Supporting Information

Pd(II)-Catalyzed Chemoselective Acetoxylation of C(sp^2)–H and C(sp^3)–H Bonds in Tertiary Amides

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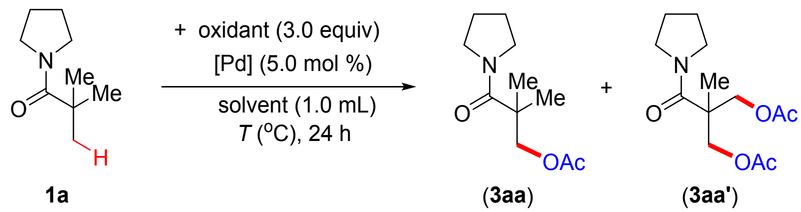
1. General Experimental (GC Method)

Gas Chromatography analyses were performed using a Shimadzu GC-2010 gas chromatograph equipped with a Shimadzu AOC-20s auto sampler and a Restek RTX-5 capillary column (30 m x 0.25 mm x 0.25 μm) The instrument was set to an injection volume of 1 μL, an inlet split ratio of 10:1, and inlet and detector temperatures of 250 and 320 ^{o}C, respectively. UHP-grade argon was used as carrier gas with a flow rate of 30 mL/min. The temperature program used for all the analyses is as follows: 80 °C, 1 min; 30 ^{o}C/min to 200 ^{o}C, 2 min; 30 ^{o}C/min to 260 ^{o}C, 3 min; 30 ^{o}C/min to 300 ^{o}C, 3 min. Response factors for all the necessary compounds with respect to standard *n*-dodecane were calculated from the average of three independent GC runs.

2. Detailed Optimization Study

Table S1. Optimization of Reaction Parameters.[a]

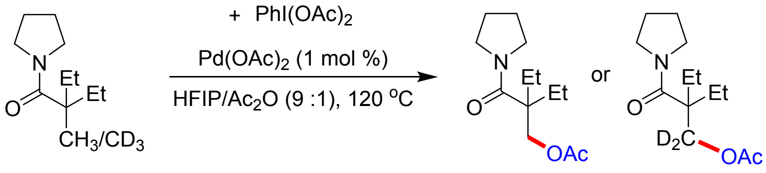


entry	[Pd]	oxidant	T(°C)	Solvent	Yield (%)[b] 3aa	3aa'
1	$Pd(OAc)_2$	$PhI(OAc)_2$	120	AcOH	47 (44)	12
2	$Pd(OAc)_2$	$PhI(OAc)_2$	120	$AcOH/Ac_2O$	70 (68)	8
3	$Pd(OAc)_2$	$K_2S_2O_8$	120	$AcOH/Ac_2O$	42	--
4	$Pd(OAc)_2$	$Na_2S_2O_8$	120	$AcOH/Ac_2O$	56	--
5	$Pd(OAc)_2$	AgOAc	120	$AcOH/Ac_2O$	35	--
6	$Pd(OAc)_2$	Oxone	120	$AcOH/Ac_2O$	16	2
7	$Pd(OAc)_2$	$Cu(OAc)_2$	120	$AcOH/Ac_2O$	15	--
8	$PdCl_2$	$PhI(OAc)_2$	120	$AcOH/Ac_2O$	53 (51)	5
9	$Pd_2(dba)_3$	$PhI(OAc)_2$	120	$AcOH/Ac_2O$	23	8
10	$Pd(cod)Cl_2$	$PhI(OAc)_2$	120	$AcOH/Ac_2O$	42	2
11	$Pd(PPh_3)_4$	$PhI(OAc)_2$	120	$AcOH/Ac_2O$	43	3
12	$Pd(OAc)_2$	$PhI(OAc)_2$	120	Toluene	25	--
13	$Pd(OAc)_2$	$PhI(OAc)_2$	120	MeOH	--	--
14	$Pd(OAc)_2$	$PhI(OAc)_2$	120	DCE	4	5
15	$Pd(OAc)_2$	$PhI(OAc)_2$	120	TFA/TFAA	--	--
16	$Pd(OAc)_2$	$PhI(OAc)_2$	120	HFIP	65 (62)	10
17	$Pd(OAc)_2$	$PhI(OAc)_2$	120	$HFIP/Ac_2O$	72 (69)	8

18	$Pd(OAc)_2$	$PhI(OAc)_2$	80	HFIP/Ac_2O	98 (94)	--
19[c]	$Pd(OAc)_2$	$PhI(OAc)_2$	80	HFIP/Ac_2O	97 (93)	--
20[d]	$Pd(OAc)_2$	$PhI(OAc)_2$	80	HFIP/Ac_2O	57	--
21[c,e]	$Pd(OAc)_2$	$PhI(OAc)_2$	80	HFIP/Ac_2O	86 (83)	--
22[c,f]	$Pd(OAc)_2$	$PhI(OAc)_2$	80	HFIP/Ac_2O	95 (94)	--
23[c,f,g]	**$Pd(OAc)_2$**	**$PhI(OAc)_2$**	**80**	**HFIP/Ac_2O**	**96 (93)**	**--**
24	$Pd(OAc)_2$	--	80	HFIP/Ac_2O	--	--
25	--	$PhI(OAc)_2$	80	HFIP/Ac_2O	--	--

[a] Reaction Conditions: 2,2-Dimethyl-1-(pyrrolidin-1-yl)propan-1-one (**1a**; 0.047 g, 0.302 mmol), oxidant (0.90 mmol), [Pd] (0.015 mmol, 5.0 mol %), solvent (1.0 mL). [b] GC yield using *n*-dodecane as internal standard, isolated yield is in parenthesis. [c] 1.0 mol % of $Pd(OAc)_2$ used. [d] 0.5 mol % of $Pd(OAc)_2$ used. [e] 2 equiv of $PhI(OAc)_2$ used. [f] Reaction performed for 20 h. [g] Solvent (0.7 mL; HFIP:Ac_2O = 9:1, v/v). HFIP = Hexafluoro-2-propanol.

3. Kinetic Isotope Effect (KIE) plot



1p or [D]-**1p** **3pa** [D]-**3pa**

k_H = 9.134 x 10^{-4} $\mathrm{Mmin^{-1}}$
k_D = 1.636 x 10^{-4} $\mathrm{Mmin^{-1}}$

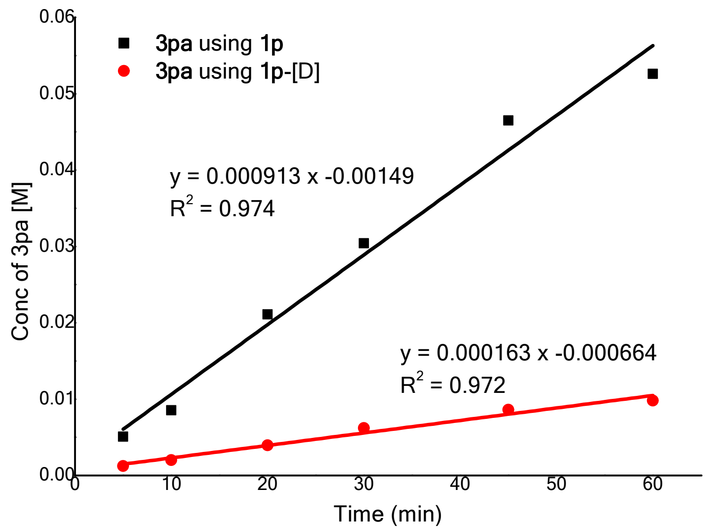
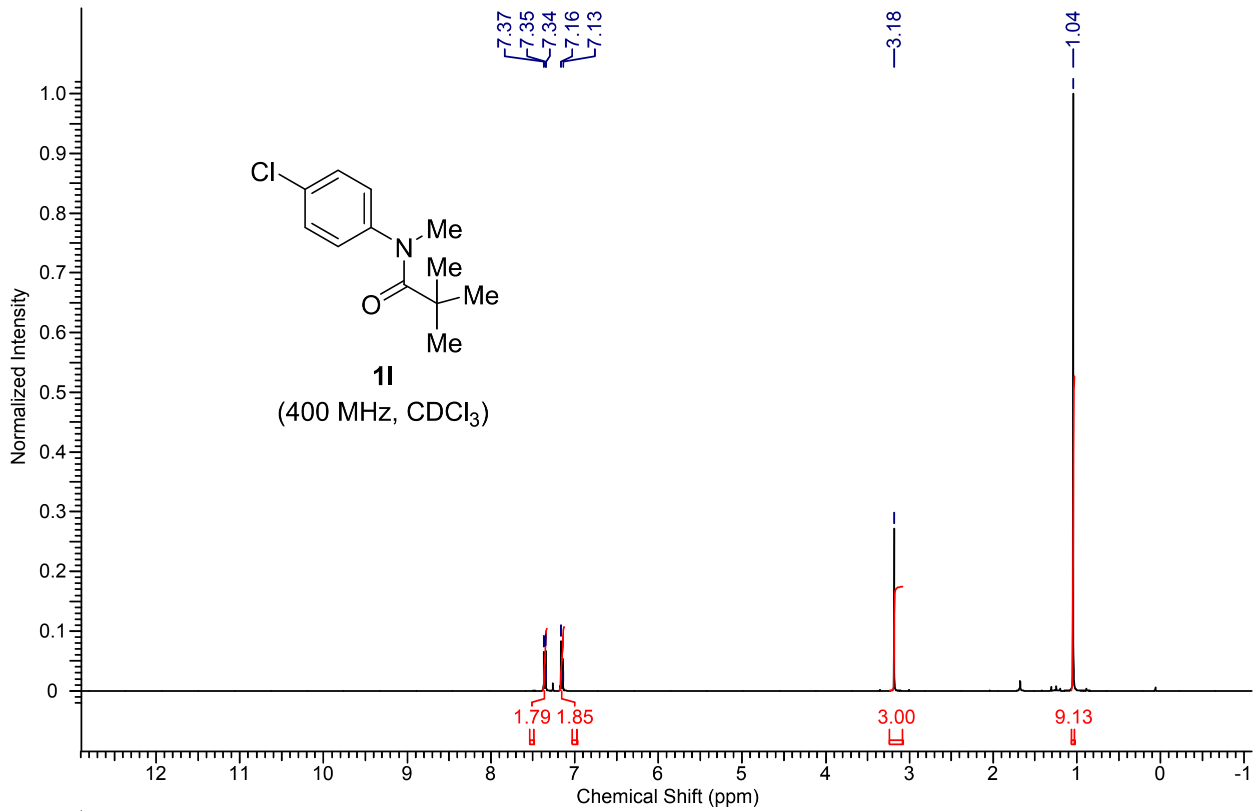
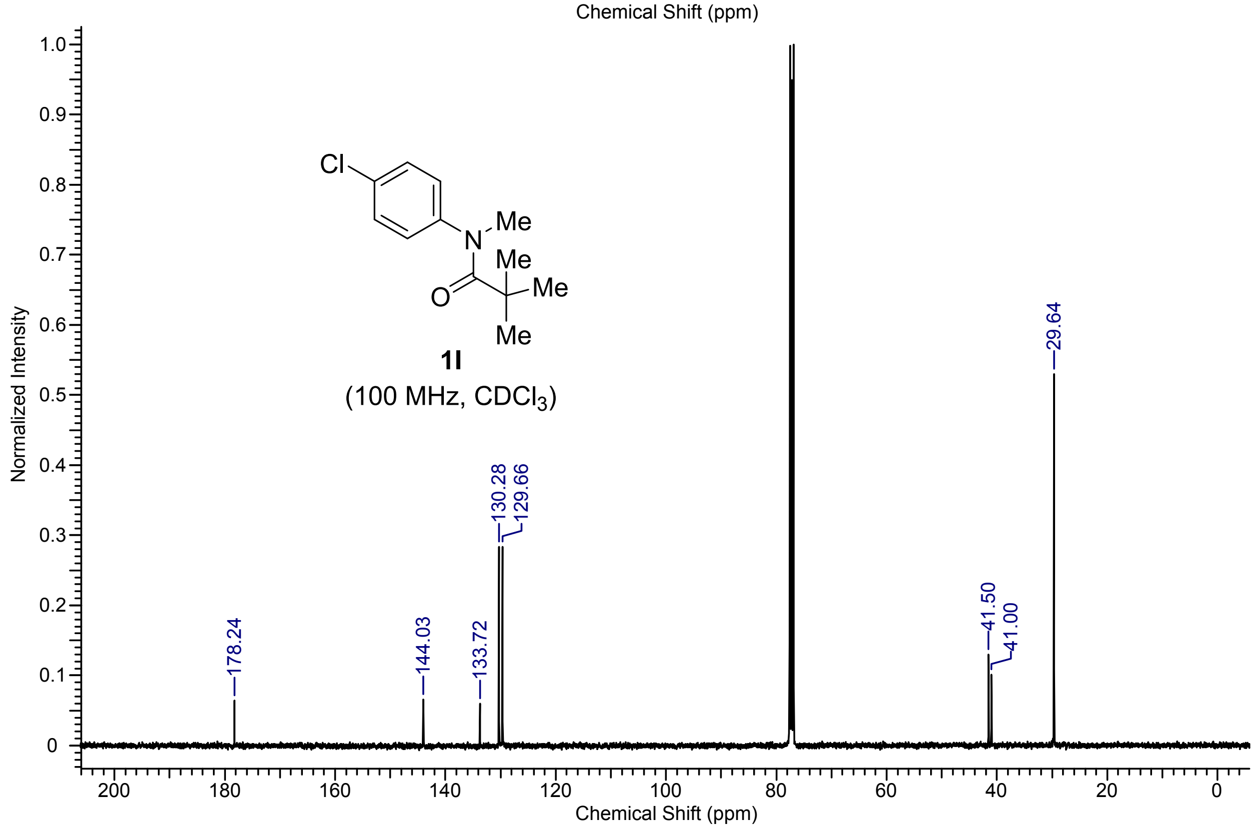
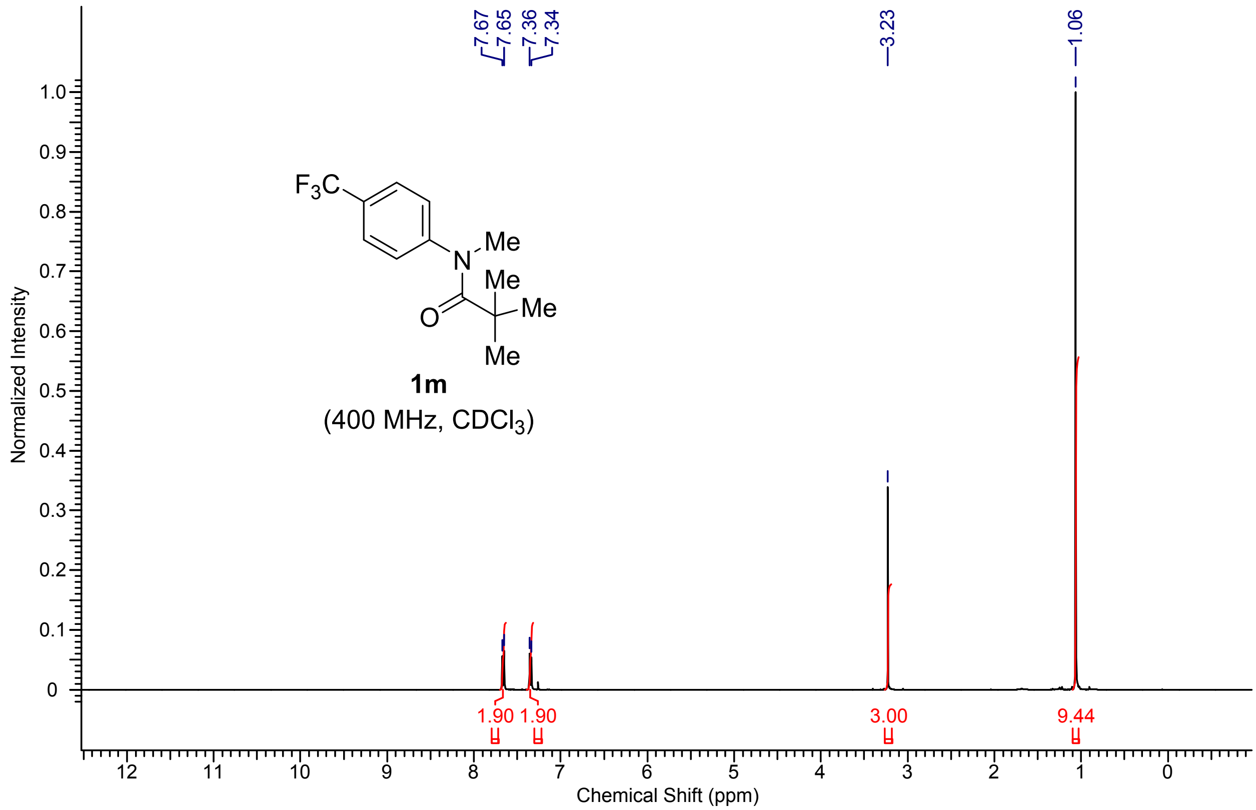


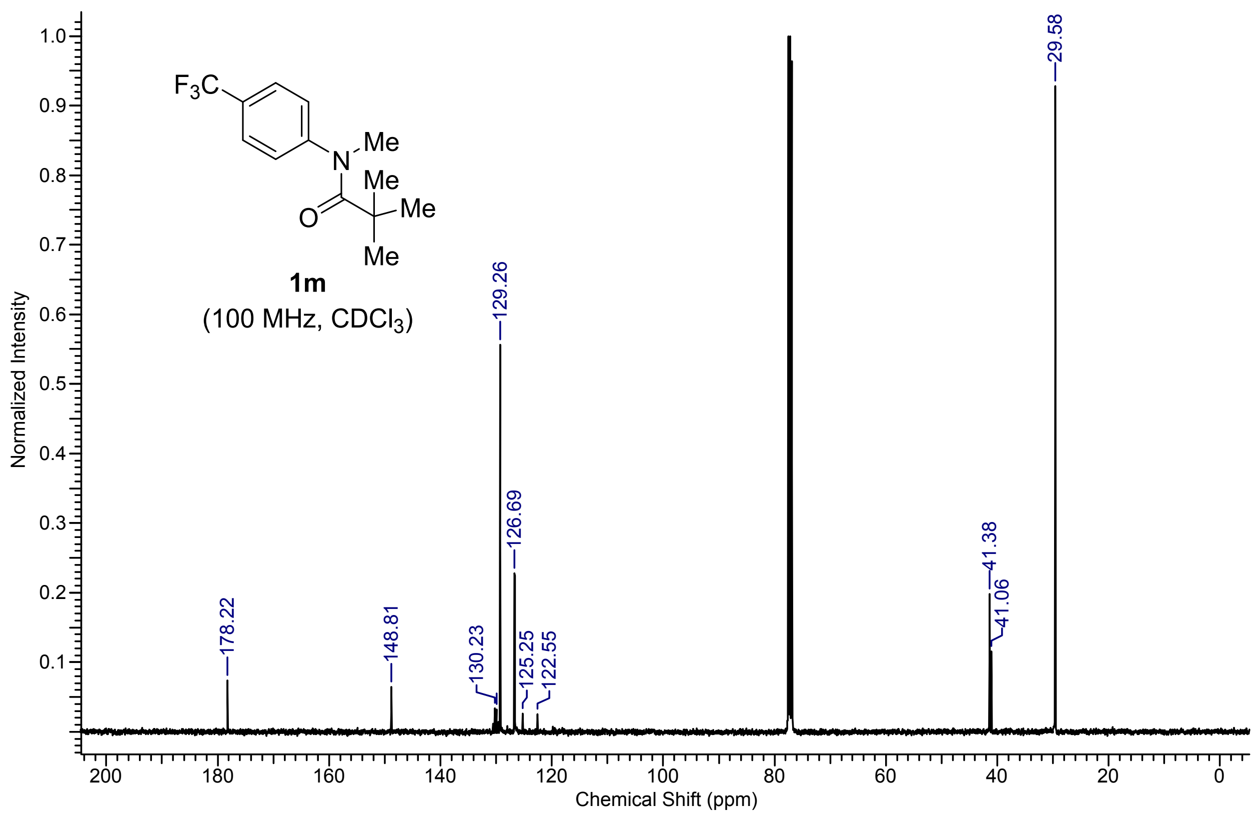
Figure S1. Time-dependent formation of **3pa** using substrates **1p** and [D]-**1p**.

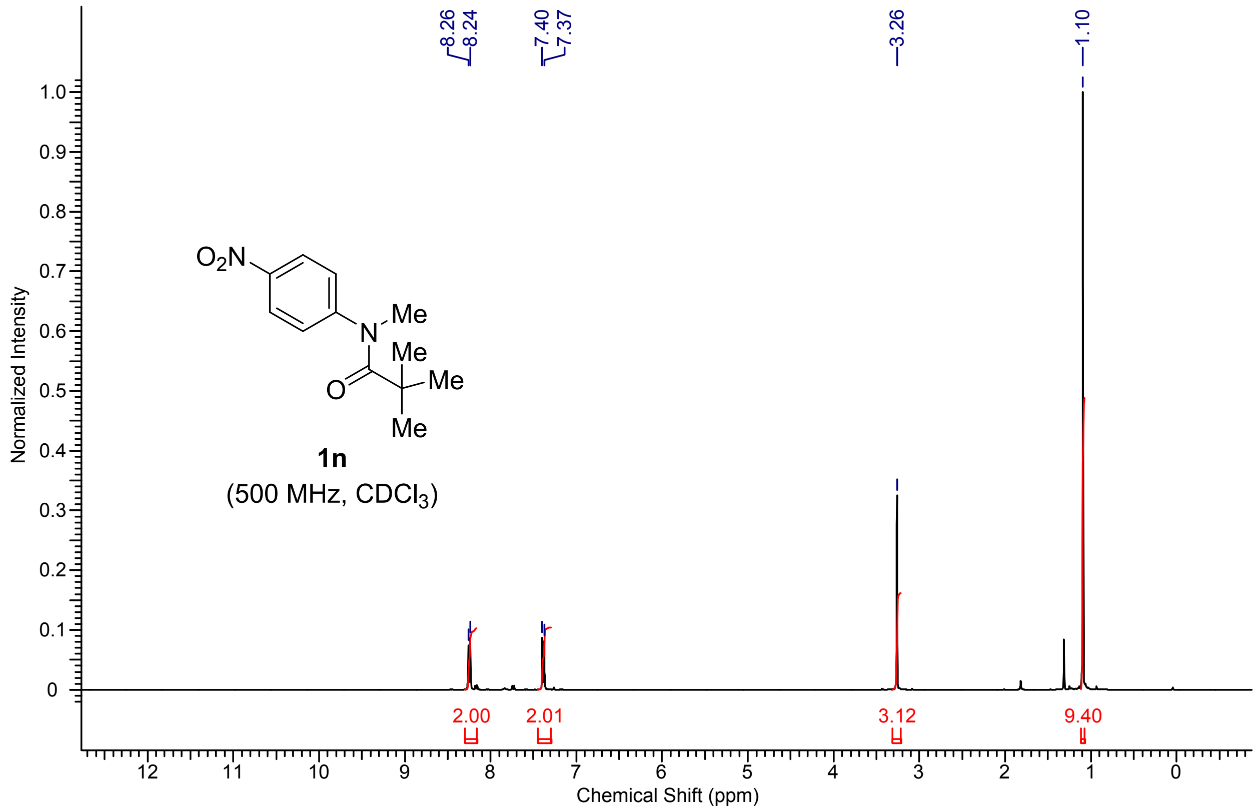
4. ^{1}H and ^{13}C NMR Spectra of Starting Compounds

