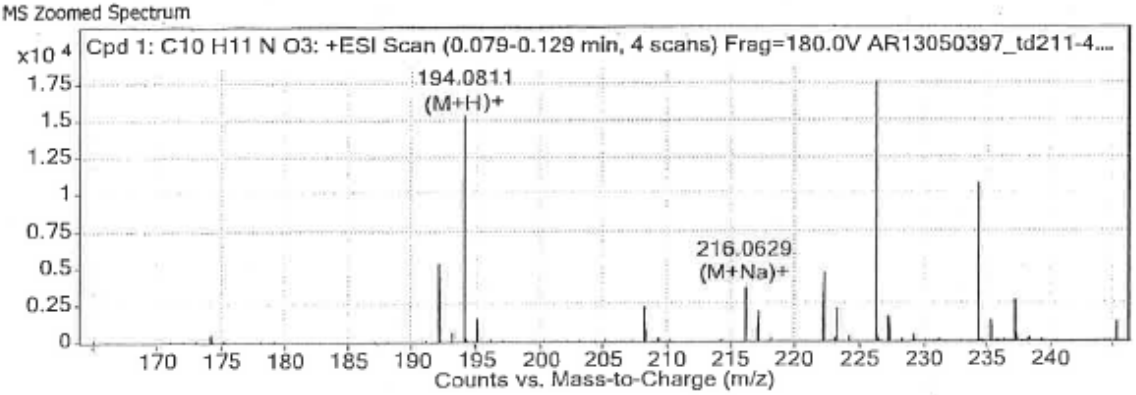
Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C10 H11 N O3	0.095	193.0738	15441	C10 H11 N O3	193.0739	-0.63

Compound Label	RT	Algorithm	Mass
Cpd 1: C10 H11 N O3	0.095	Find By Formula	193.0738

MS Spectrum



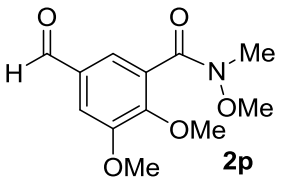
MS Zoomed Spectrum



MS Spectrum Peak List

m/z	Calc m/z	Diff(ppm)	z	Abund	Formula	Ion
194.0811	194.0812	-0.62	1	16278	C10 H12 N O3	(M+H)+
216.0629	216.0631	-1.13	1	3974	C10 H11 N Na O3	(M+Na)+

--- End Of Report ---



2p

Qualitative Compound Report

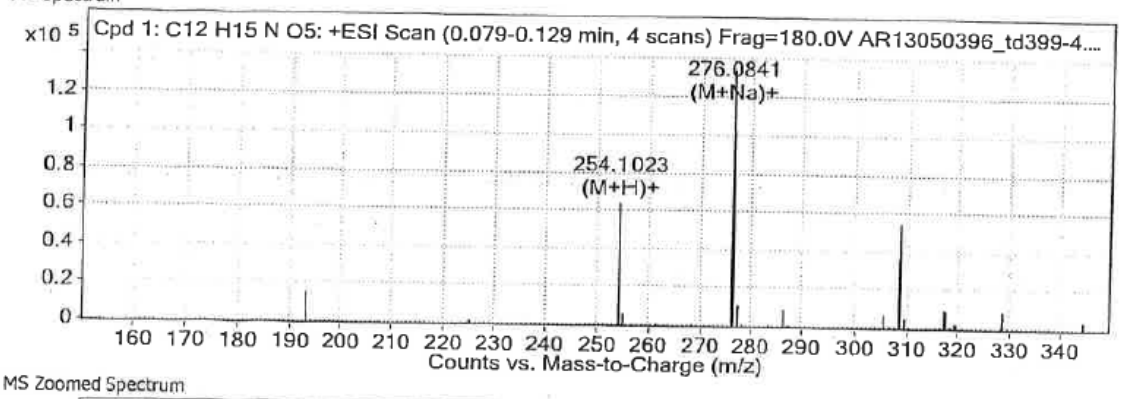
Data File	AR13050396_td399-4.d	**Sample Name**	AR13050396_td399-4
Sample Type	Sample	**Position**	Vial 21
Instrument Name	ESI TOF	**User Name**	
Acq Method	ESI.m	**IRM Calibration Status**	Success
DA Method	Test.m	**Comment**	

Compound Table

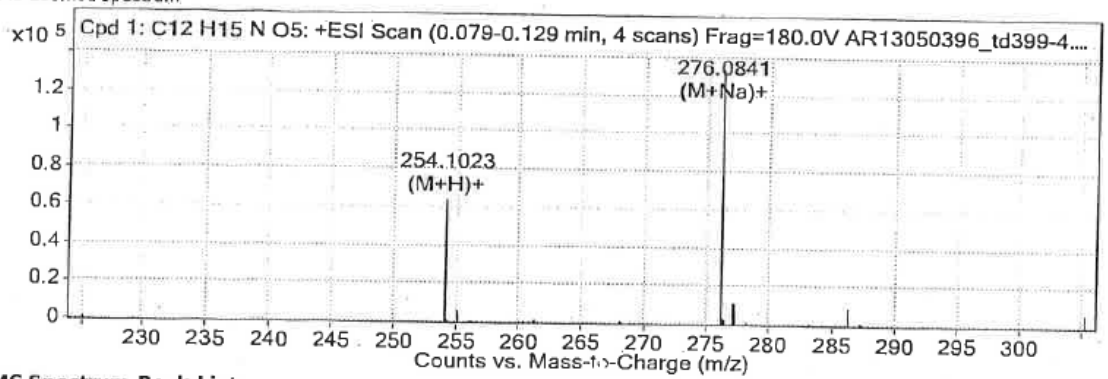
Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)
Cpd 1: C12 H15 N O5	0.096	253.0948	134098	C12 H15 N O5	253.095	-0.7

Compound Label	RT	Algorithm	Mass
Cpd 1: C12 H15 N O5	0.096	Find By Formula	253.0948

MS Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

m/z	Calc m/z	Diff(ppm)	z	Abund	Formula	Ion
254.1023	254.1023	-0.15	1	64611	C12 H16 N O5	(M+H)+
276.0841	276.0842	-0.7		134282	C12 H15 N Na O5	(M+Na)+

--- End Of Report ---