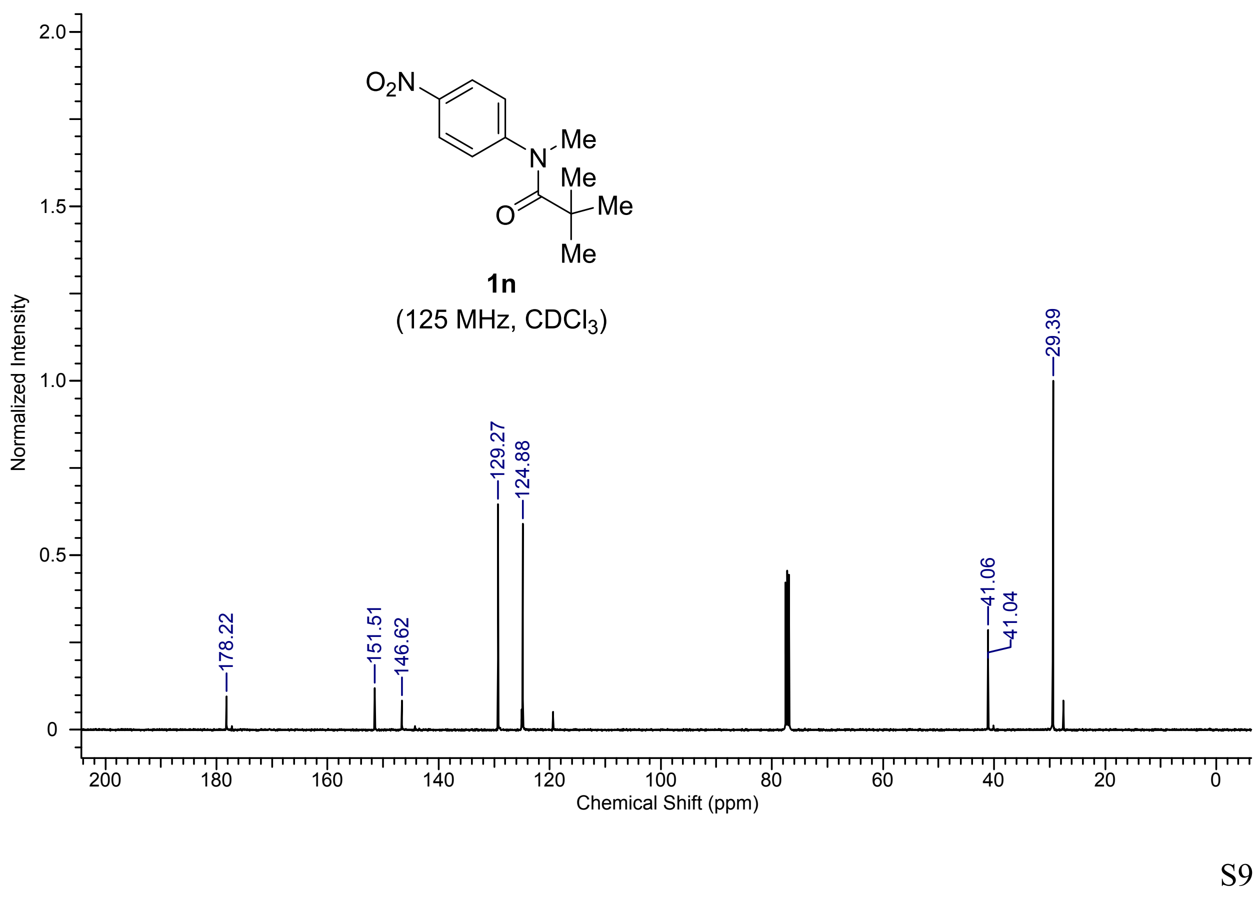


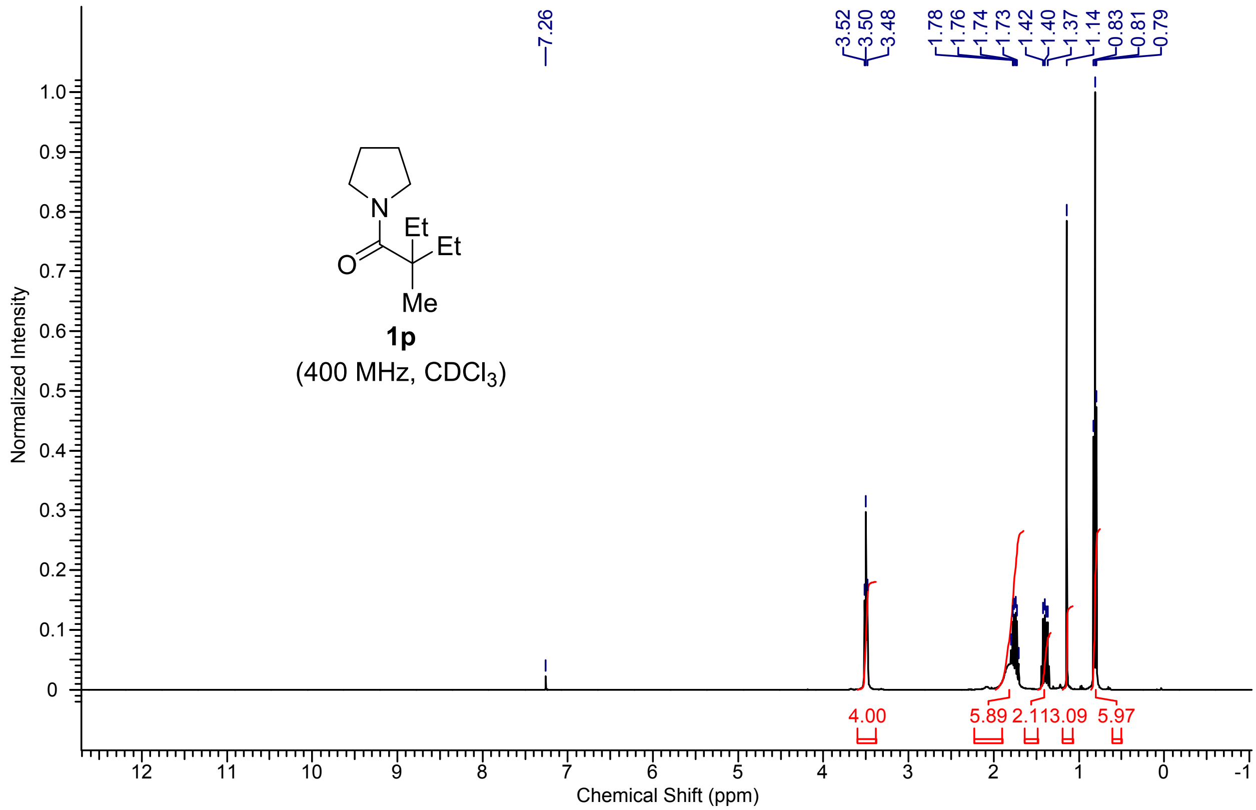


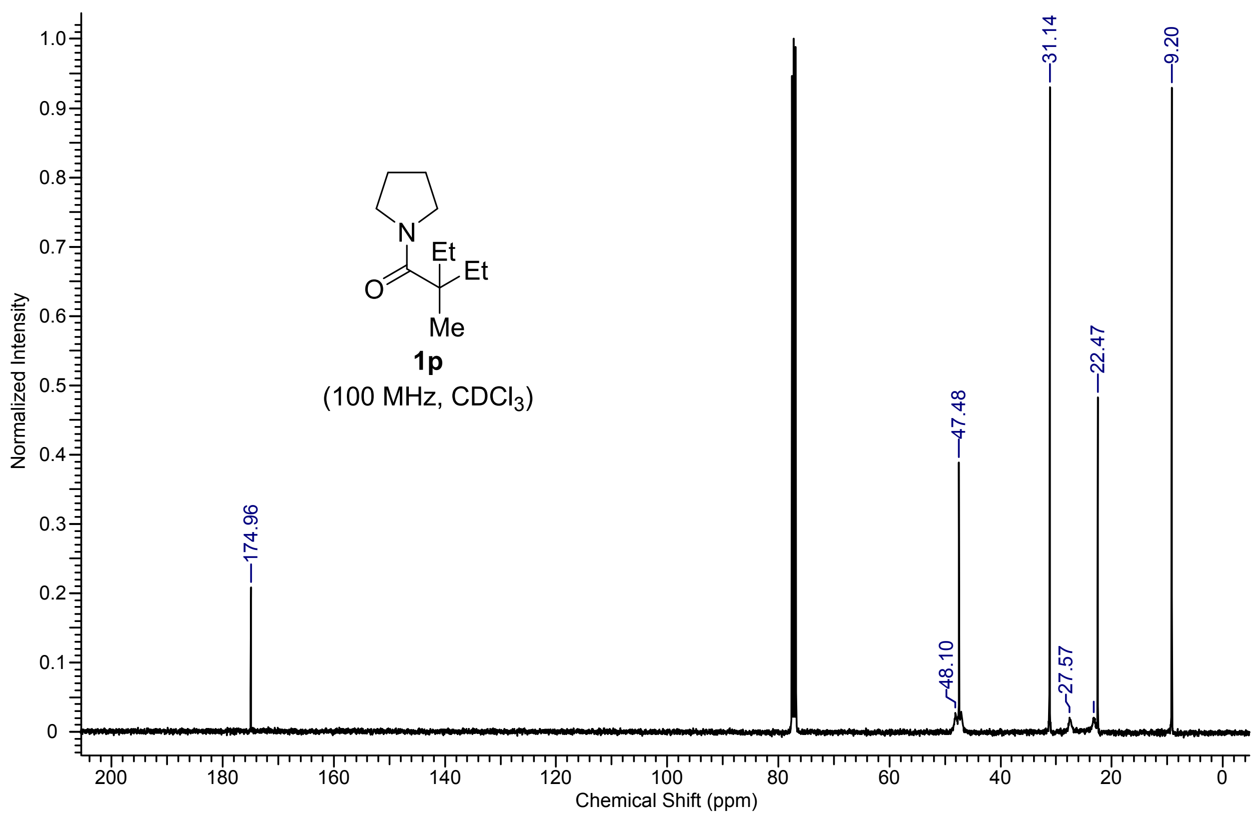

7.67
7.65
7.36
7.34
3.23
1.06
F3C
N
Me
Me
Me
Me
O
1m
(400 MHz, CDCl3)
Normalized Intensity
1.90
1.90
3.00
9.44
Chemical Shift (ppm)

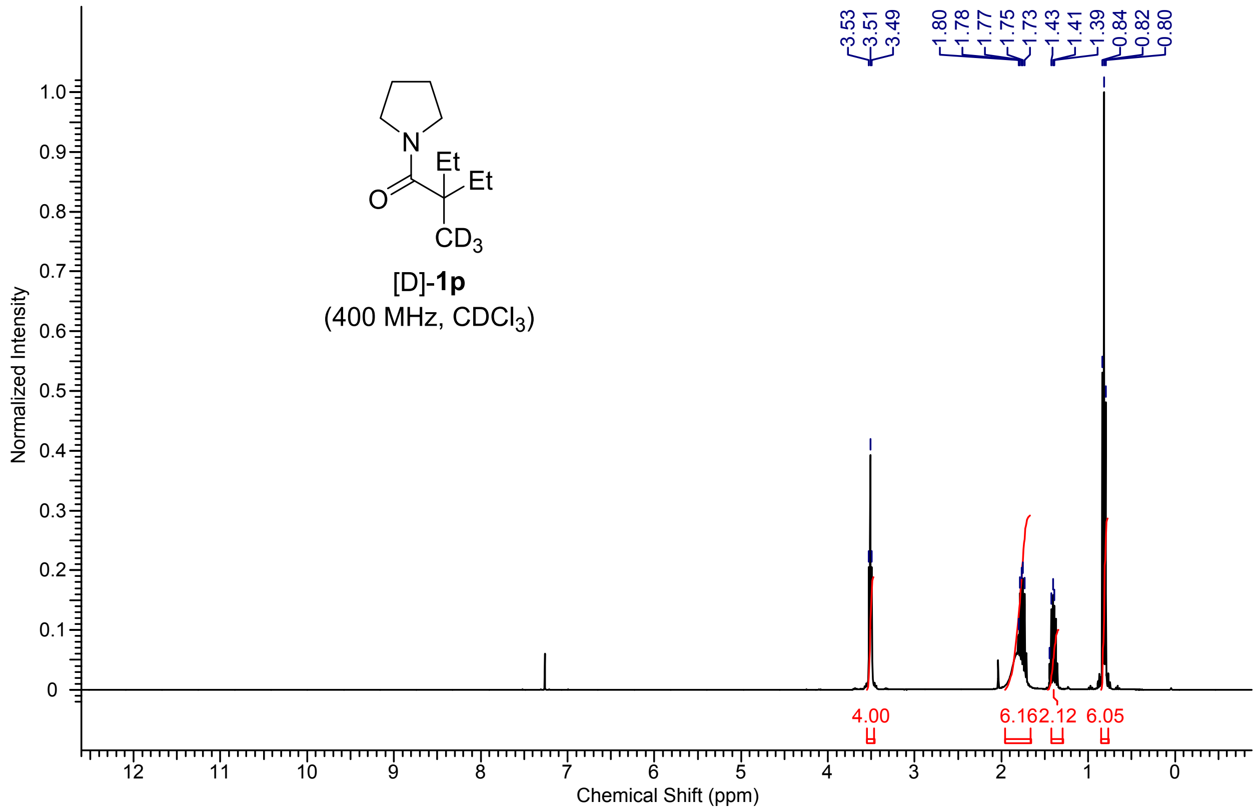

F3C
N
Me
Me
Me
Me
O
1m
(100 MHz, CDCl3)
Normalized Intensity
178.22
148.81
130.23
129.26
126.69
125.25
122.55
41.38
41.06
29.58
Chemical Shift (ppm)

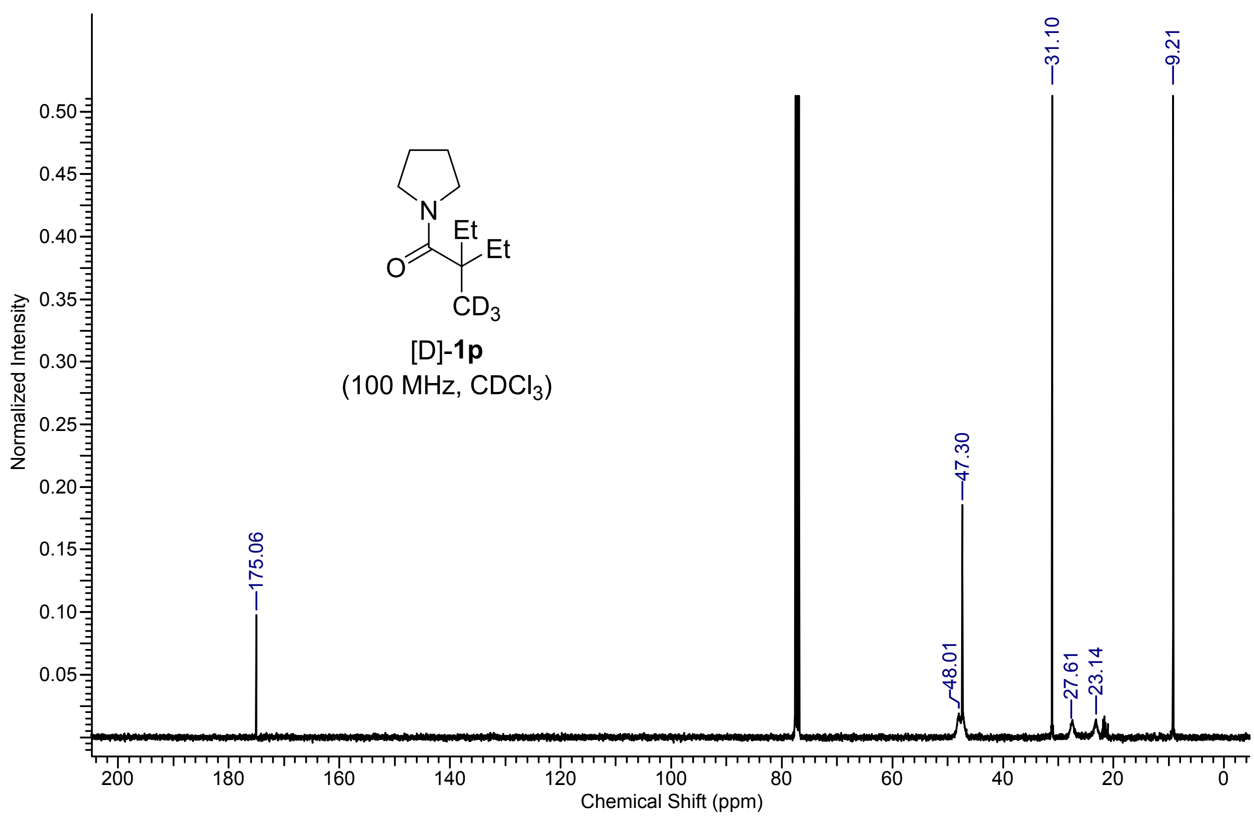

8.26
8.24
7.40
7.37
3.26
1.10
Normalized Intensity
O2N
N
Me
O
Me
Me
Me
1n
(500 MHz, CDCl3)
2.00
2.01
3.12
9.40
Chemical Shift (ppm)

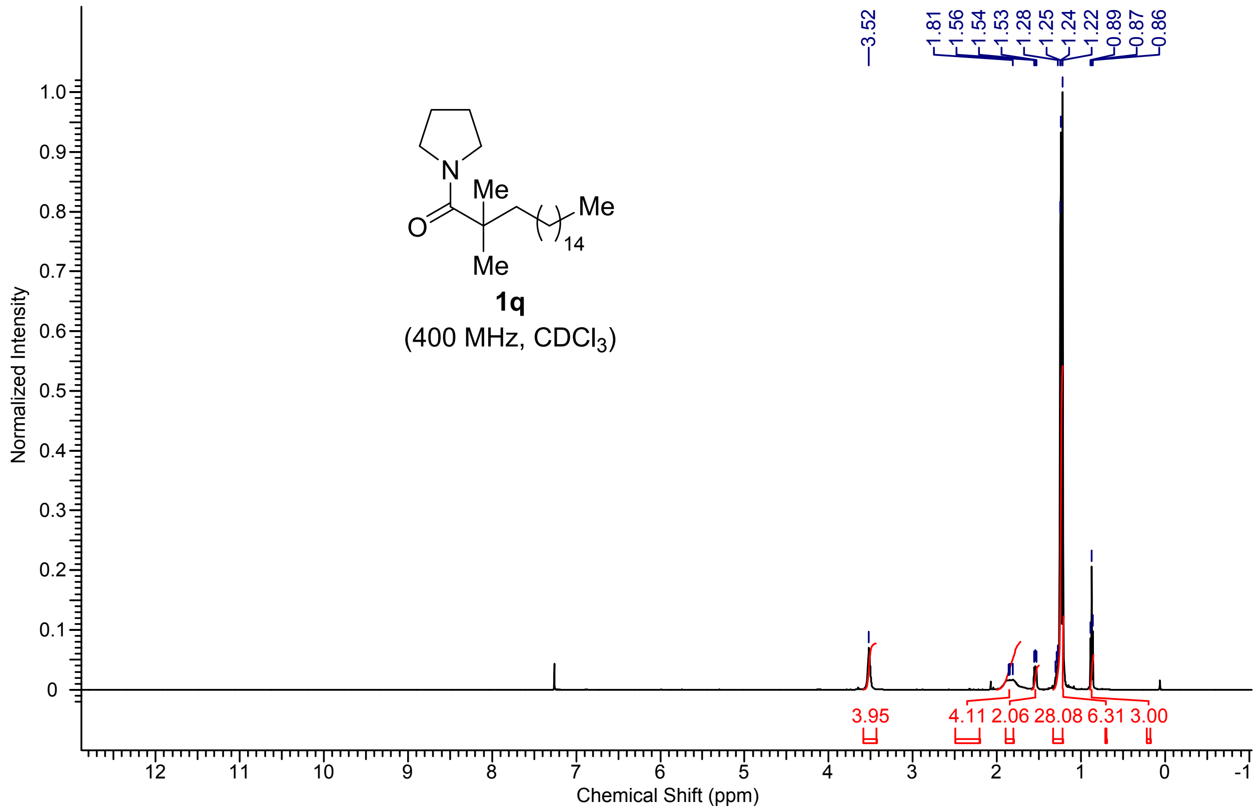

Normalized Intensity
O2N
N
Me
O
Me
Me
Me
1n
(125 MHz, CDCl3)
178.22
151.51
146.62
129.27
124.88
41.06
41.04
29.39
Chemical Shift (ppm)


7.26
3.52
3.50
3.48
1.78
1.76
1.74
1.73
1.42
1.40
1.37
1.14
0.83
0.81
0.79
N
Et
Et
O
Me
1p
(400 MHz, $CDCl_3$)
Normalized Intensity
4.00
5.89
2.11
3.09
5.97
Chemical Shift (ppm)

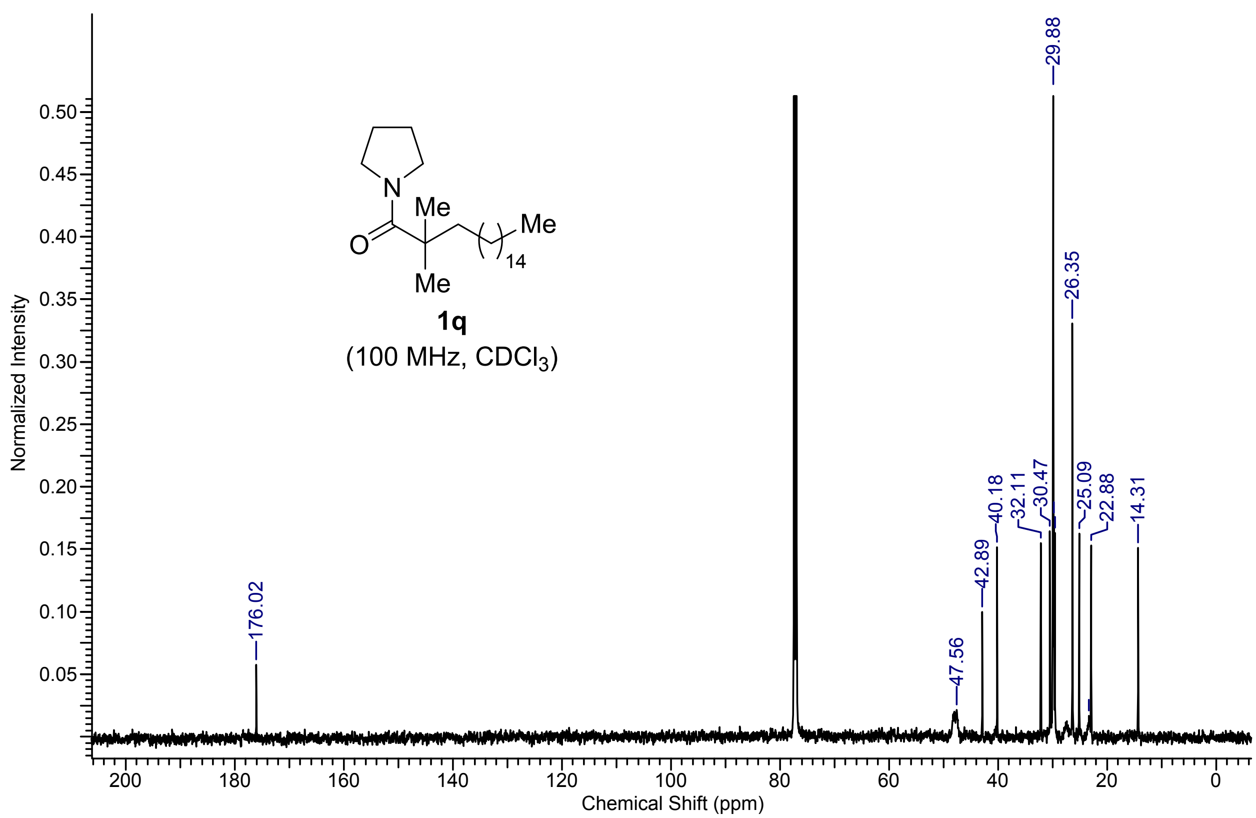

31.14
9.20
N
Et
Et
O
Me
1p
(100 MHz, $CDCl_3$)
22.47
47.48
174.96
48.10
27.57
Normalized Intensity
Chemical Shift (ppm)


3.53
3.51
3.49
1.80
1.78
1.77
1.75
1.73
1.43
1.41
1.39
0.84
0.82
0.80
N
Et
Et
O
CD3
[D]-**1p**
(400 MHz, CDCl3)
Normalized Intensity
4.00
6.16
2.12
6.05
Chemical Shift (ppm)

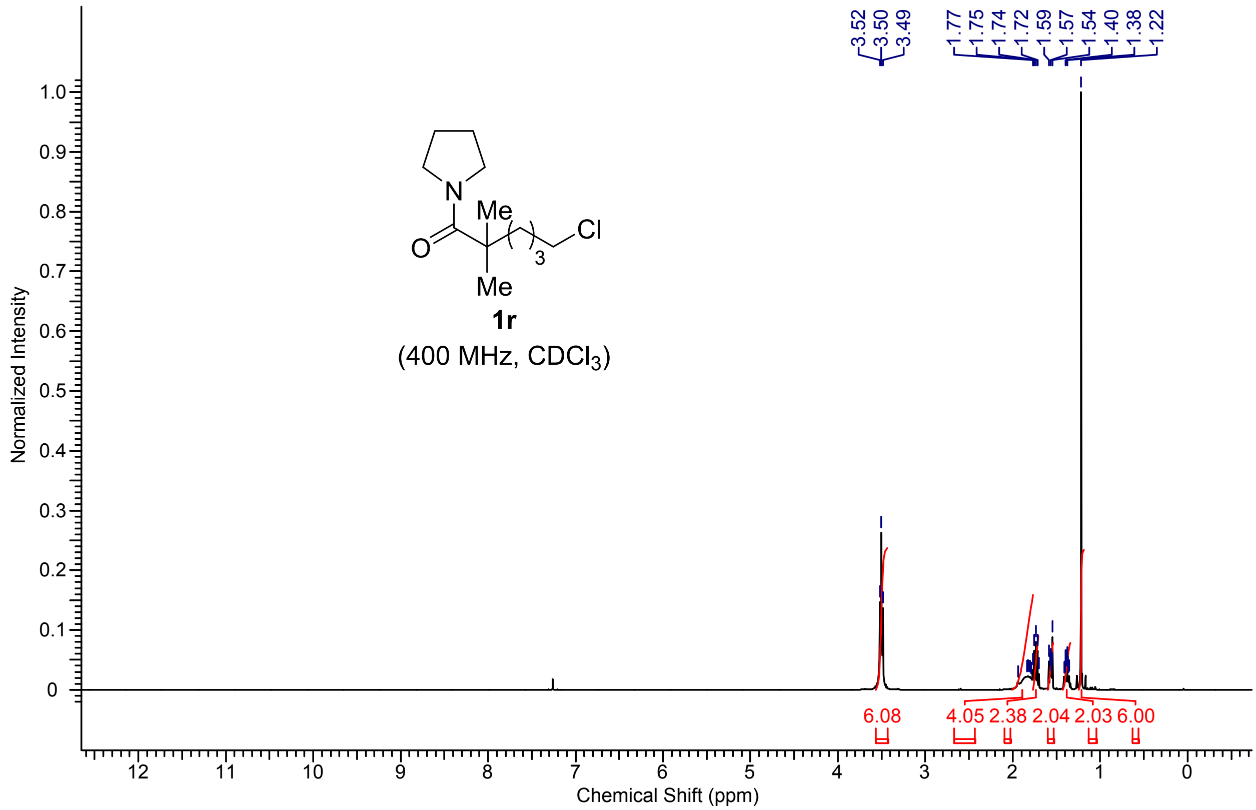

31.10
9.21
N
Et
Et
O
CD3
[D]-**1p**
(100 MHz, CDCl3)
Normalized Intensity
175.06
47.30
48.01
27.61
23.14
Chemical Shift (ppm)

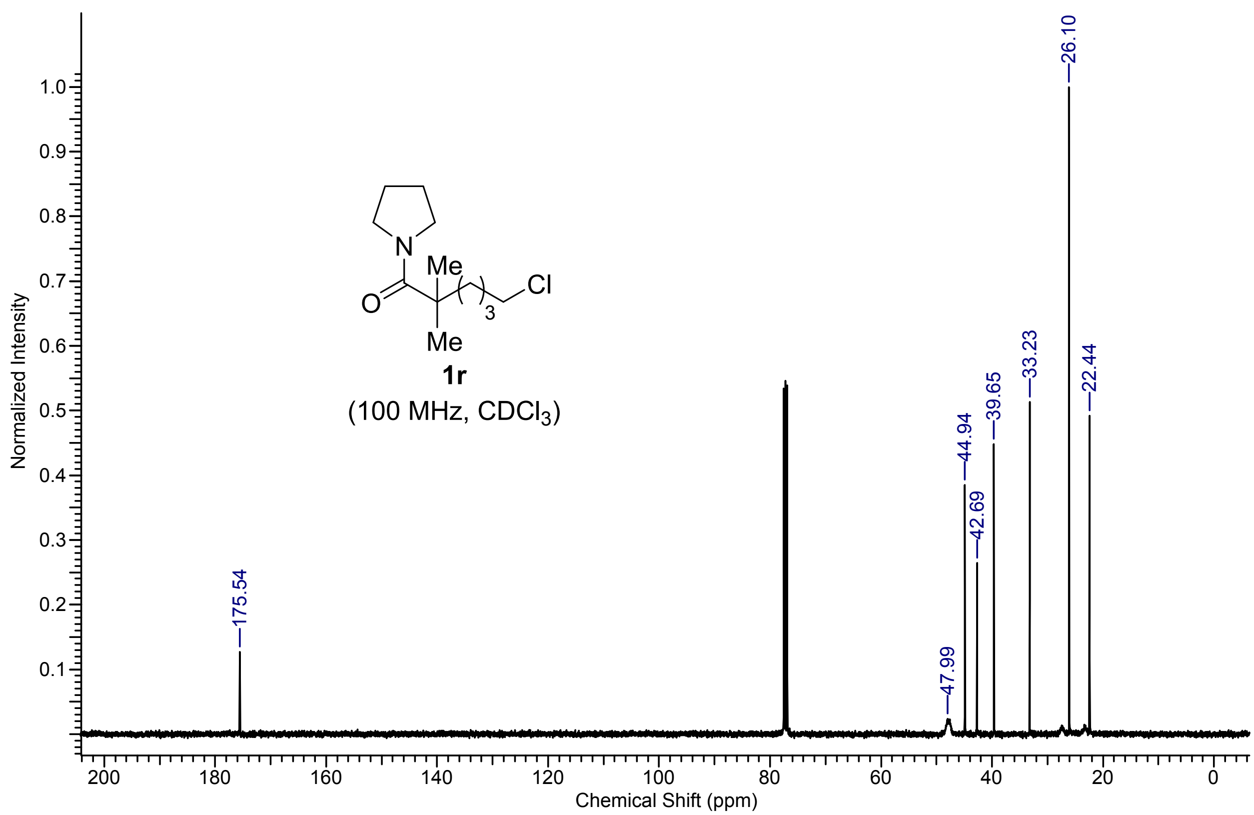


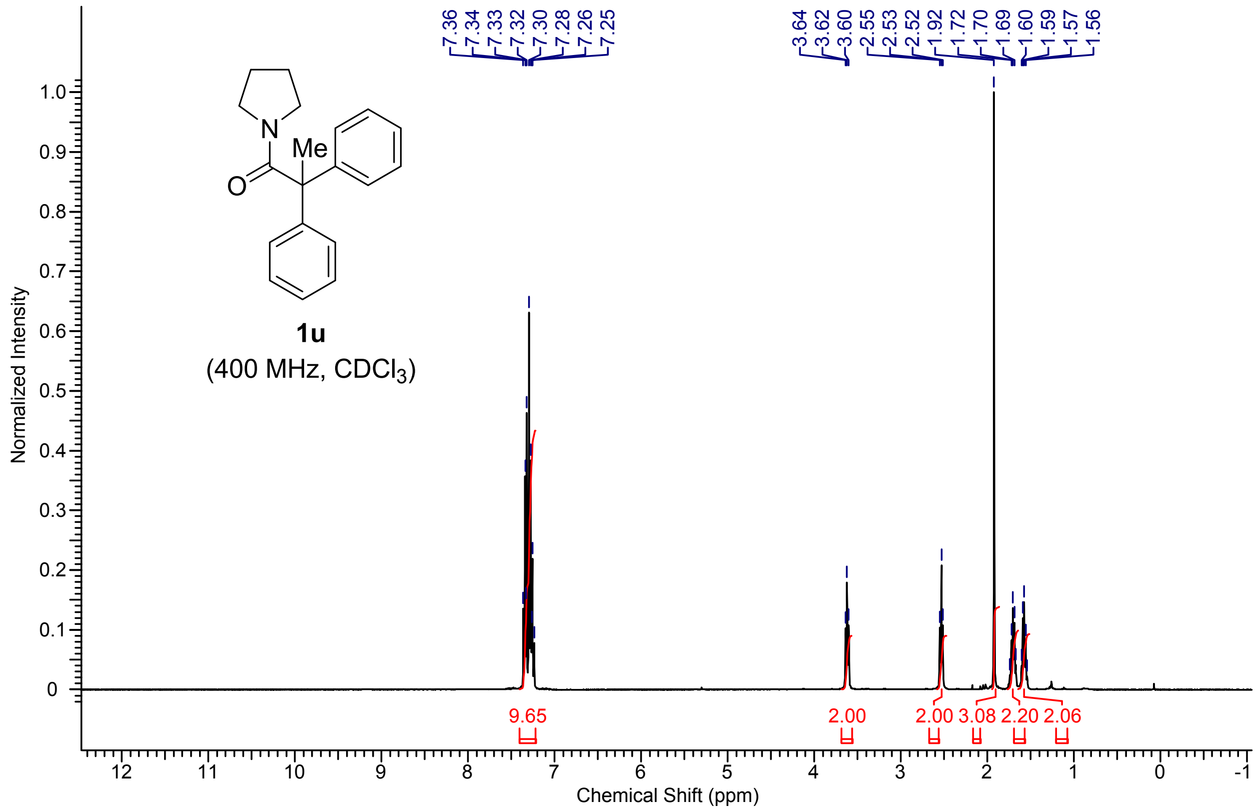
3.52
1.81
1.56
1.54
1.53
1.28
1.25
1.24
1.22
0.89
0.87
0.86
N
Me
Me
O
Me
14
1q
(400 MHz, CDCl3)
3.95
4.11
2.06
28.08
6.31
3.00
Normalized Intensity
Chemical Shift (ppm)

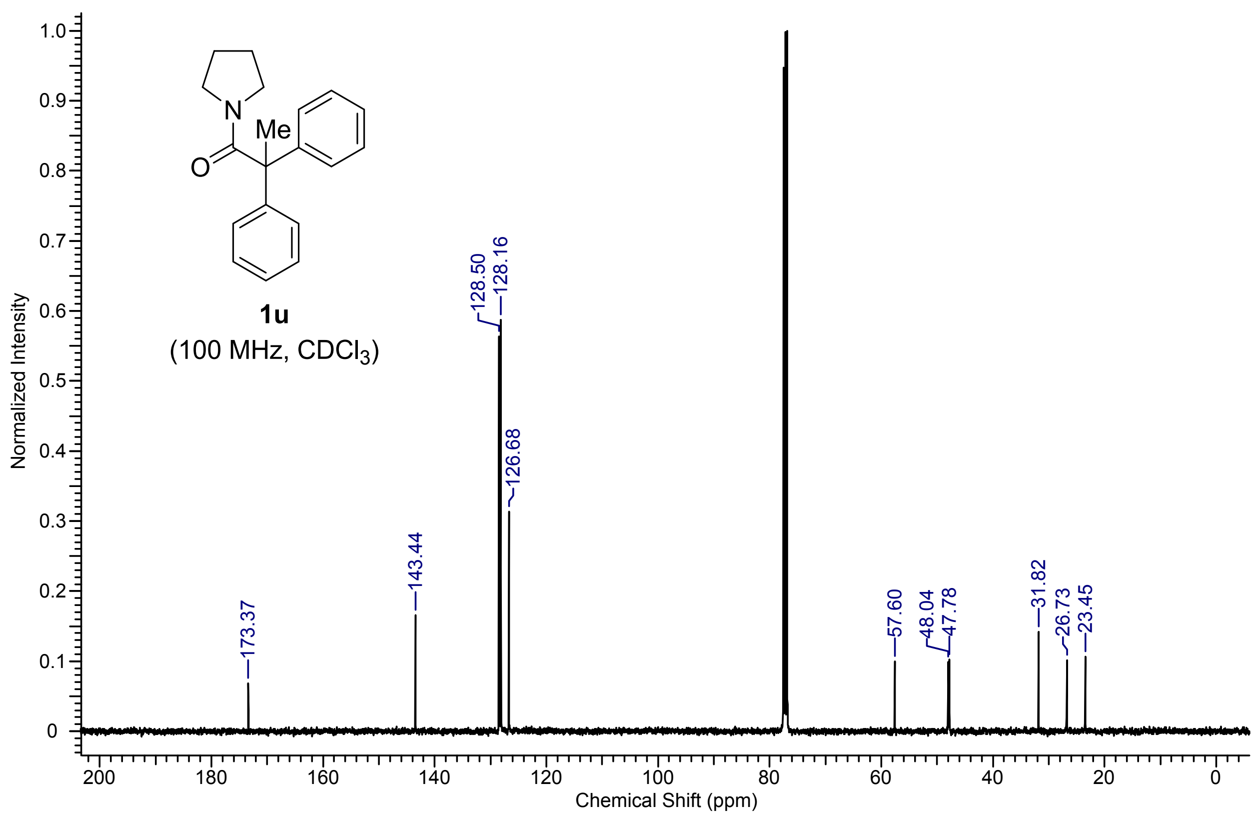


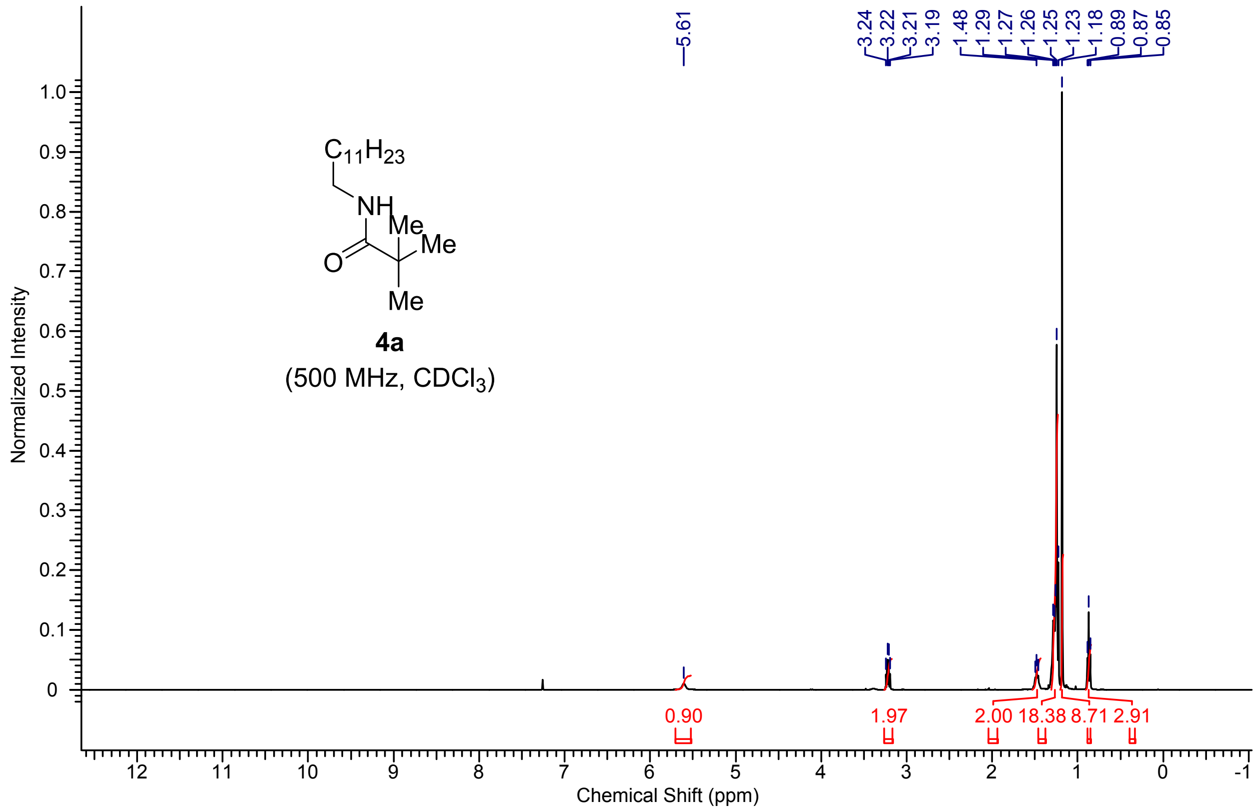
29.88
26.35
40.18
32.11
30.47
25.09
22.88
14.31
42.89
47.56
176.02
N
Me
Me
O
Me
14
1q
(100 MHz, CDCl3)
Normalized Intensity
Chemical Shift (ppm)

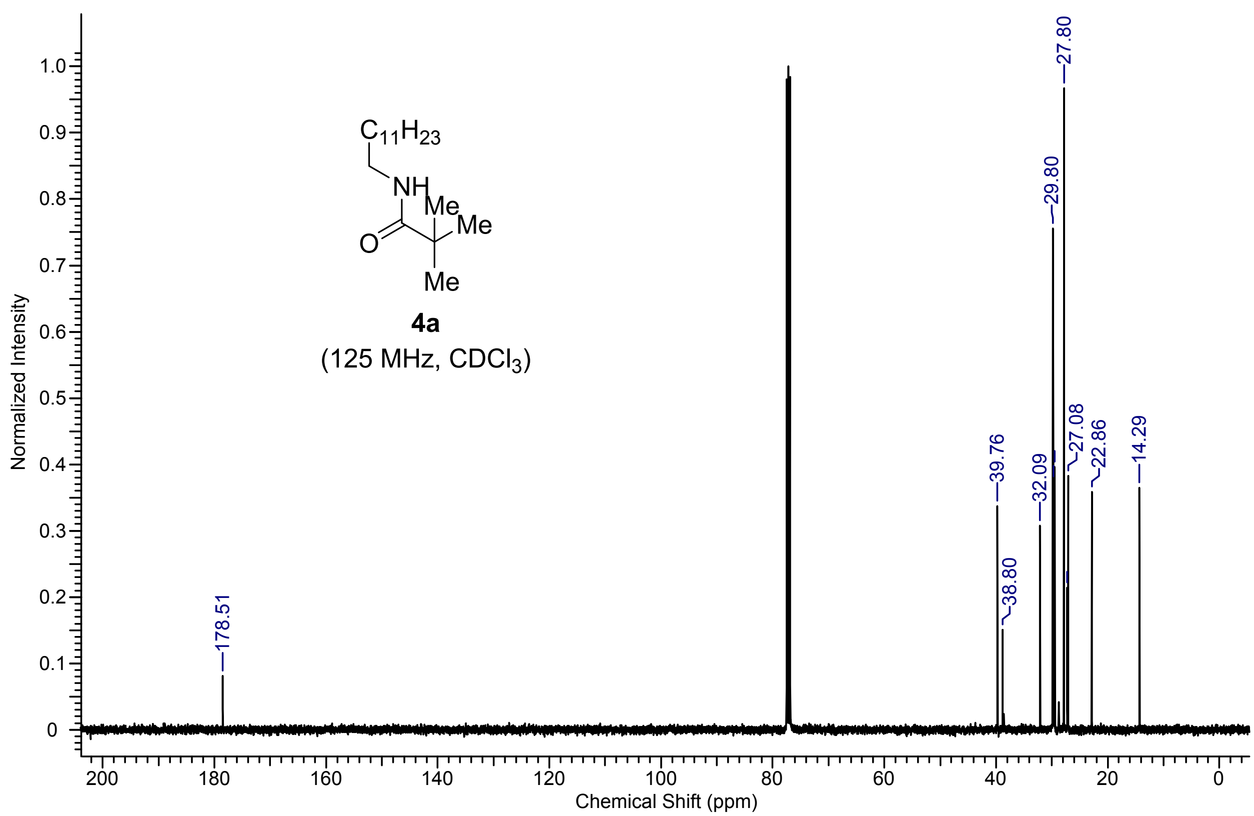

3.52
3.50
3.49
1.77
1.75
1.74
1.72
1.59
1.57
1.54
1.40
1.38
1.22
Me
N
O
3
Cl
Me
1r
(400 MHz, CDCl3)
Normalized Intensity
6.08
4.05
2.38
2.04
2.03
6.00
Chemical Shift (ppm)

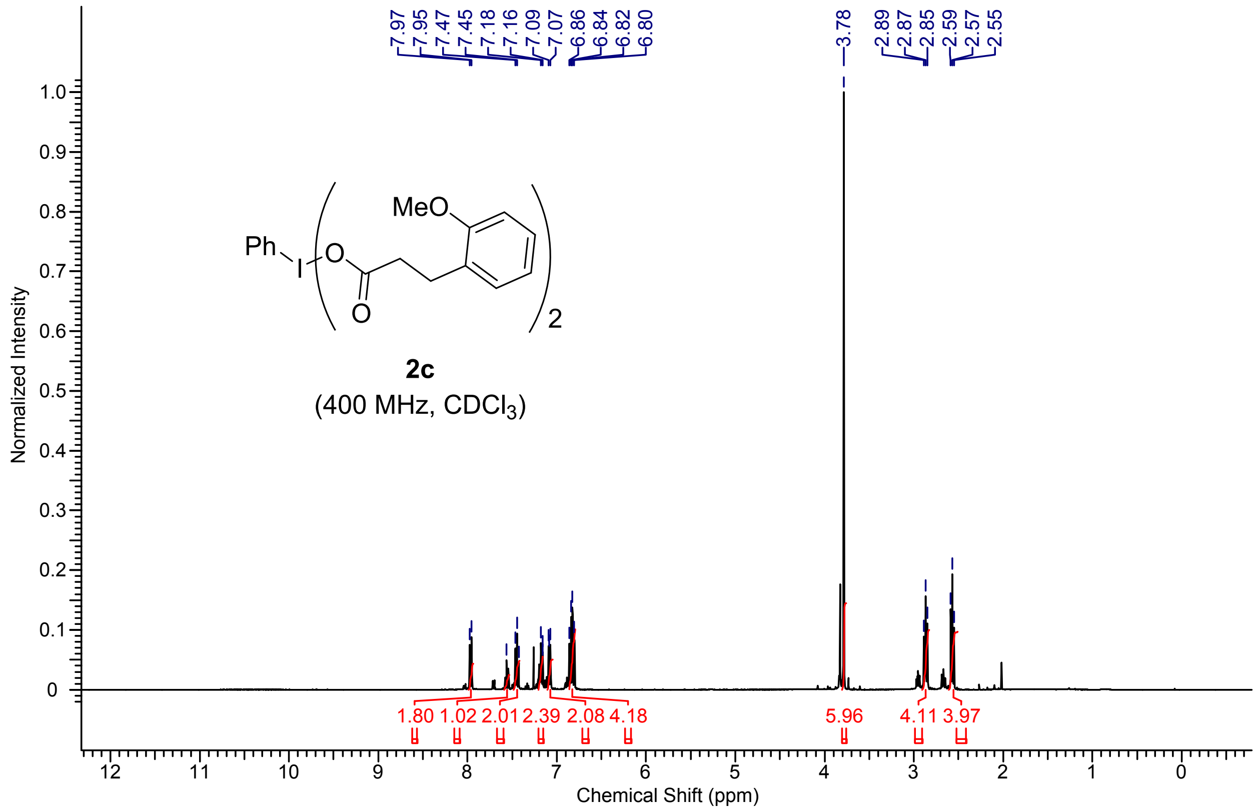

26.10
N
Me
O
3
Cl
Me
1r
(100 MHz, CDCl3)
Normalized Intensity
175.54
47.99
44.94
42.69
39.65
33.23
22.44
Chemical Shift (ppm)

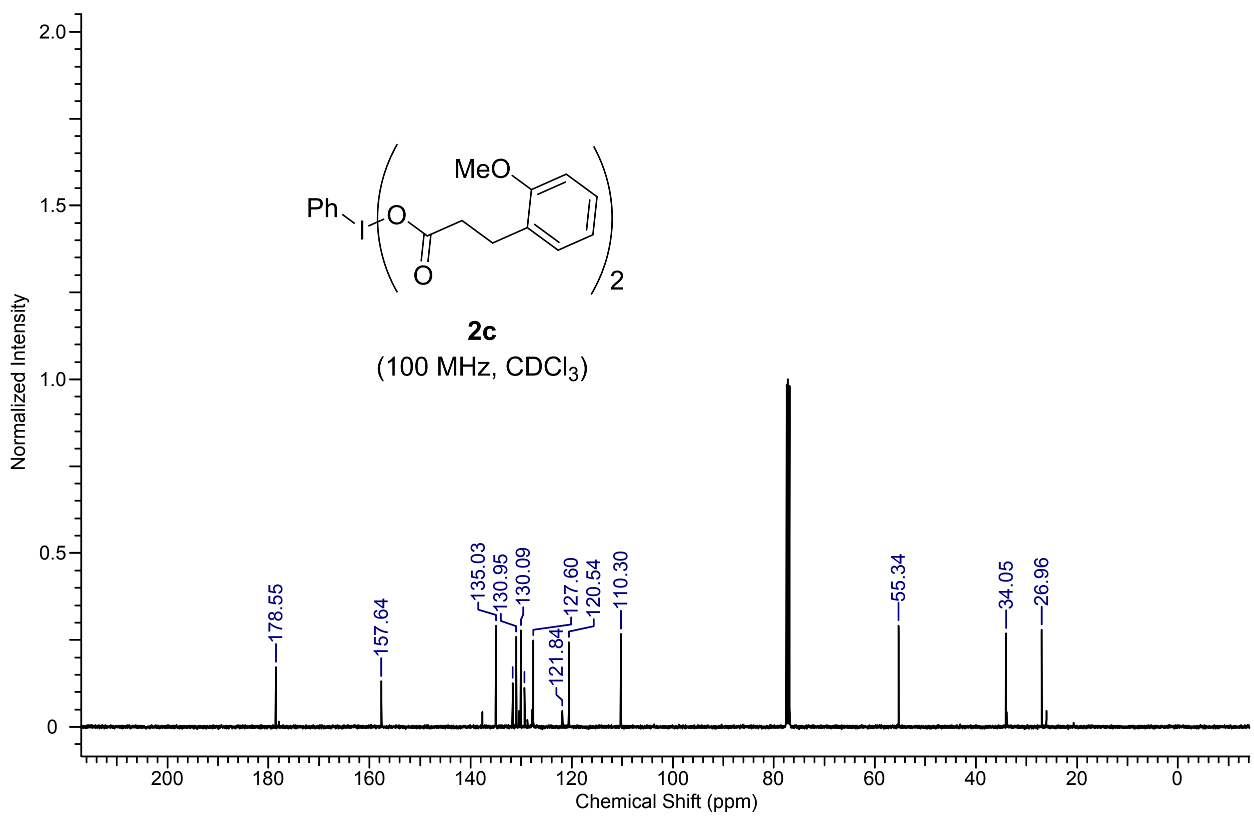

1u
(400 MHz, $CDCl_3$)
Chemical Shift (ppm)
Normalized Intensity


1u
(100 MHz, $CDCl_3$)
Chemical Shift (ppm)
Normalized Intensity

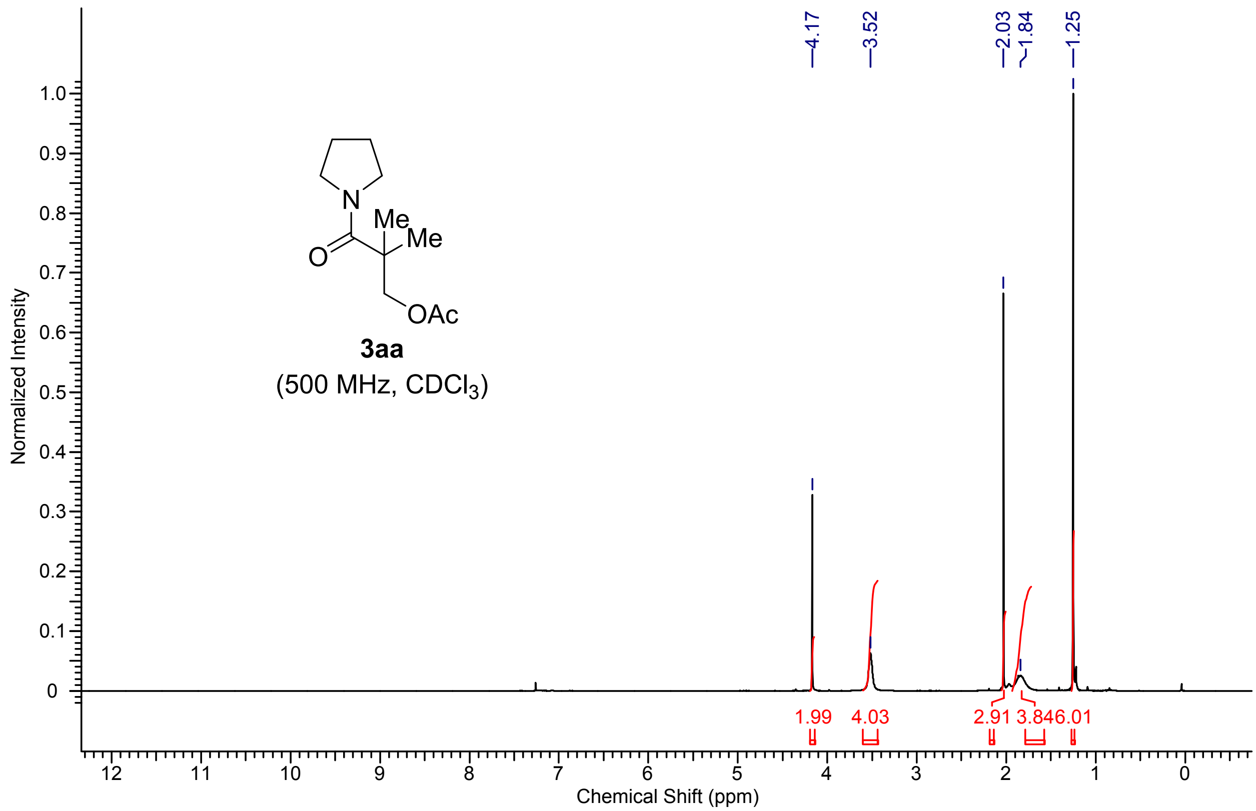

5.61
3.24
3.22
3.21
3.19
1.48
1.29
1.27
1.26
1.25
1.23
1.18
0.89
0.87
0.85
$C_{11}H_{23}$
NH
O
Me
Me
Me
4a
(500 MHz, $CDCl_3$)
Normalized Intensity
0.90
1.97
2.00
18.38
8.71
2.91
Chemical Shift (ppm)

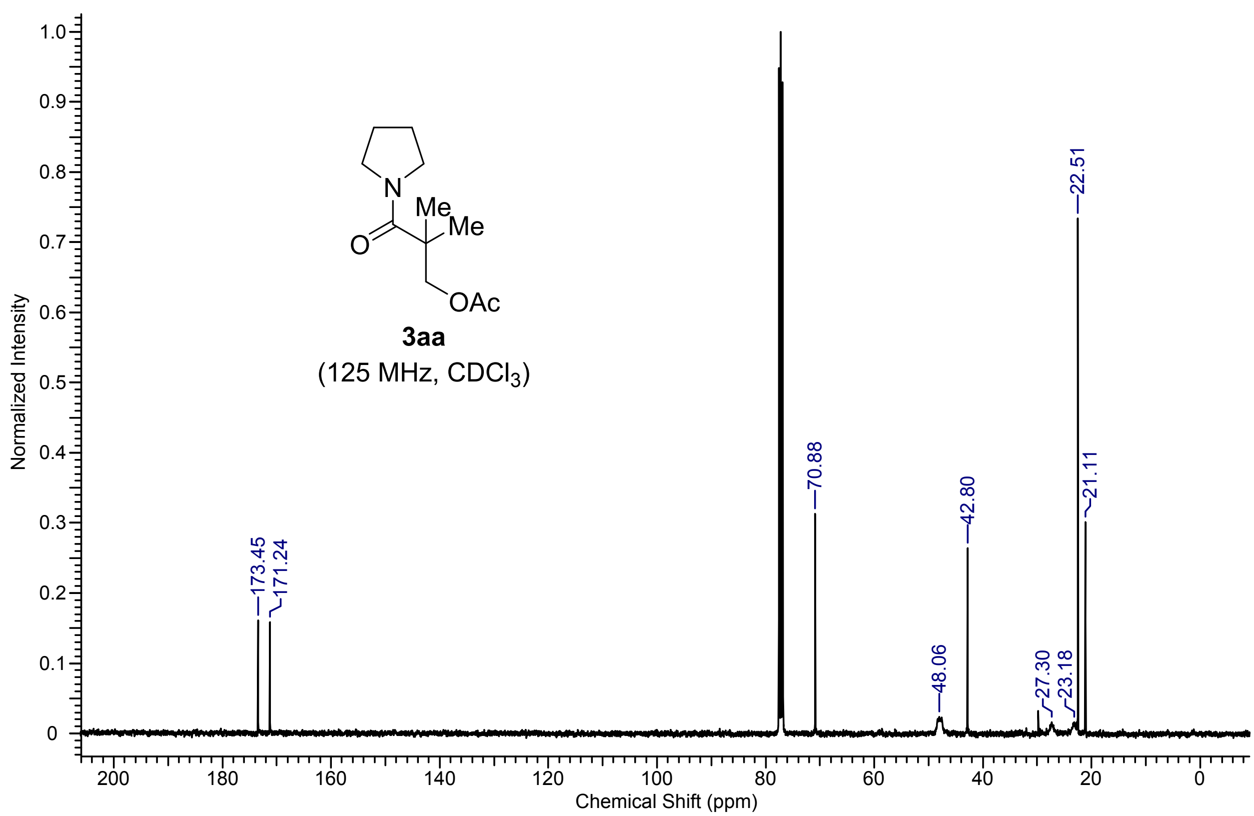

178.51
39.76
38.80
32.09
29.80
27.80
27.08
22.86
14.29
$C_{11}H_{23}$
NH
O
Me
Me
Me
4a
(125 MHz, $CDCl_3$)
Normalized Intensity
Chemical Shift (ppm)

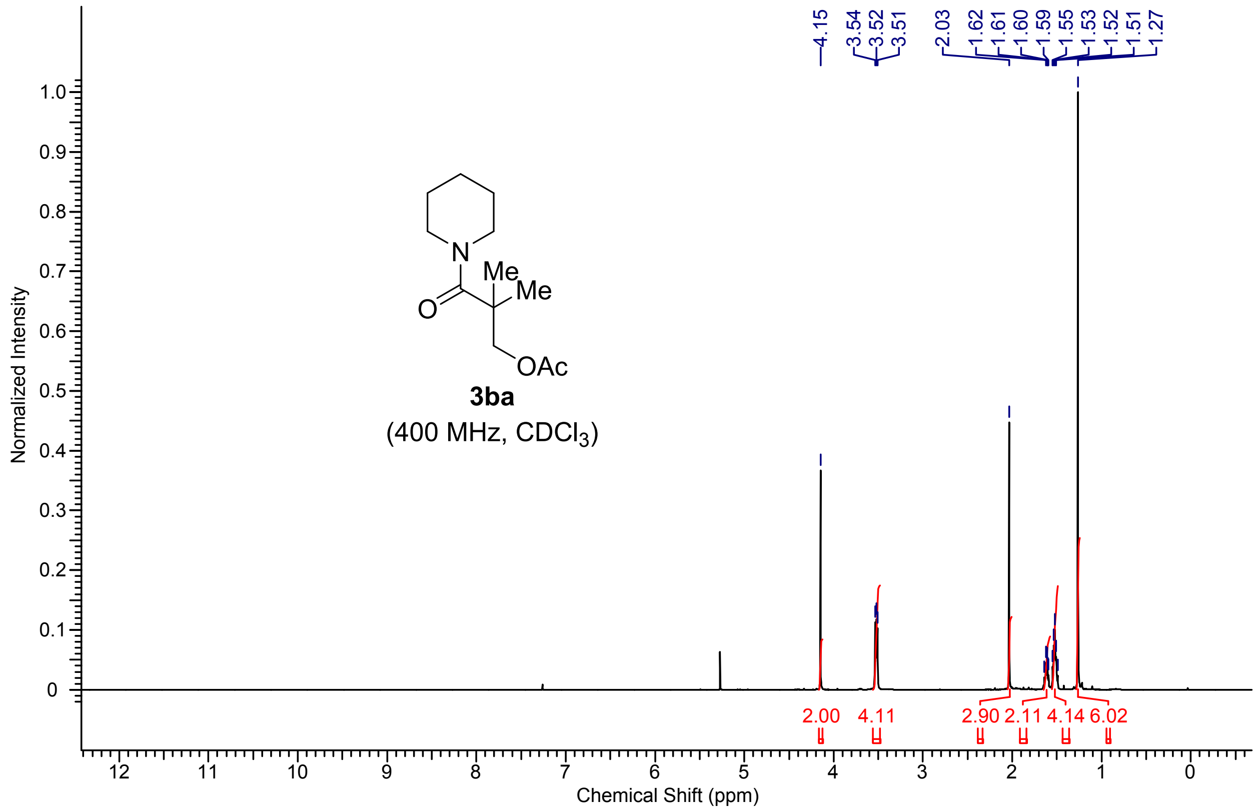

7.97
7.95
7.47
7.45
7.18
7.16
7.09
7.07
6.86
6.84
6.82
6.80
3.78
2.89
2.87
2.85
2.59
2.57
2.55
MeO
Ph
I
O
O
2
2c
(400 MHz, CDCl3)
1.80 1.02 2.01 2.39 2.08 4.18
5.96
4.11 3.97
Normalized Intensity
Chemical Shift (ppm)

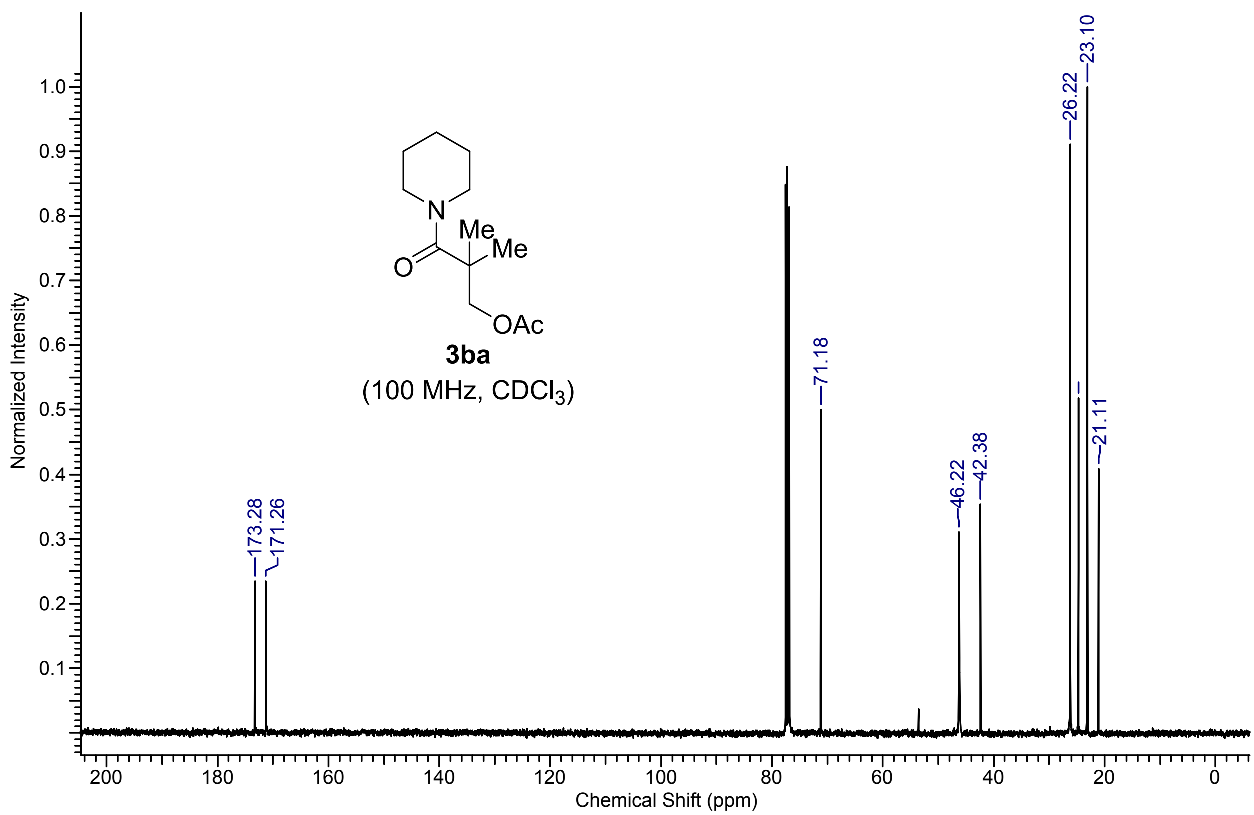

MeO
Ph
I
O
O
2
2c
(100 MHz, CDCl3)
178.55
157.64
135.03
130.95
130.09
121.84
127.60
120.54
110.30
55.34
34.05
26.96
Normalized Intensity
Chemical Shift (ppm)

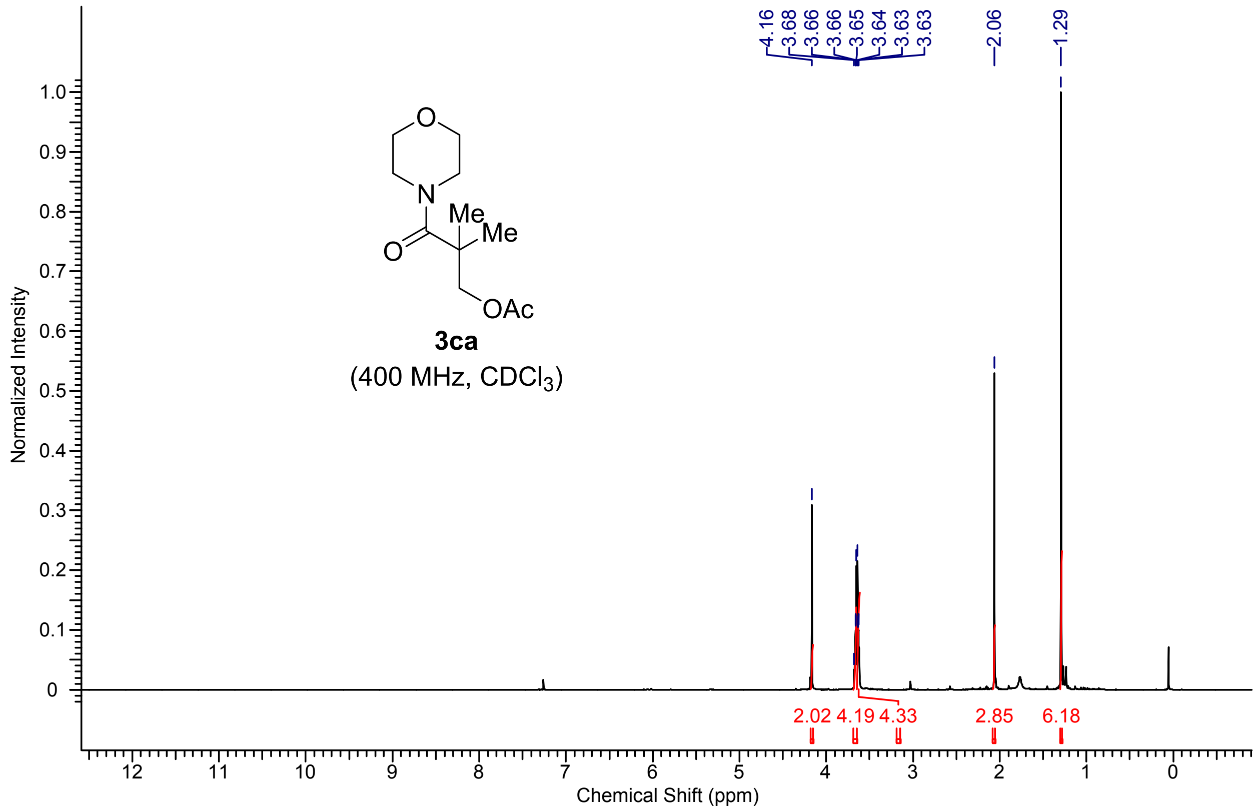
5. ^{1}H and ^{13}C NMR Spectra of Acetoxylated Compounds

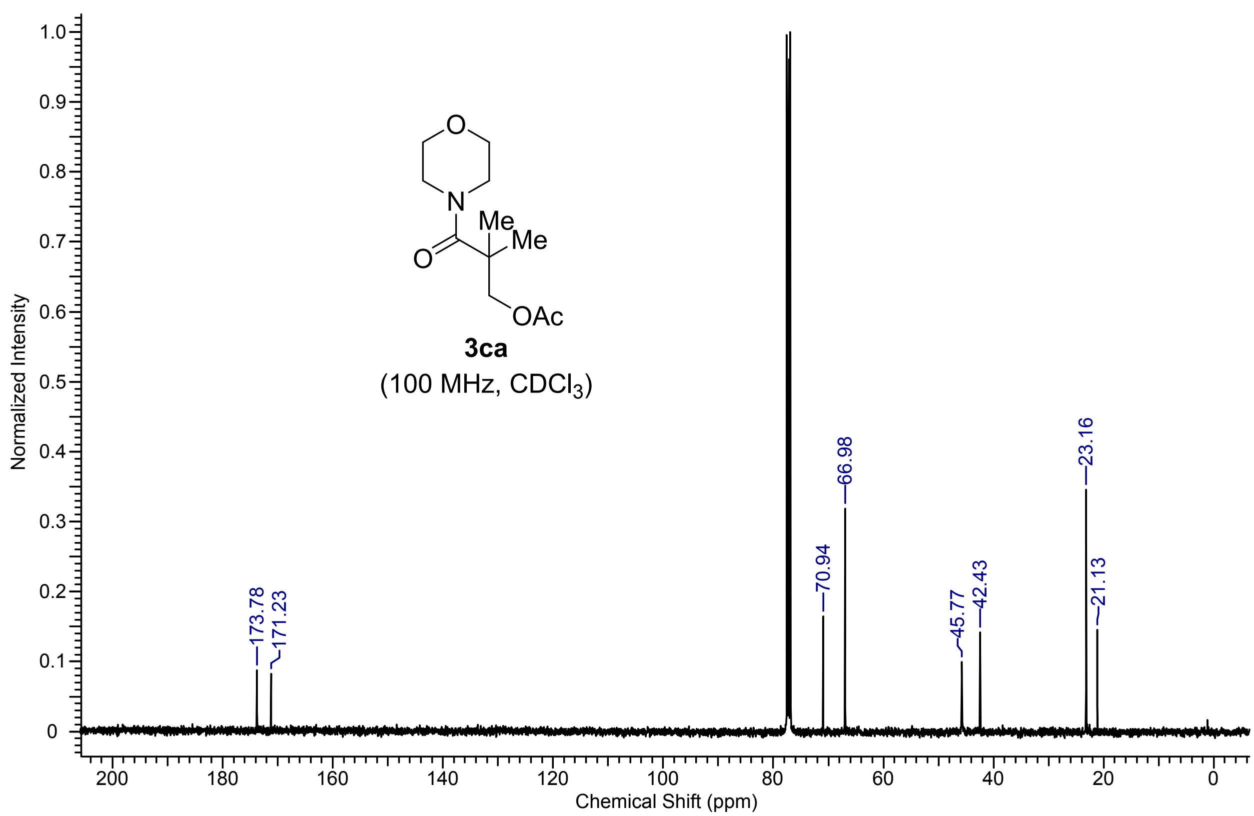


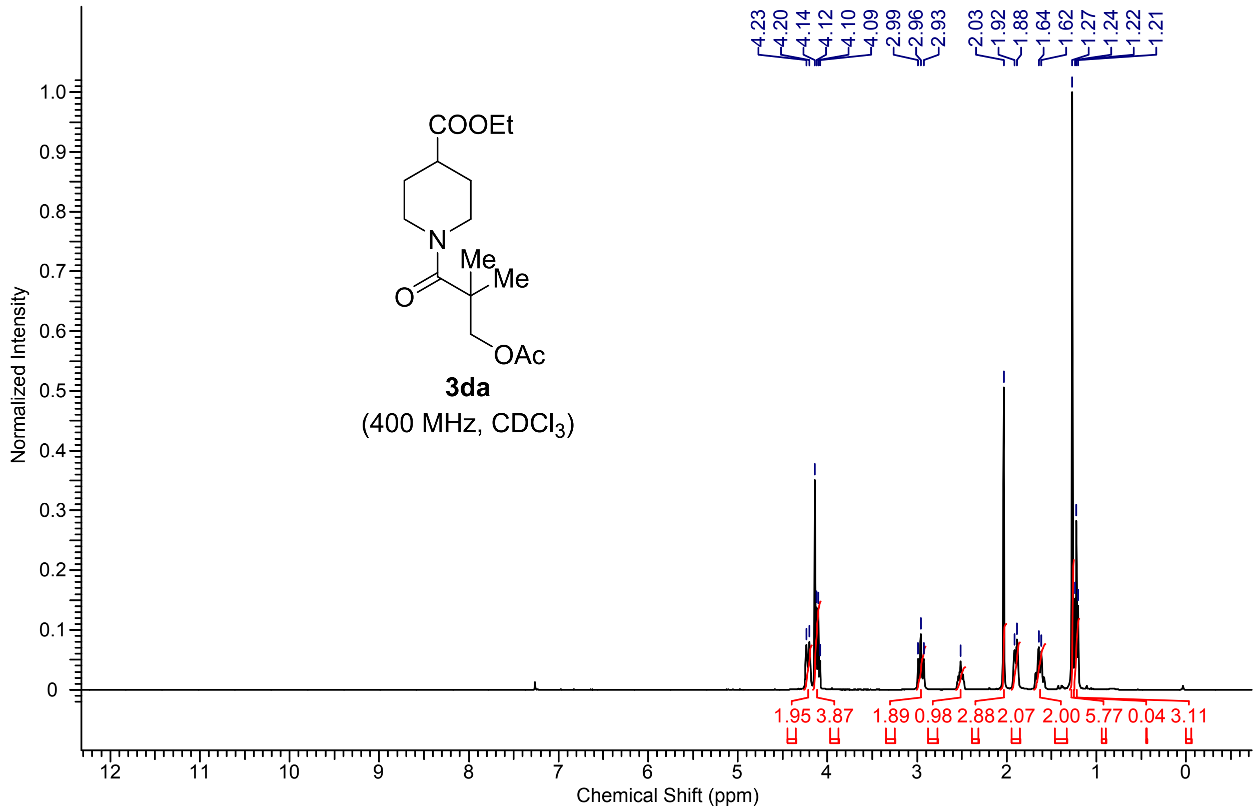


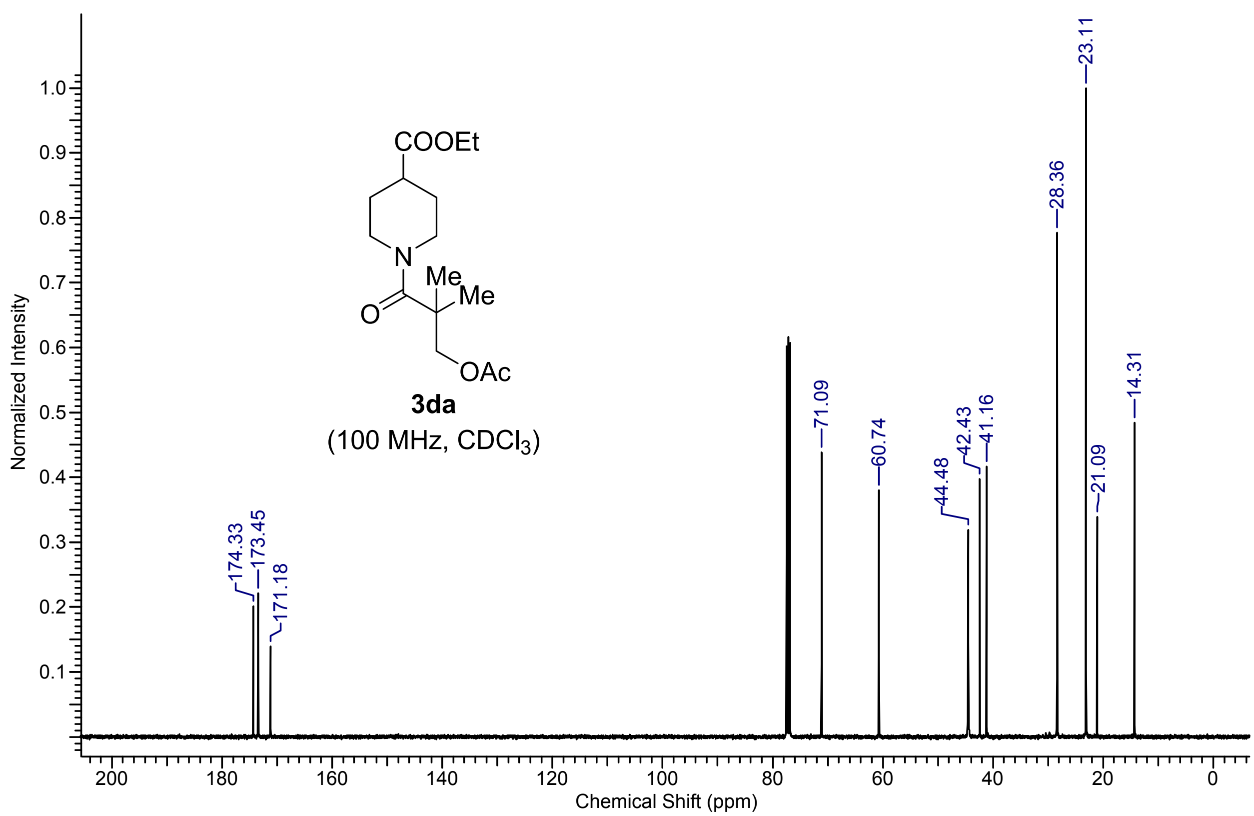

4.15
3.54
3.52
3.51
2.03
1.62
1.61
1.60
1.59
1.55
1.53
1.52
1.51
1.27
Normalized Intensity
Me
Me
N
O
OAc
3ba
(400 MHz, CDCl3)
2.00
4.11
2.90
2.11
4.14
6.02
Chemical Shift (ppm)

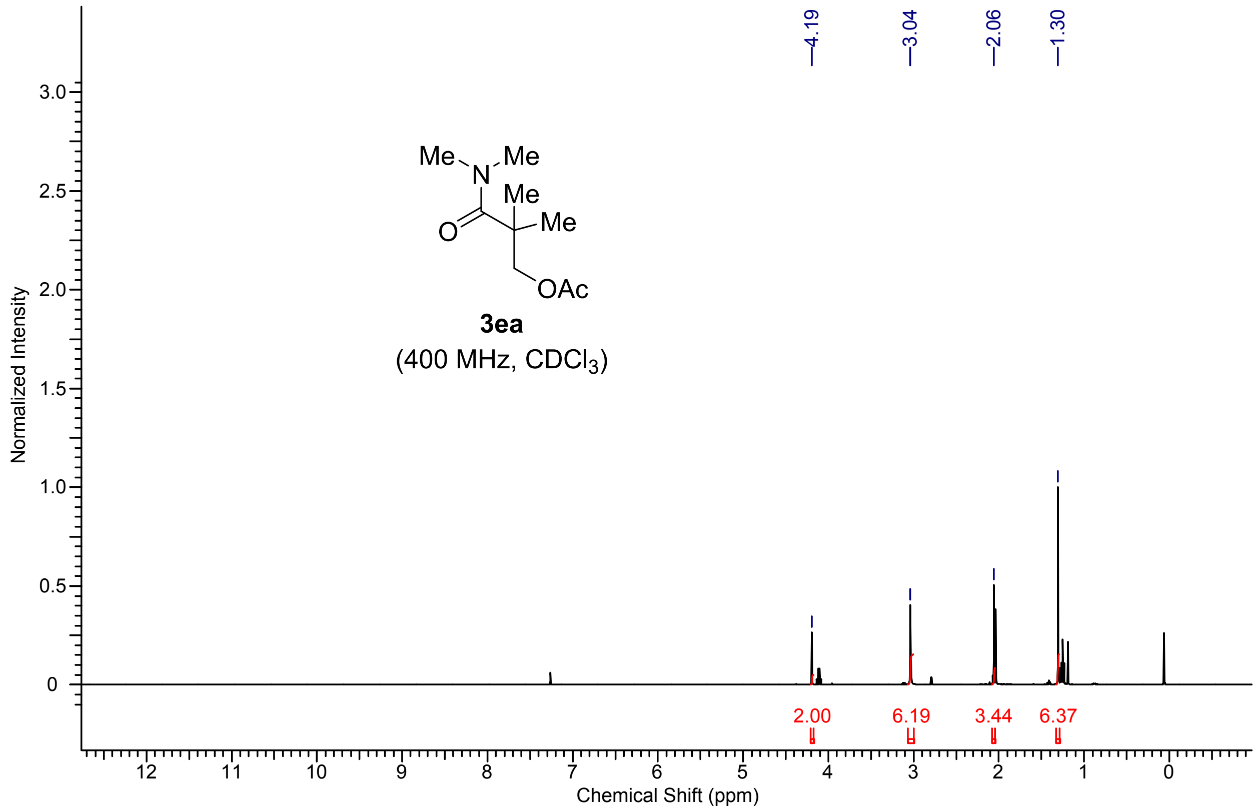

173.28
171.26
71.18
46.22
42.38
26.22
23.10
21.11
Normalized Intensity
Me
Me
N
O
OAc
3ba
(100 MHz, CDCl3)
Chemical Shift (ppm)

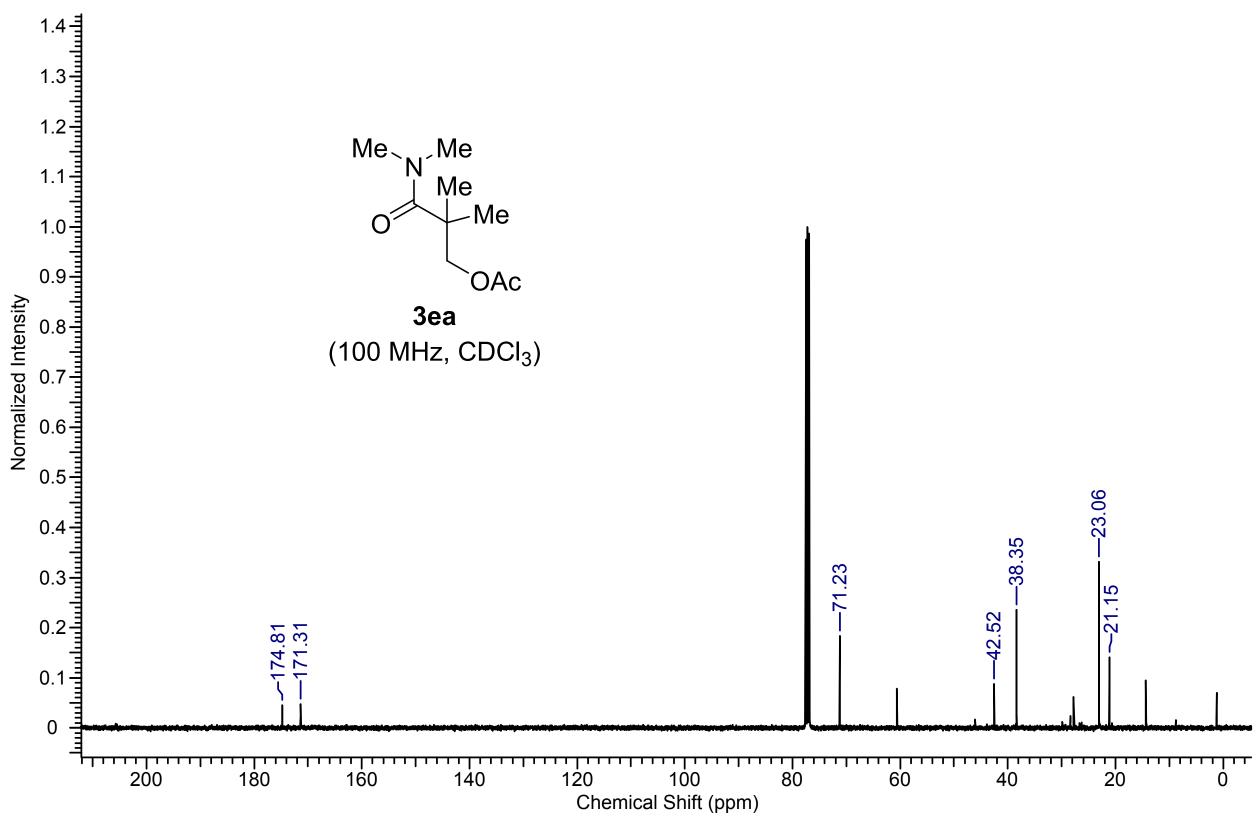

4.16
3.68
3.66
3.66
3.65
3.64
3.63
3.63
2.06
1.29
O
N
Me
Me
O
OAc
3ca
(400 MHz, CDCl3)
Normalized Intensity
2.02
4.19
4.33
2.85
6.18
Chemical Shift (ppm)

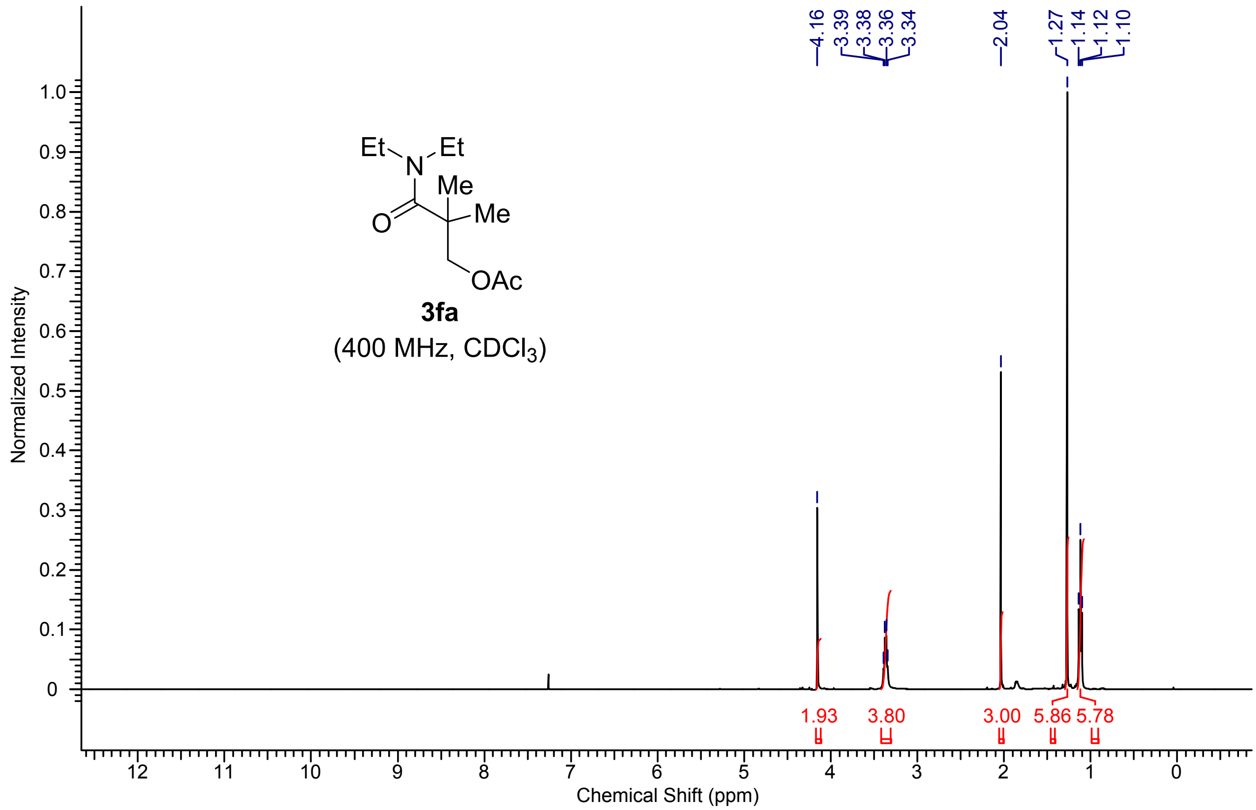

O
N
Me
Me
O
OAc
3ca
(100 MHz, CDCl3)
Normalized Intensity
173.78
171.23
70.94
66.98
45.77
42.43
23.16
21.13
Chemical Shift (ppm)

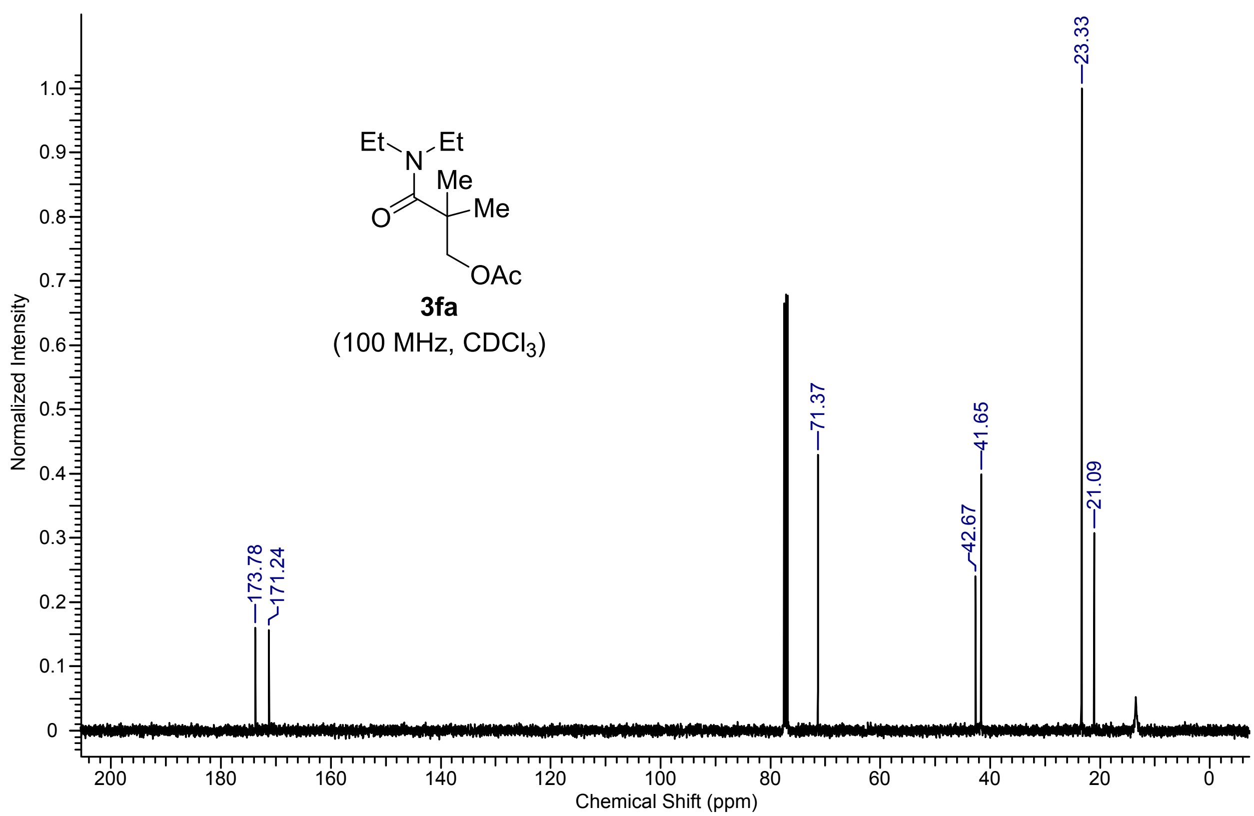

4.23 4.20 4.14 4.12 4.10 4.09 2.99 2.96 2.93 2.03 1.92 1.88 1.64 1.62 1.27 1.24 1.22 1.21
COOEt
N
Me
Me
O
OAc
3da
(400 MHz, CDCl3)
1.95 3.87 1.89 0.98 2.88 2.07 2.00 5.77 0.04 3.11
Normalized Intensity
Chemical Shift (ppm)

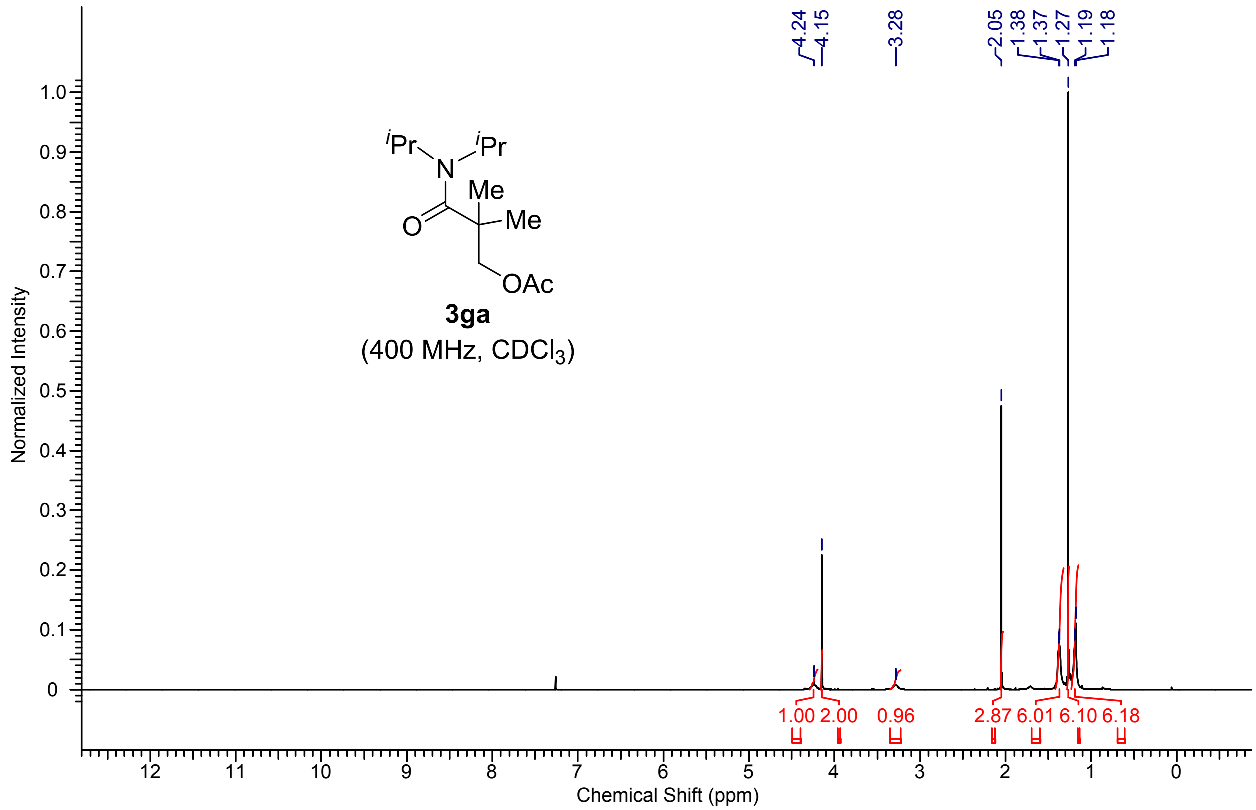

174.33 173.45 171.18 71.09 60.74 44.48 42.43 41.16 28.36 23.11 21.09 14.31
COOEt
N
Me
Me
O
OAc
3da
(100 MHz, CDCl3)
Normalized Intensity
Chemical Shift (ppm)

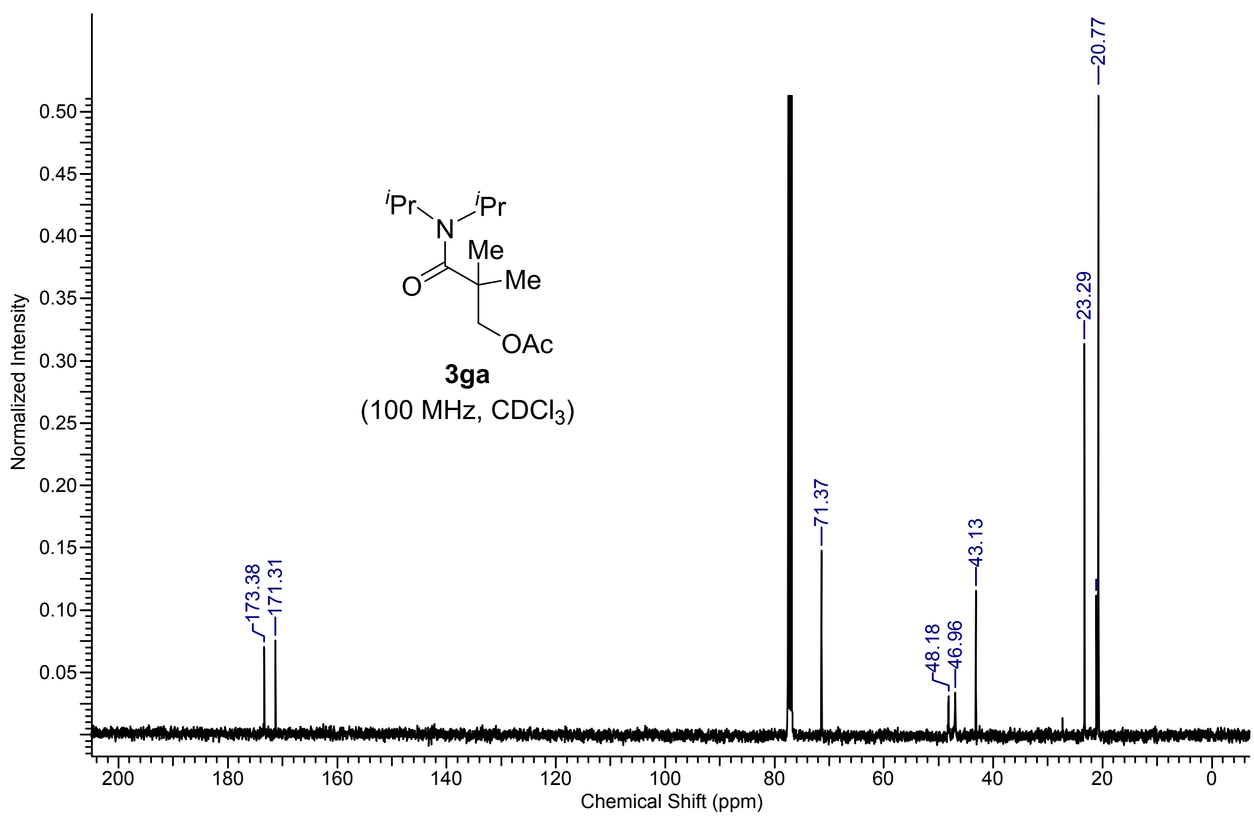

4.19
3.04
2.06
1.30
Me
N
Me
Me
Me
O
OAc
3ea
(400 MHz, CDCl3)
2.00
6.19
3.44
6.37
Normalized Intensity
Chemical Shift (ppm)

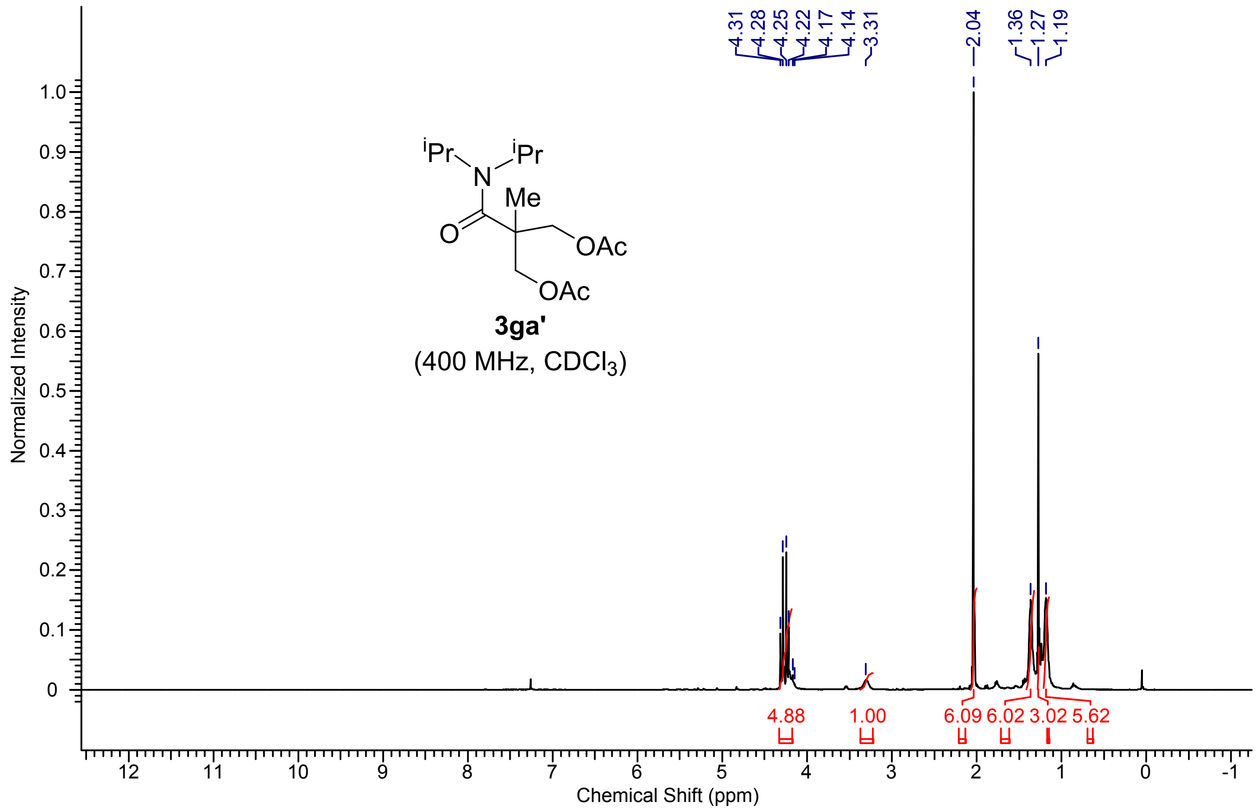

Me
N
Me
Me
Me
O
OAc
3ea
(100 MHz, CDCl3)
174.81
171.31
71.23
42.52
38.35
23.06
21.15
Normalized Intensity
Chemical Shift (ppm)


4.16
3.39
3.38
3.36
3.34
2.04
1.27
1.14
1.12
1.10
Et
N
Et
Me
O
Me
OAc
3fa
(400 MHz, CDCl3)
1.93
3.80
3.00
5.86
5.78
Normalized Intensity
Chemical Shift (ppm)

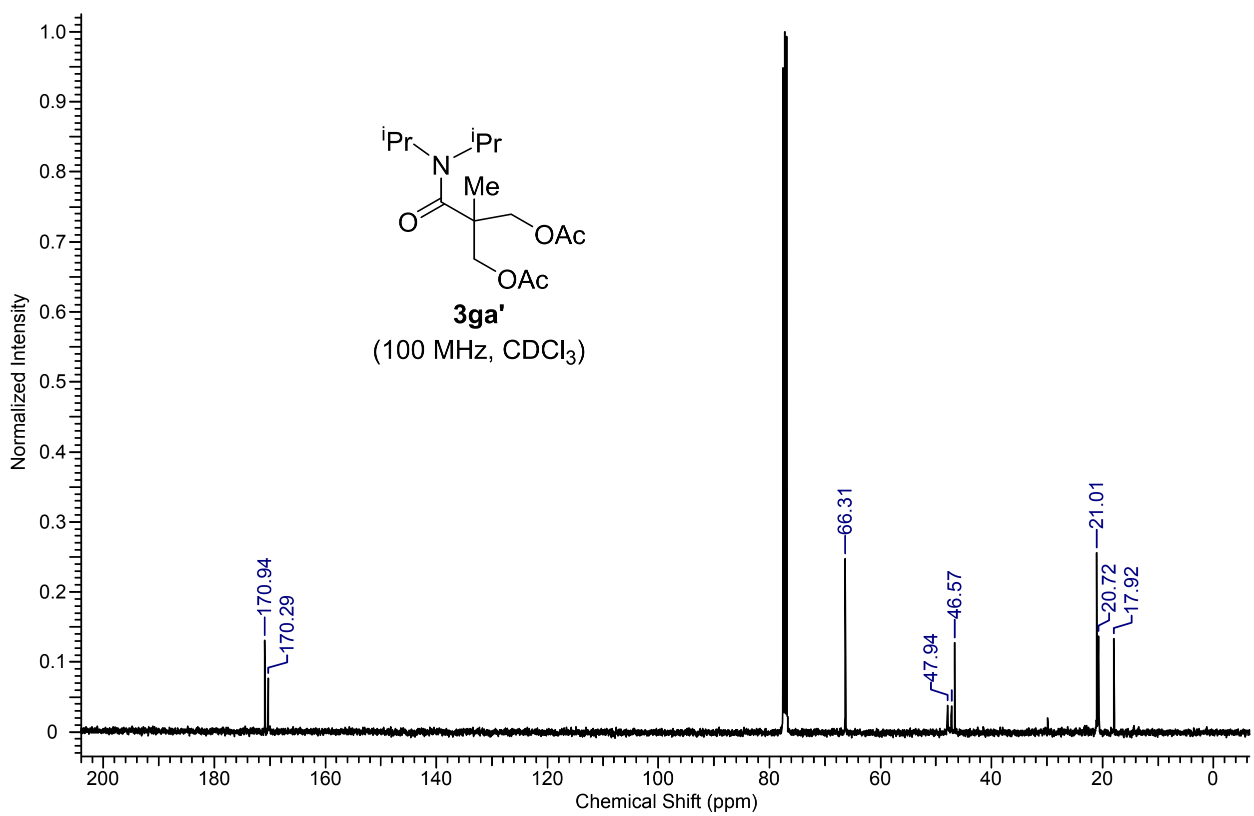

23.33
Et
N
Et
Me
O
Me
OAc
3fa
(100 MHz, CDCl3)
71.37
41.65
21.09
42.67
173.78
171.24
Normalized Intensity
Chemical Shift (ppm)


4.24
4.15
3.28
2.05
1.38
1.37
1.27
1.19
1.18
^{i}Pr
N
^{i}Pr
Me
Me
O
OAc
3ga
(400 MHz, $CDCl_3$)
Normalized Intensity
1.00 2.00 0.96 2.87 6.01 6.10 6.18
Chemical Shift (ppm)

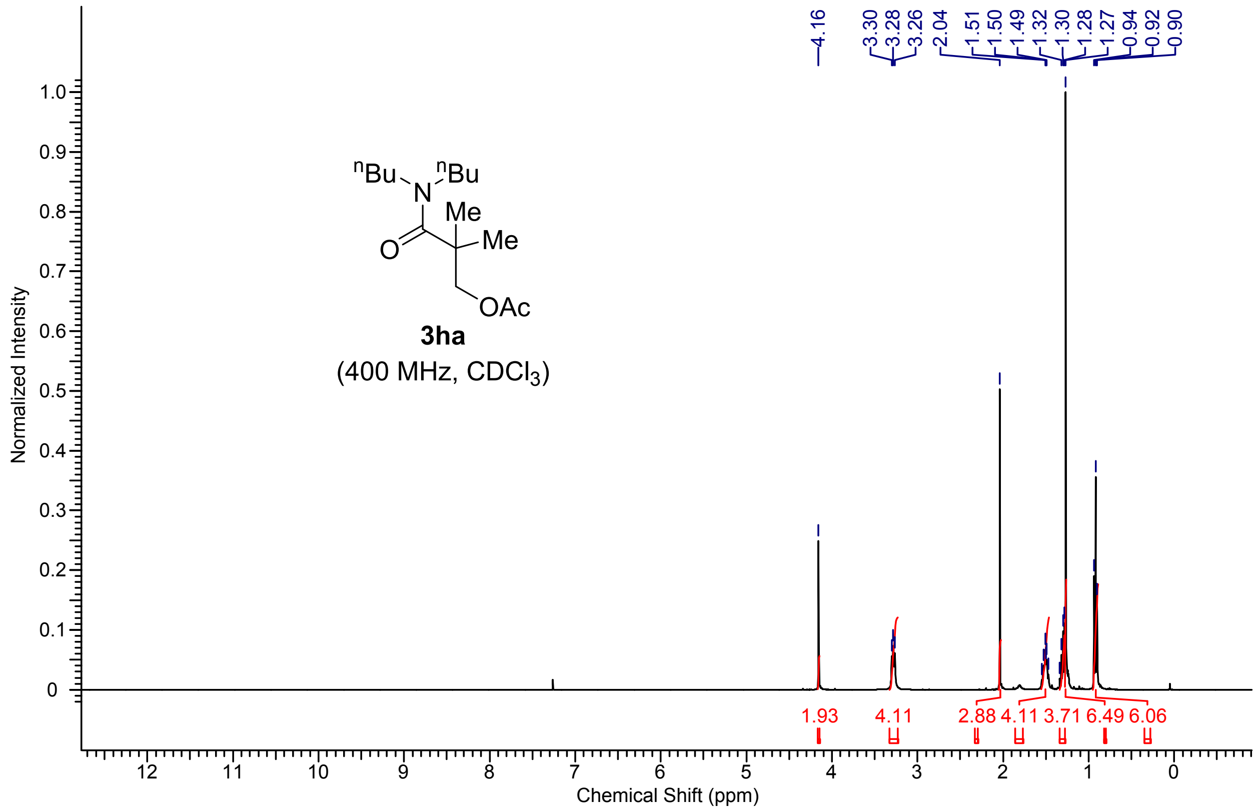

20.77
23.29
71.37
43.13
173.38
171.31
48.18
46.96
^{i}Pr
N
^{i}Pr
Me
Me
O
OAc
3ga
(100 MHz, $CDCl_3$)
Normalized Intensity
Chemical Shift (ppm)



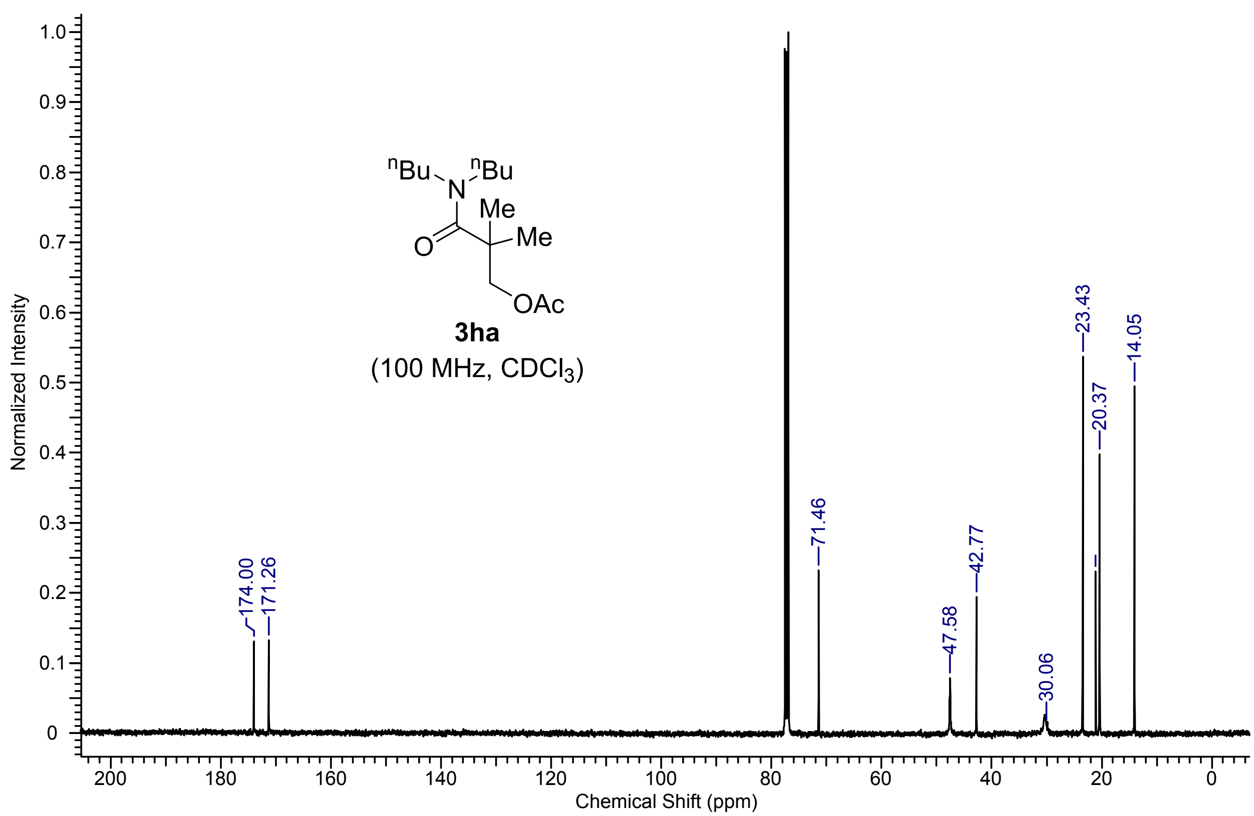
4.31
4.28
4.25
4.22
4.17
4.14
3.31
2.04
1.36
1.27
1.19
iPr
N
iPr
Me
O
OAc
OAc
3ga'
(400 MHz, CDCl3)
Normalized Intensity
4.88
1.00
6.09
6.02
3.02
5.62
Chemical Shift (ppm)



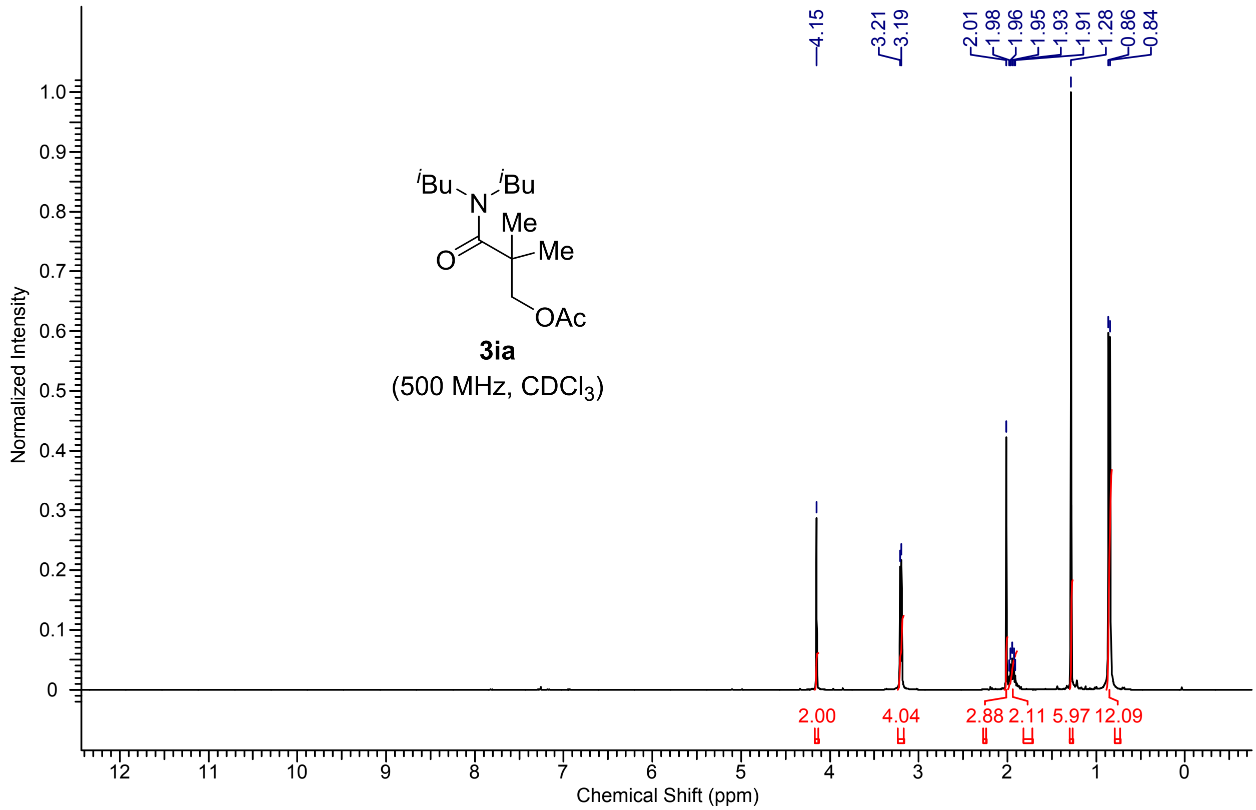
iPr
N
iPr
Me
O
OAc
OAc
3ga'
(100 MHz, CDCl3)
Normalized Intensity
170.94
170.29
66.31
47.94
46.57
21.01
20.72
17.92
Chemical Shift (ppm)



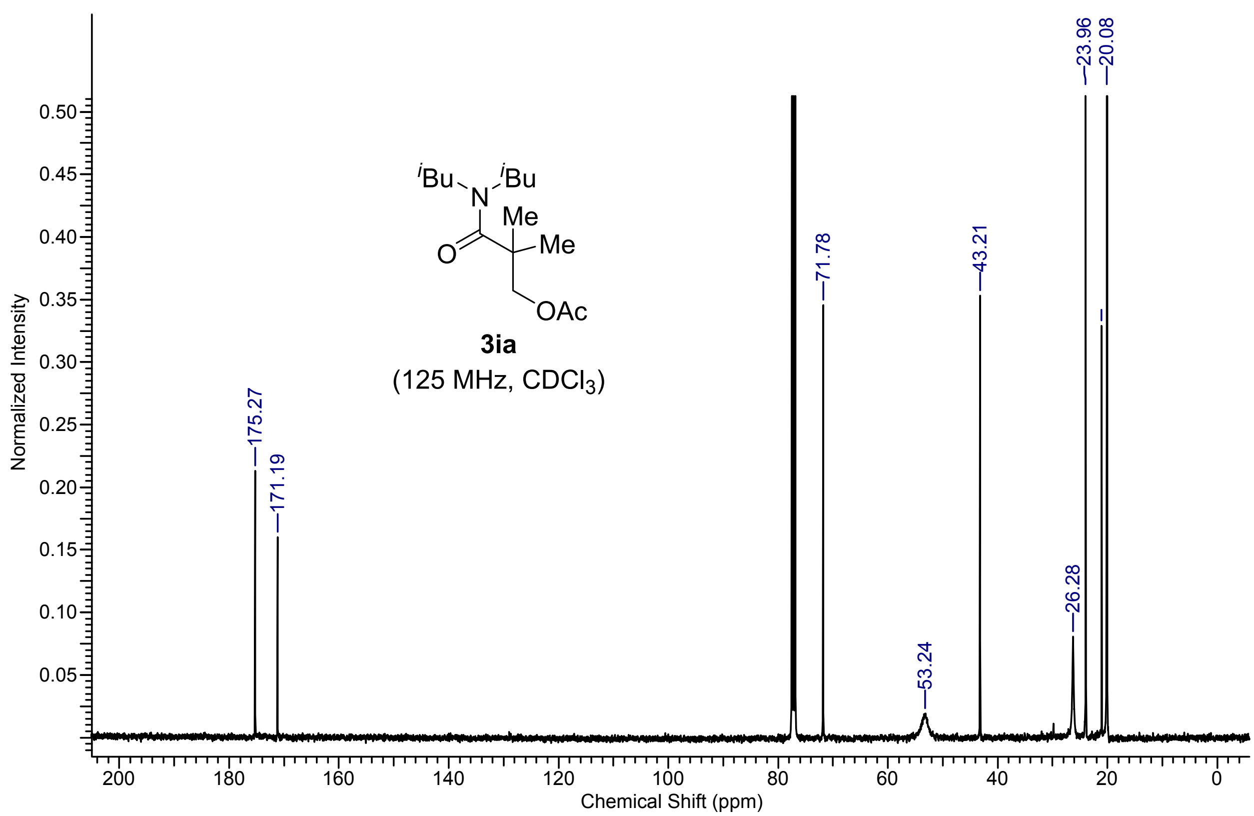
4.16
3.30
3.28
3.26
2.04
1.51
1.50
1.49
1.32
1.30
1.28
1.27
0.94
0.92
0.90
ⁿBu
N
ⁿBu
Me
Me
O
OAc
3ha
(400 MHz, CDCl3)
1.93
4.11
2.88
4.11
3.71
6.49
6.06
Normalized Intensity
Chemical Shift (ppm)



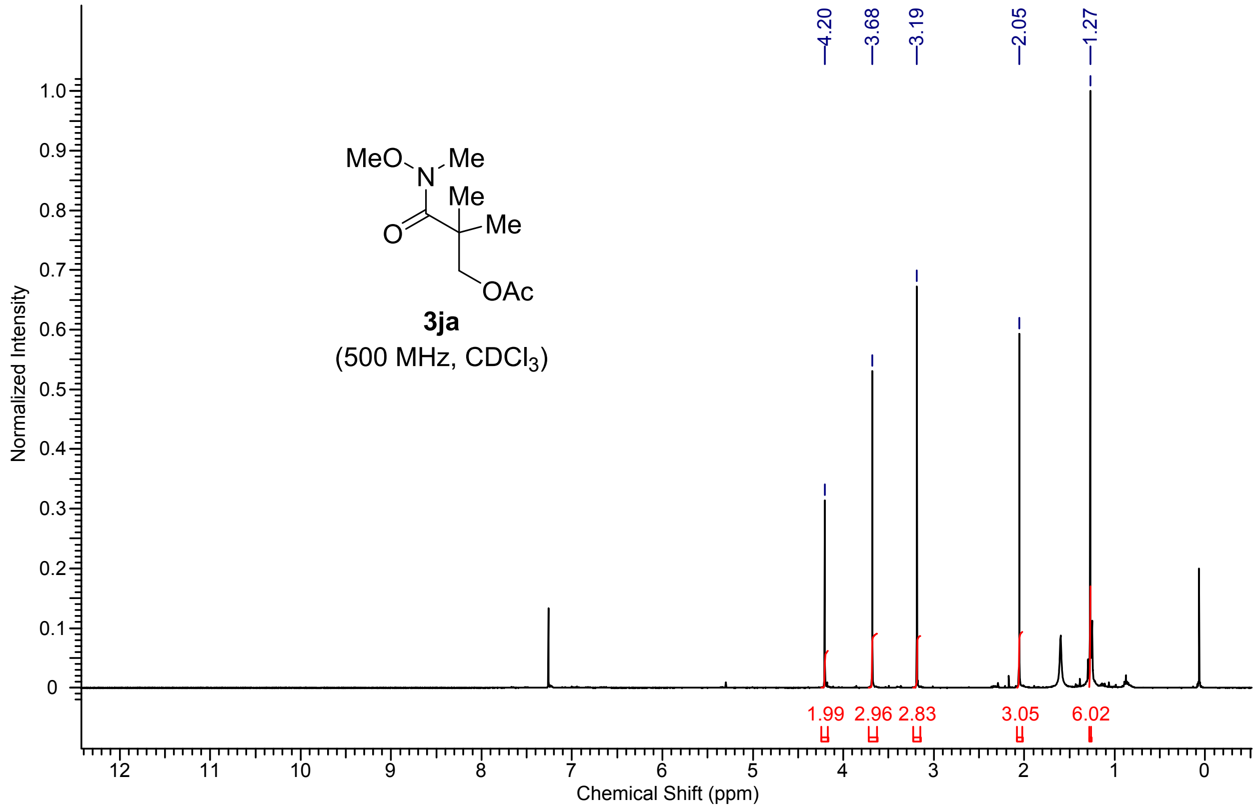
ⁿBu
N
ⁿBu
Me
Me
O
OAc
3ha
(100 MHz, CDCl3)
174.00
171.26
71.46
47.58
42.77
30.06
23.43
20.37
14.05
Normalized Intensity
Chemical Shift (ppm)



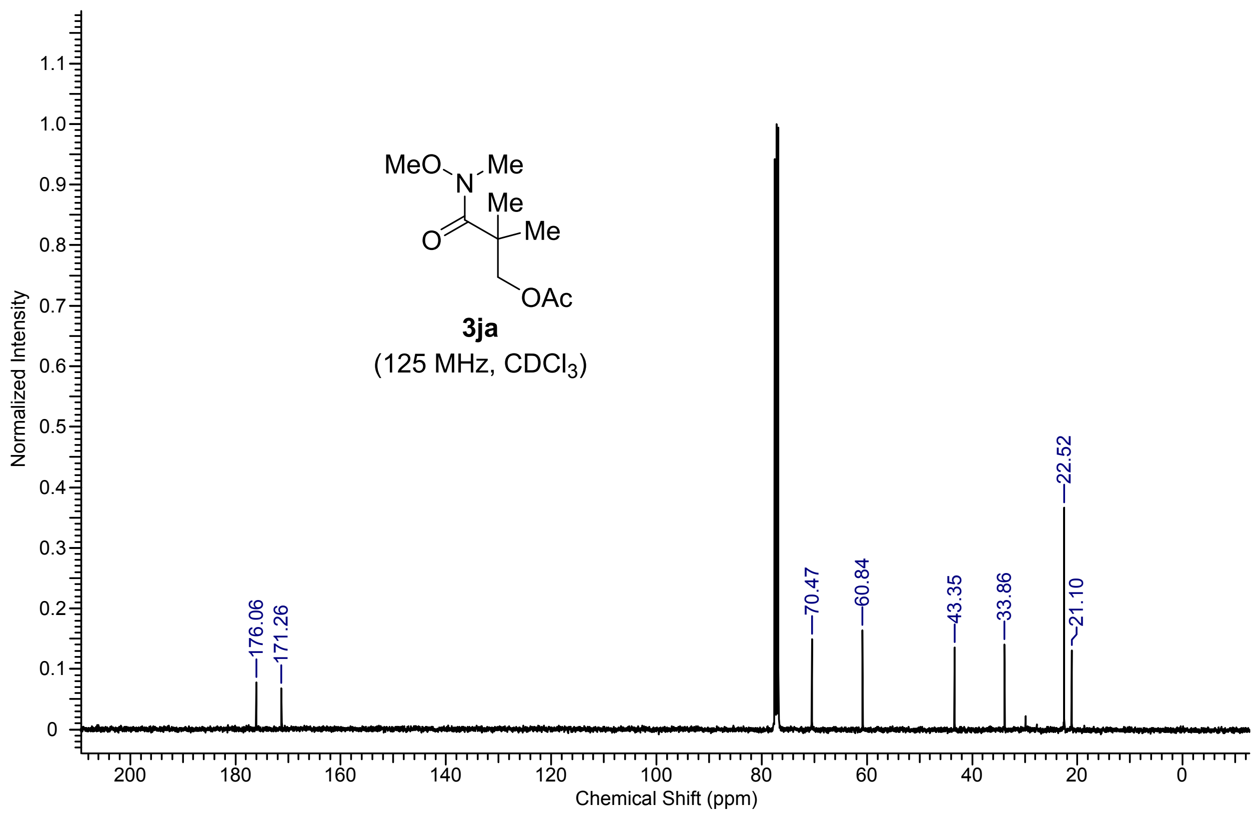
4.15
3.21
3.19
2.01
1.98
1.96
1.95
1.93
1.91
1.28
0.86
0.84
^{i}Bu
N
^{i}Bu
Me
Me
O
OAc
3ia
(500 MHz, CDCl$_3$)
2.00
4.04
2.88
2.11
5.97
12.09
Normalized Intensity
Chemical Shift (ppm)



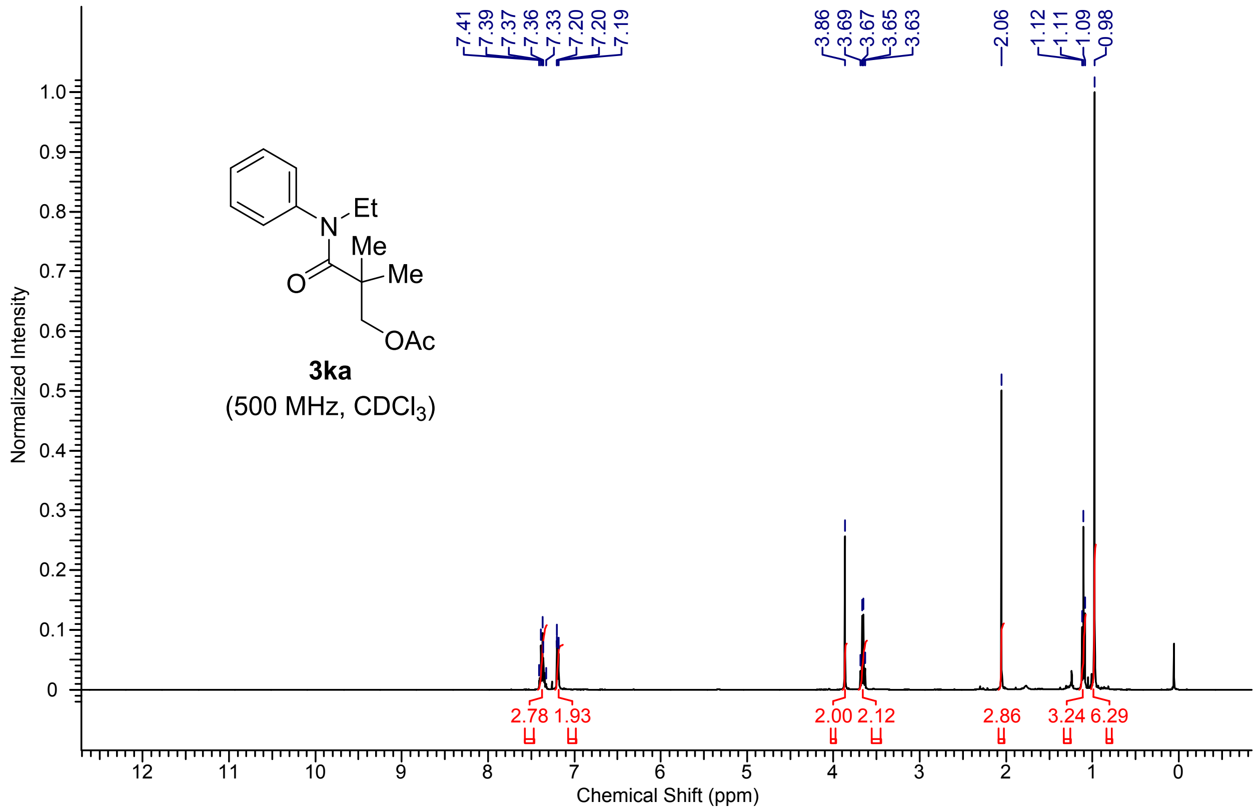
175.27
171.19
71.78
53.24
43.21
26.28
23.96
20.08
^{i}Bu
N
^{i}Bu
Me
Me
O
OAc
3ia
(125 MHz, CDCl$_3$)
Normalized Intensity
Chemical Shift (ppm)



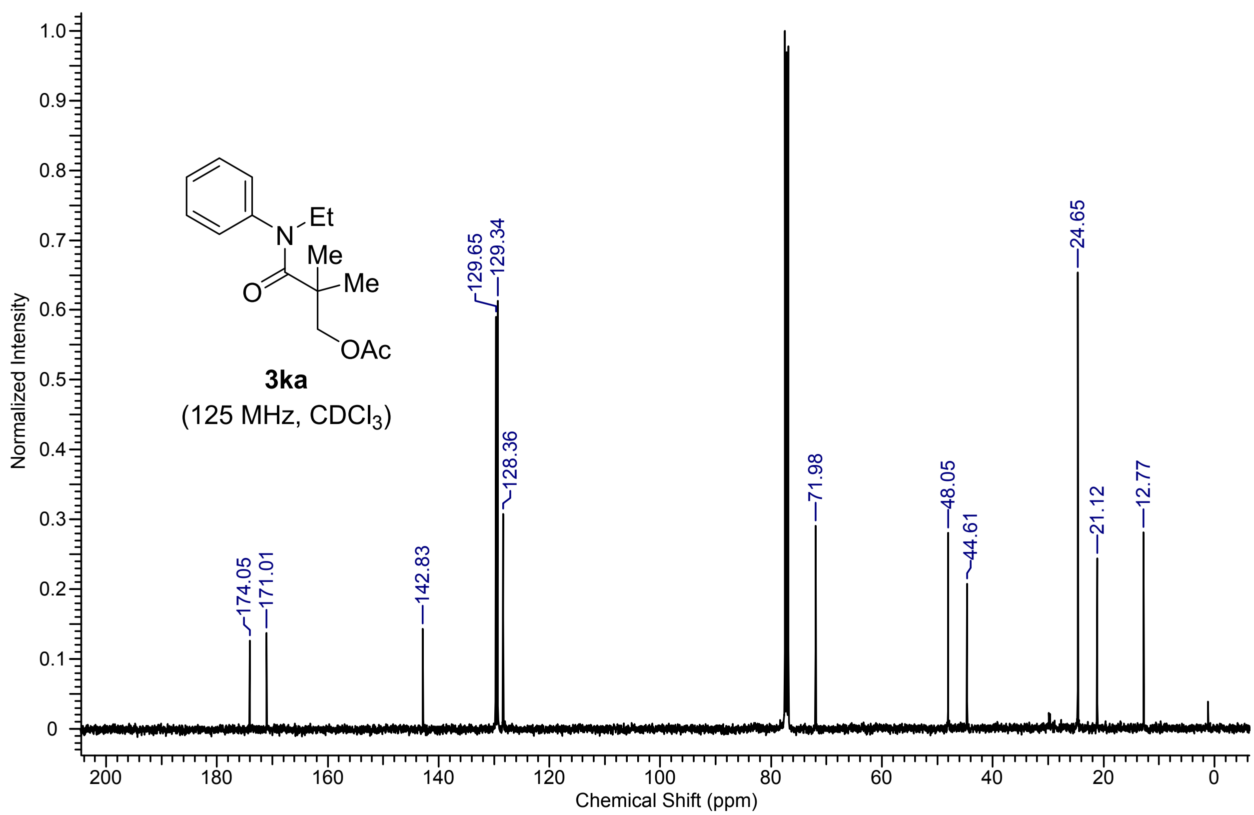
4.20
3.68
3.19
2.05
1.27
MeO
N
Me
Me
Me
O
OAc
3ja
(500 MHz, CDCl3)
1.99
2.96
2.83
3.05
6.02
Normalized Intensity
Chemical Shift (ppm)



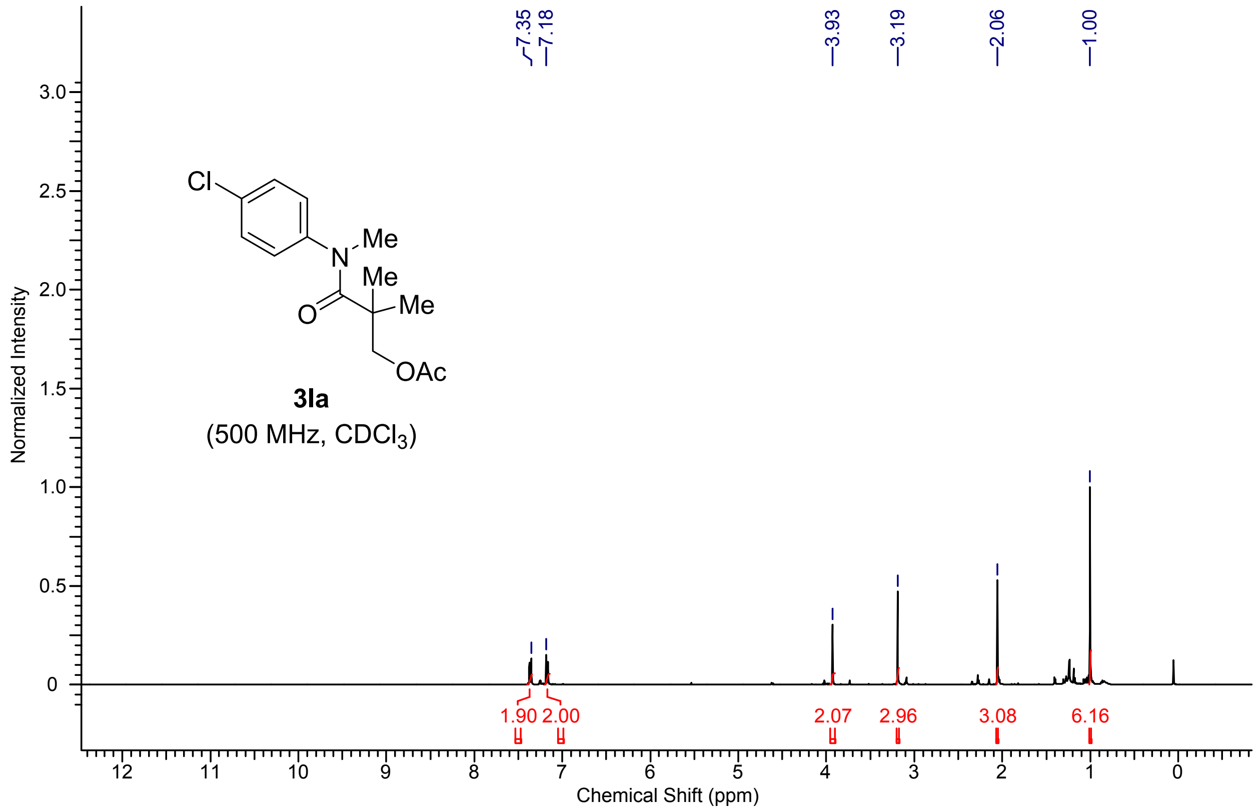
MeO
N
Me
Me
Me
O
OAc
3ja
(125 MHz, CDCl3)
176.06
171.26
70.47
60.84
43.35
33.86
22.52
21.10
Normalized Intensity
Chemical Shift (ppm)



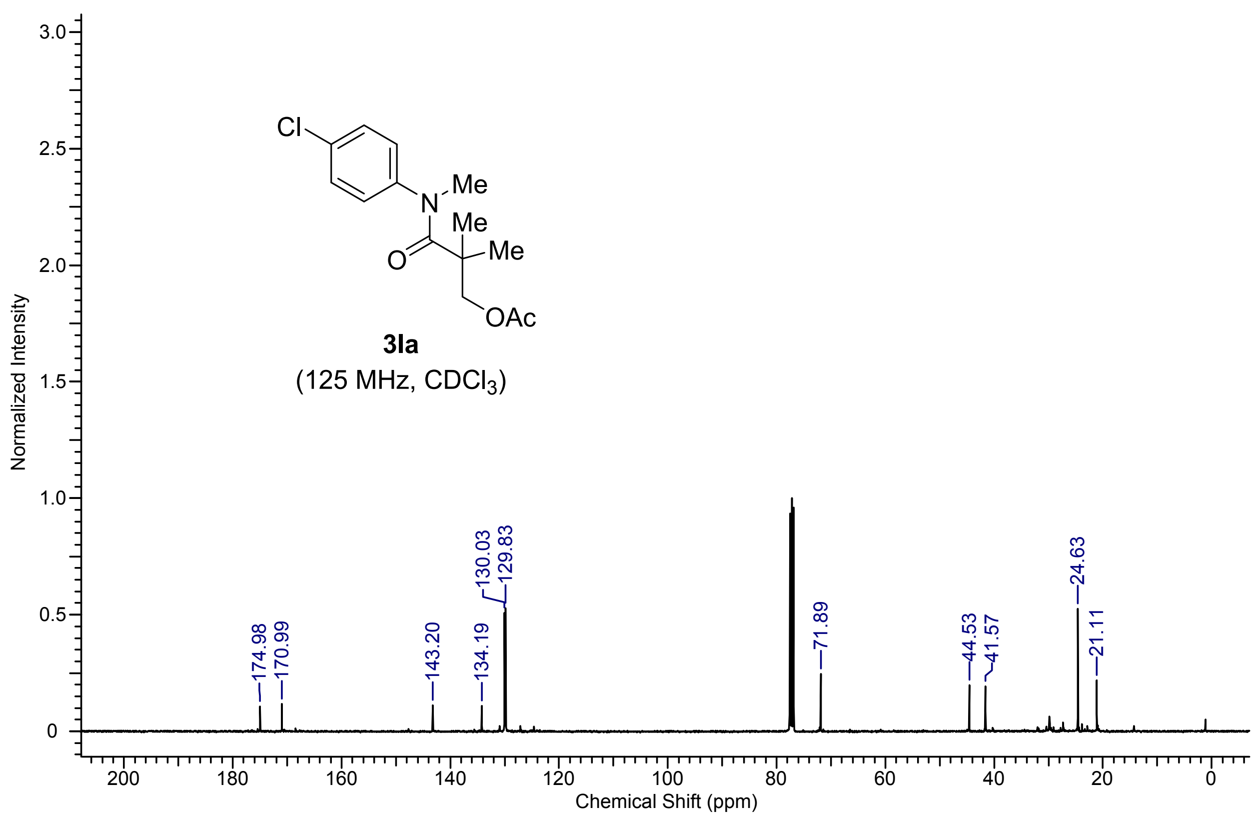
7.41
7.39
7.37
7.36
7.33
7.20
7.20
7.19
3.86
3.69
3.67
3.65
3.63
2.06
1.12
1.11
1.09
0.98
N
Et
Me
Me
O
OAc
3ka
(500 MHz, $CDCl_3$)
2.78
1.93
2.00
2.12
2.86
3.24
6.29
Normalized Intensity
Chemical Shift (ppm)



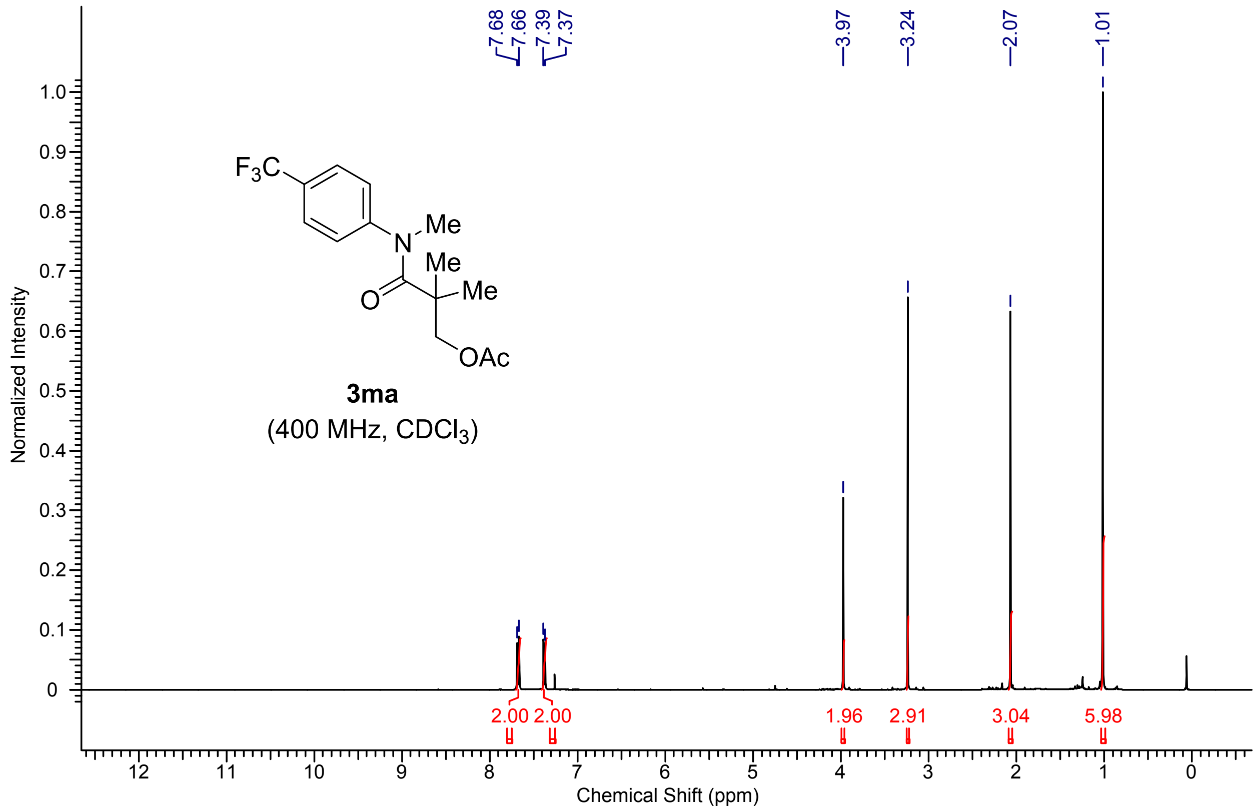
N
Et
Me
Me
O
OAc
3ka
(125 MHz, $CDCl_3$)
174.05
171.01
142.83
129.65
129.34
128.36
71.98
48.05
44.61
24.65
21.12
12.77
Normalized Intensity
Chemical Shift (ppm)

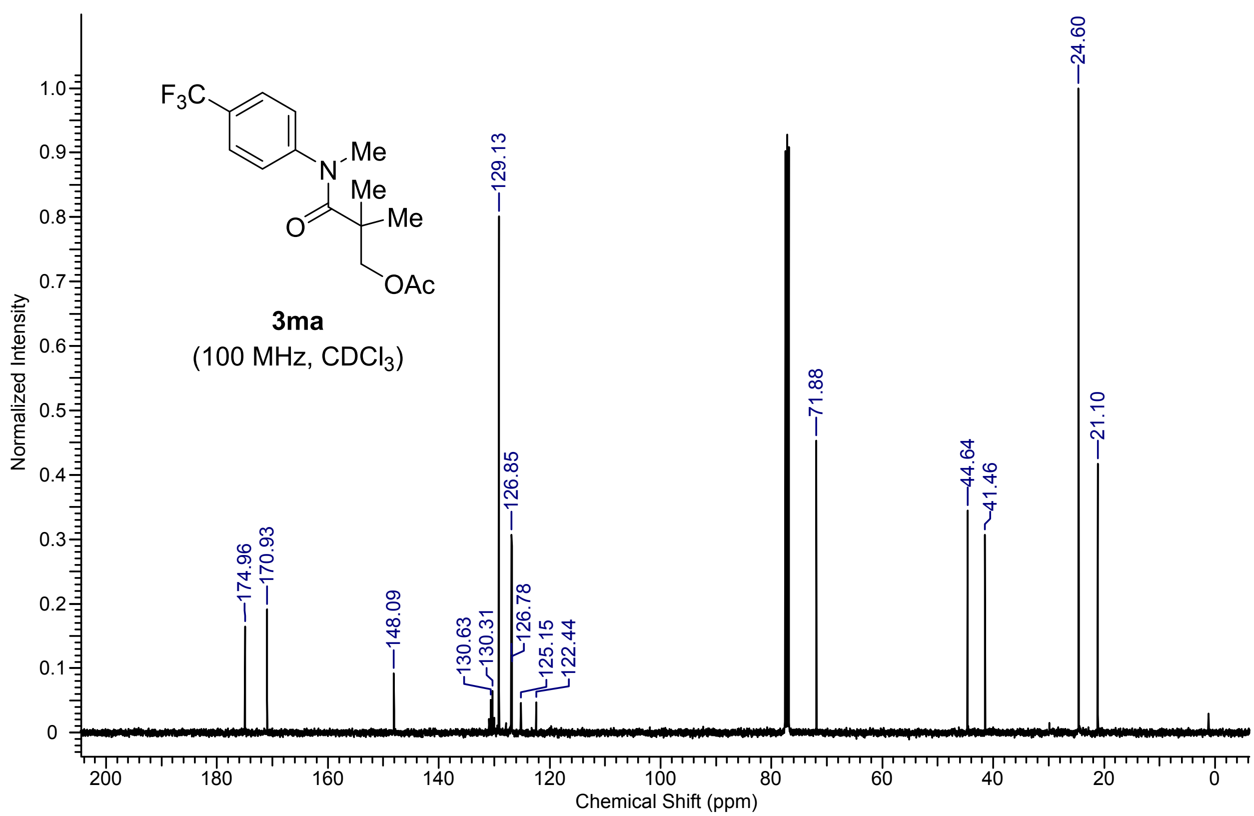


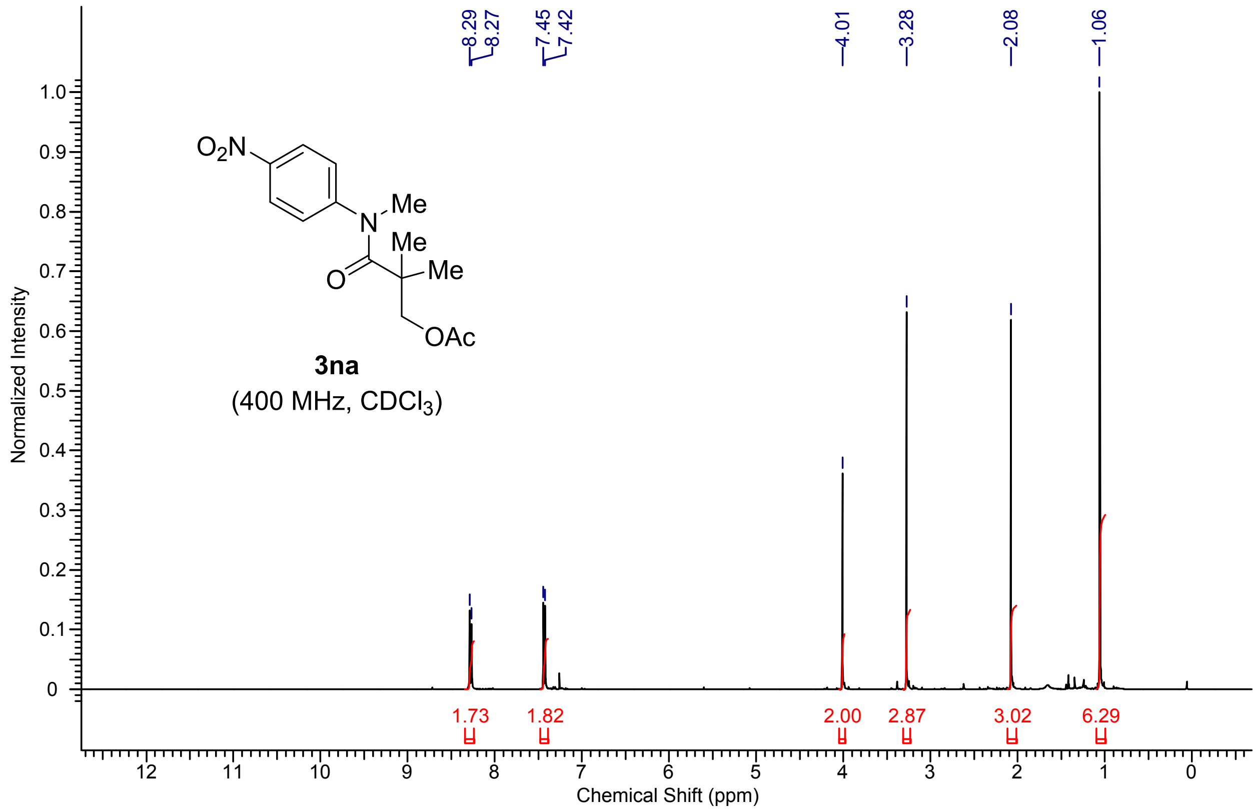
7.35
7.18
3.93
3.19
2.06
1.00
Cl
N
Me
Me
Me
O
OAc
3la
(500 MHz, CDCl3)
Normalized Intensity
1.90
2.00
2.07
2.96
3.08
6.16
Chemical Shift (ppm)



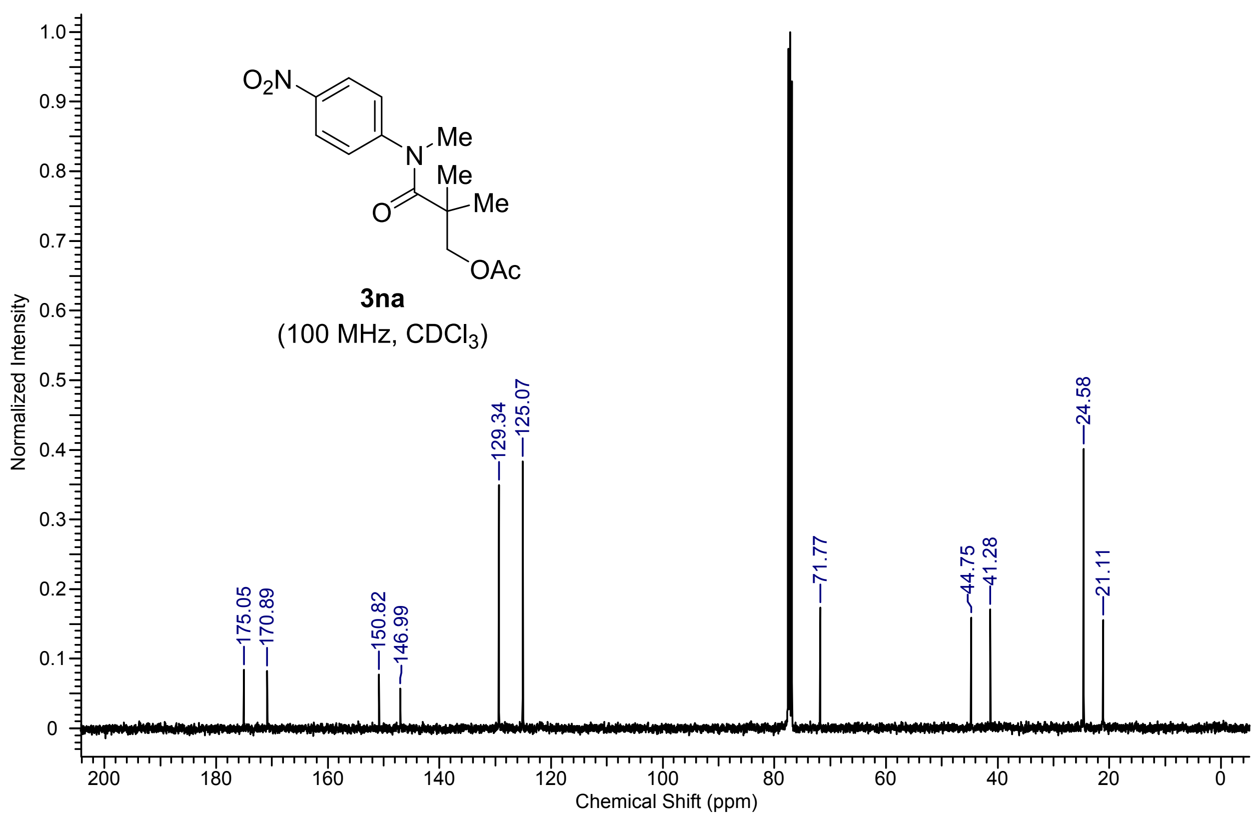
Cl
N
Me
Me
Me
O
OAc
3la
(125 MHz, CDCl3)
Normalized Intensity
174.98
170.99
143.20
134.19
130.03
129.83
71.89
44.53
41.57
24.63
21.11
Chemical Shift (ppm)


7.68
7.66
7.39
7.37
3.97
3.24
2.07
1.01
F3C
N
Me
Me
Me
O
OAc
3ma
(400 MHz, CDCl3)
Normalized Intensity
2.00
2.00
1.96
2.91
3.04
5.98
Chemical Shift (ppm)

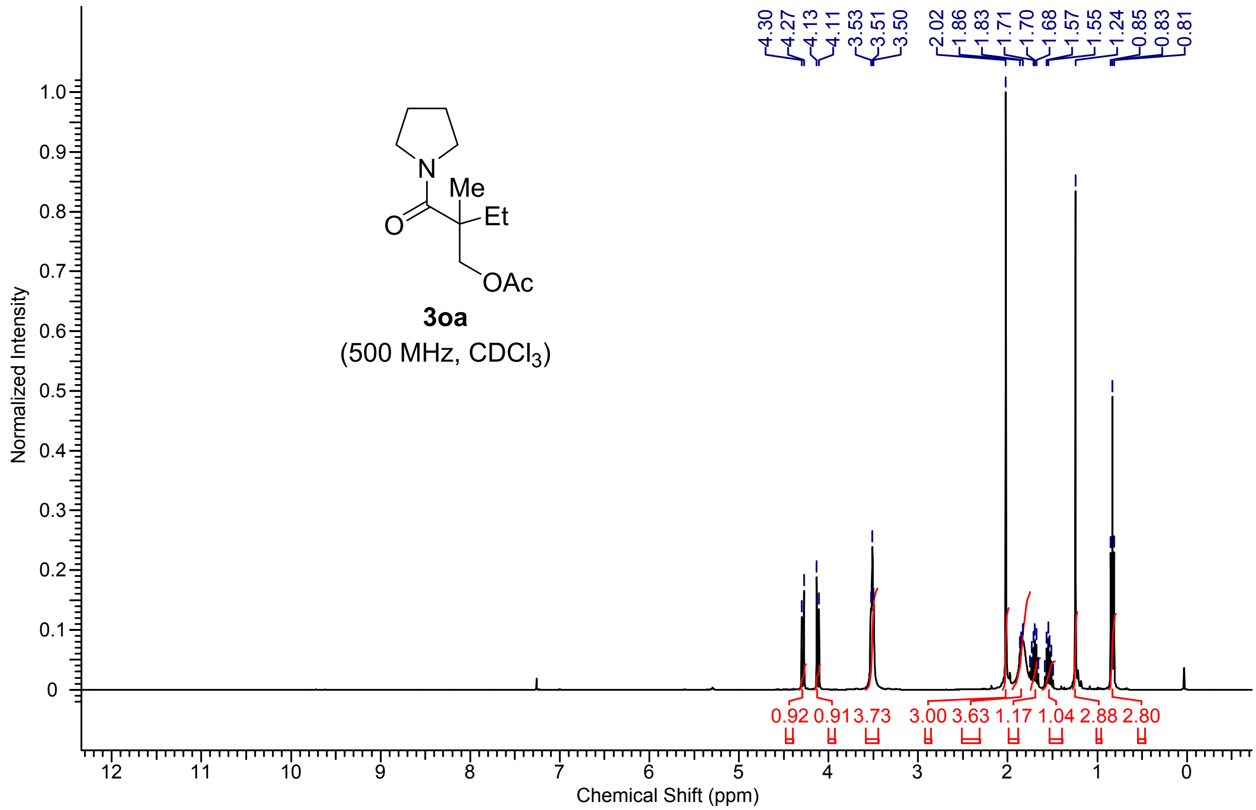

24.60
129.13
71.88
21.10
126.85
44.64
41.46
174.96
170.93
148.09
130.63
130.31
126.78
125.15
122.44
F3C
N
Me
Me
Me
O
OAc
3ma
(100 MHz, CDCl3)
Normalized Intensity
Chemical Shift (ppm)

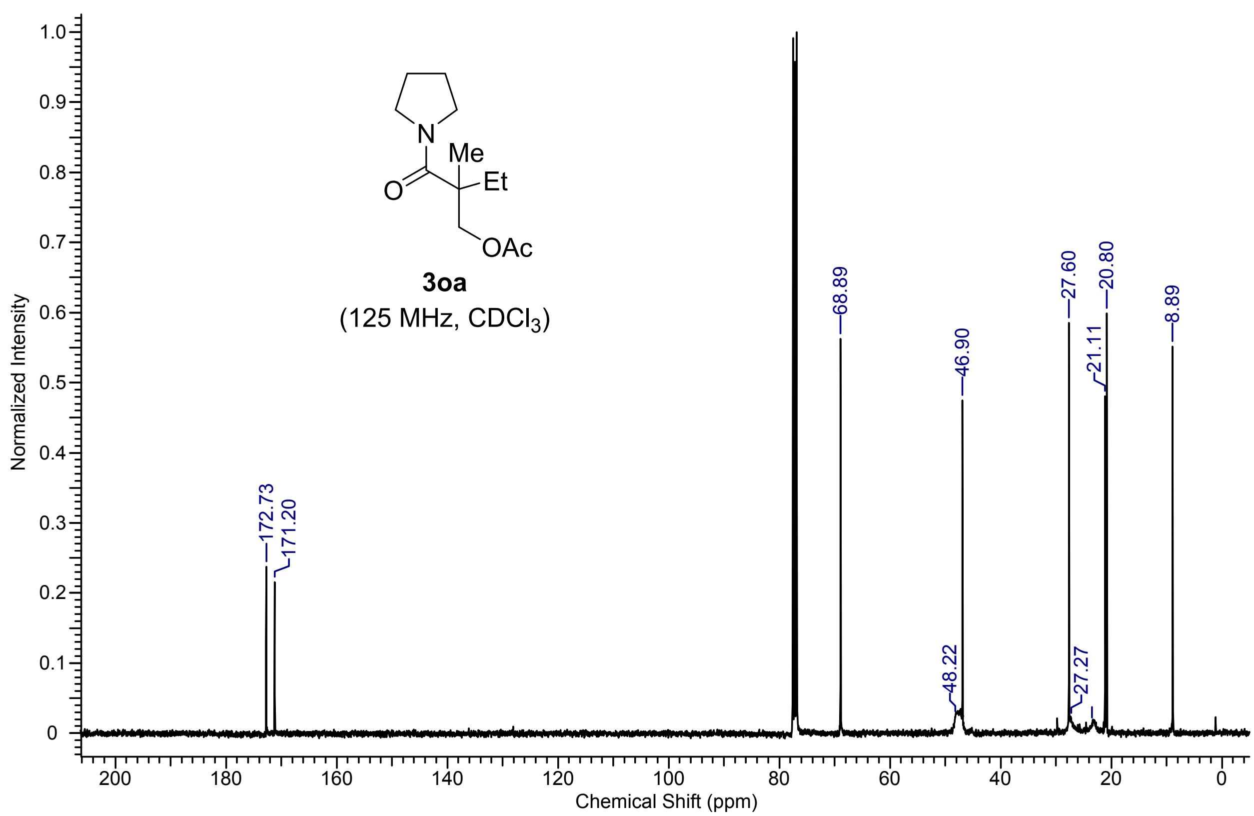


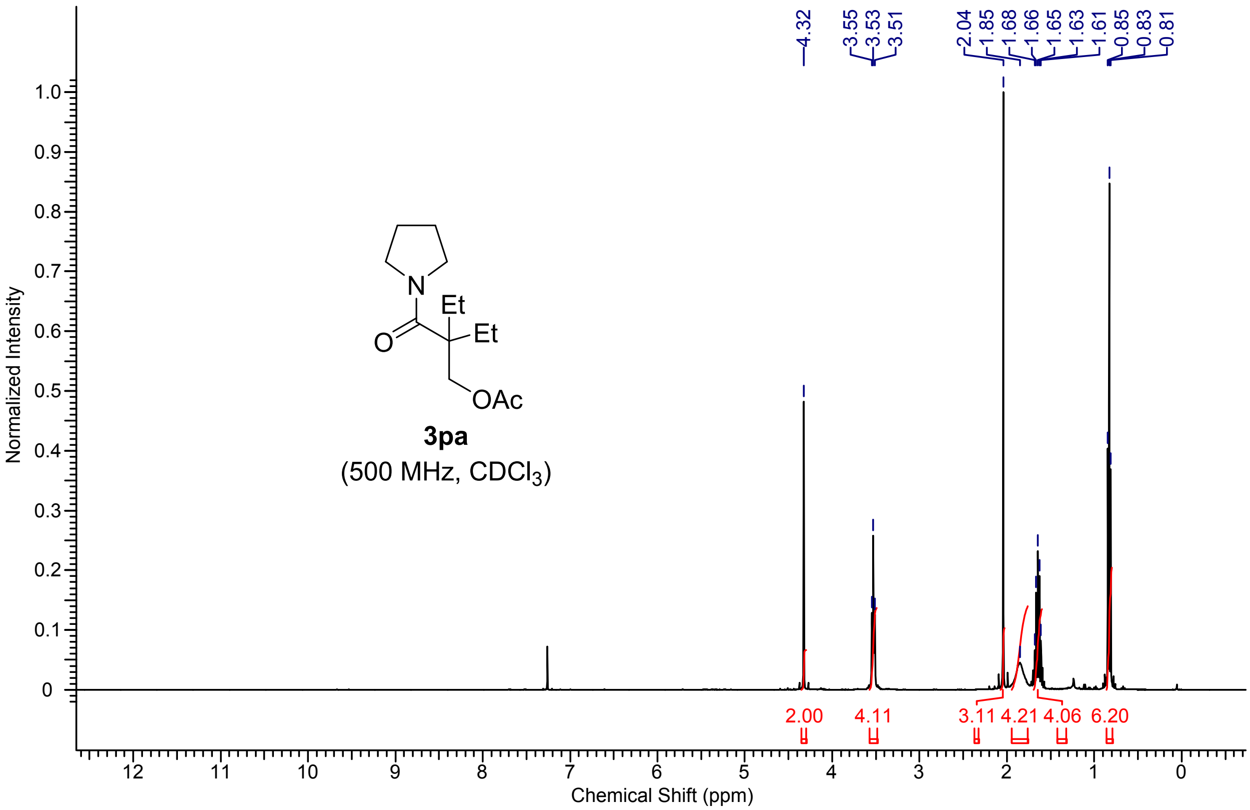
8.29
8.27
7.45
7.42
4.01
3.28
2.08
1.06
O_2N
N
Me
Me
Me
O
OAc
3na
(400 MHz, $CDCl_3$)
1.73
1.82
2.00
2.87
3.02
6.29
Normalized Intensity
Chemical Shift (ppm)

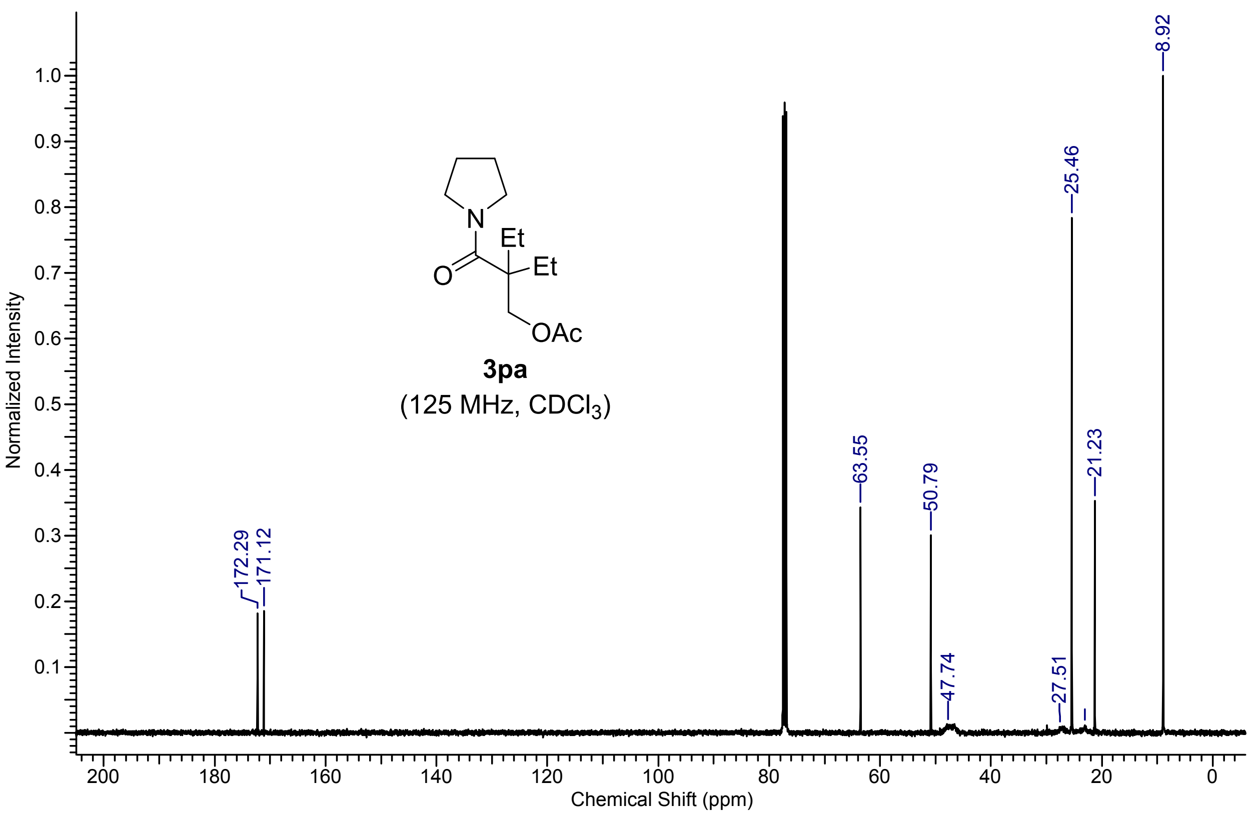


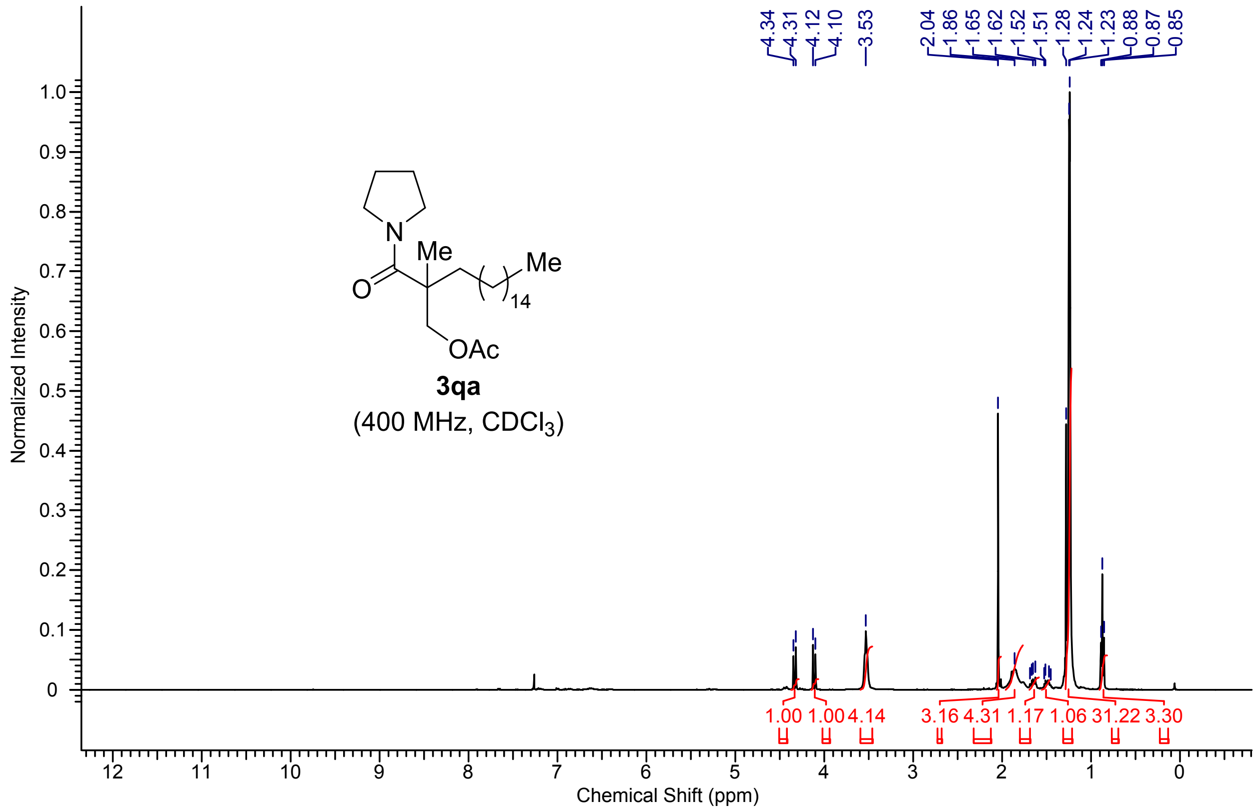
O_2N
N
Me
Me
Me
O
OAc
3na
(100 MHz, $CDCl_3$)
175.05
170.89
150.82
146.99
129.34
125.07
71.77
44.75
41.28
24.58
21.11
Normalized Intensity
Chemical Shift (ppm)

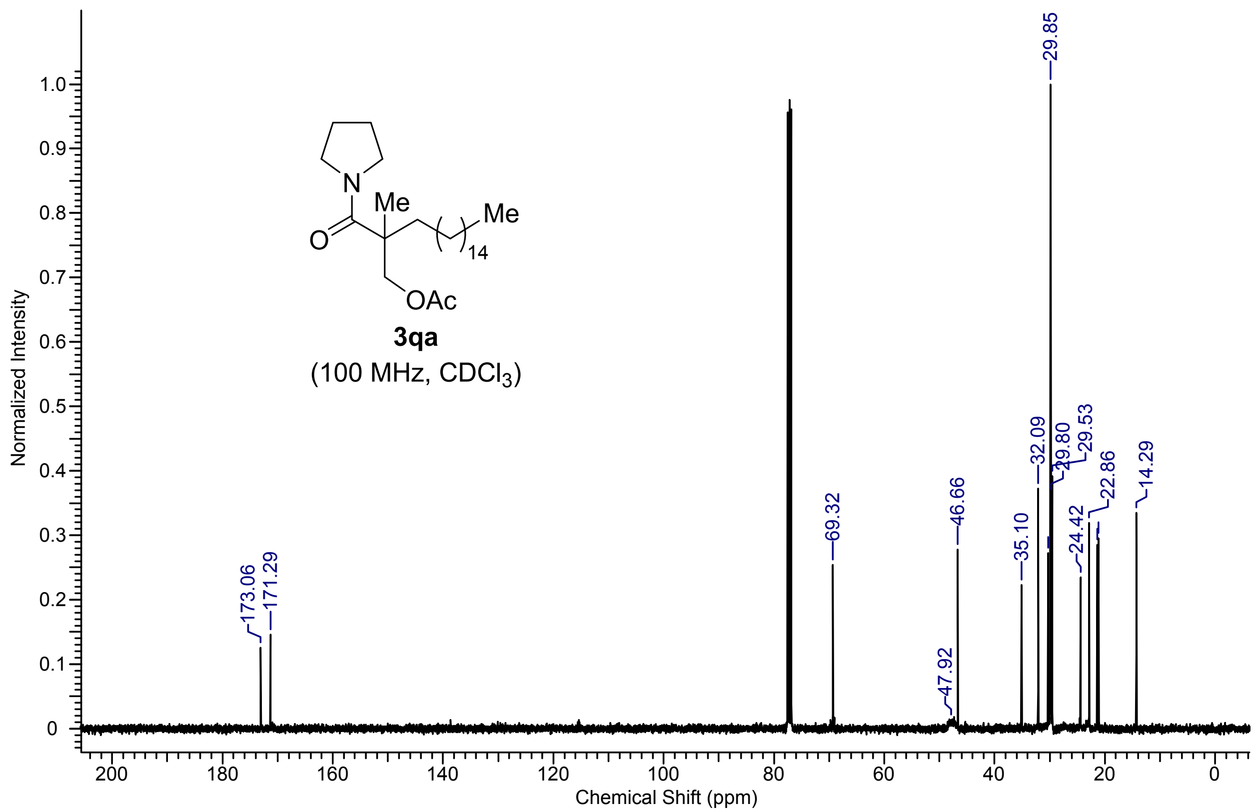

4.30 4.27 4.13 4.11 3.53 3.51 3.50 2.02 1.86 1.83 1.71 1.70 1.68 1.57 1.55 1.24 0.85 0.83 0.81
Me
Et
O
N
OAc
3oa
(500 MHz, $CDCl_3$)
0.92 0.91 3.73 3.00 3.63 1.17 1.04 2.88 2.80
Normalized Intensity
Chemical Shift (ppm)

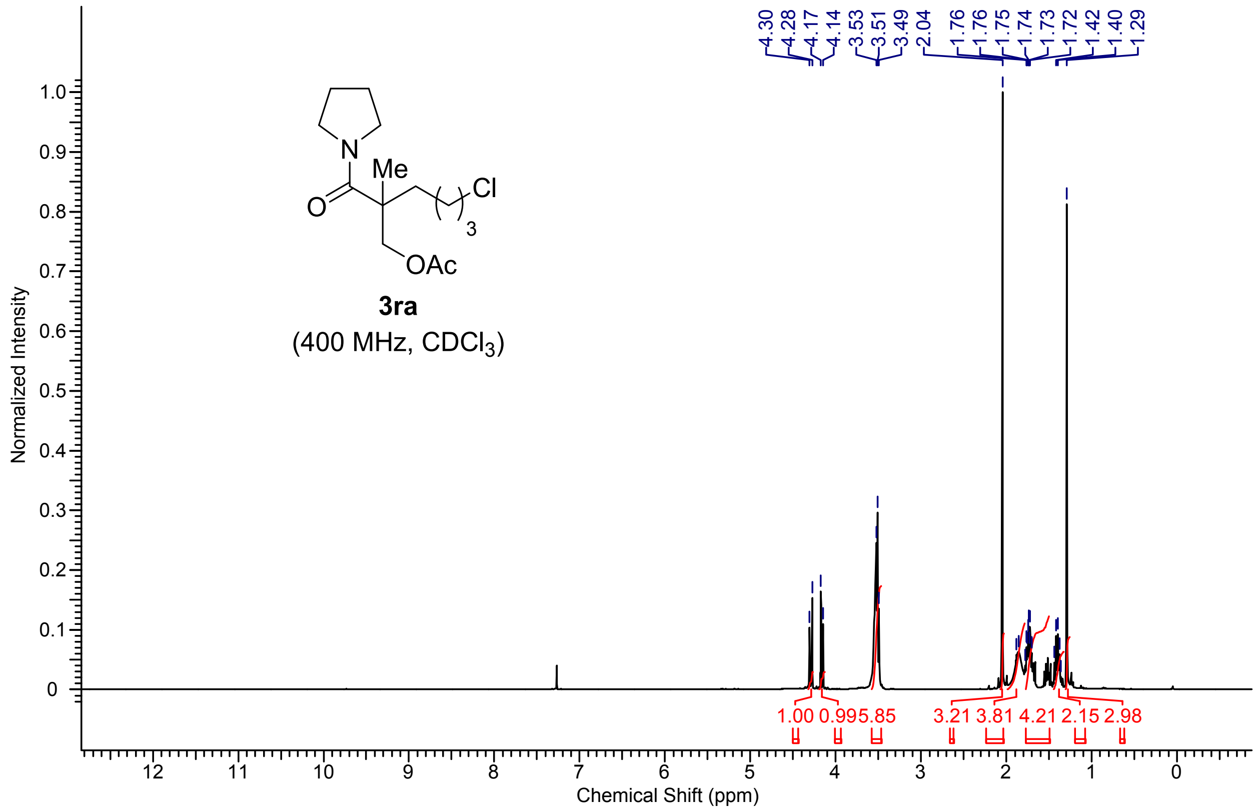

Me
Et
O
N
OAc
3oa
(125 MHz, $CDCl_3$)
172.73
171.20
68.89
48.22
46.90
27.60
27.27
21.11
20.80
8.89
Normalized Intensity
Chemical Shift (ppm)

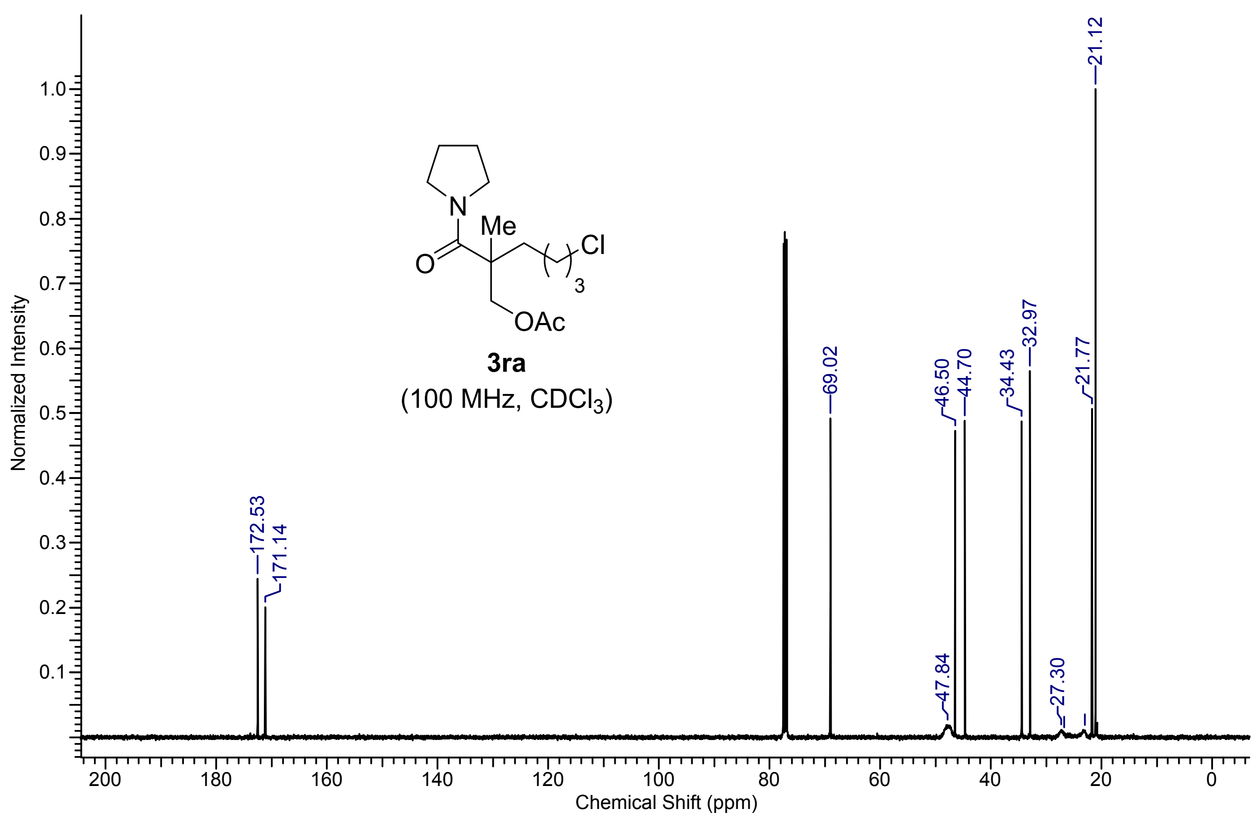

4.32
3.55
3.53
3.51
2.04
1.85
1.68
1.66
1.65
1.63
1.61
0.85
0.83
0.81
Et
Et
N
O
OAc
3pa
(500 MHz, CDCl3)
Normalized Intensity
2.00
4.11
3.11
4.21
4.06
6.20
Chemical Shift (ppm)

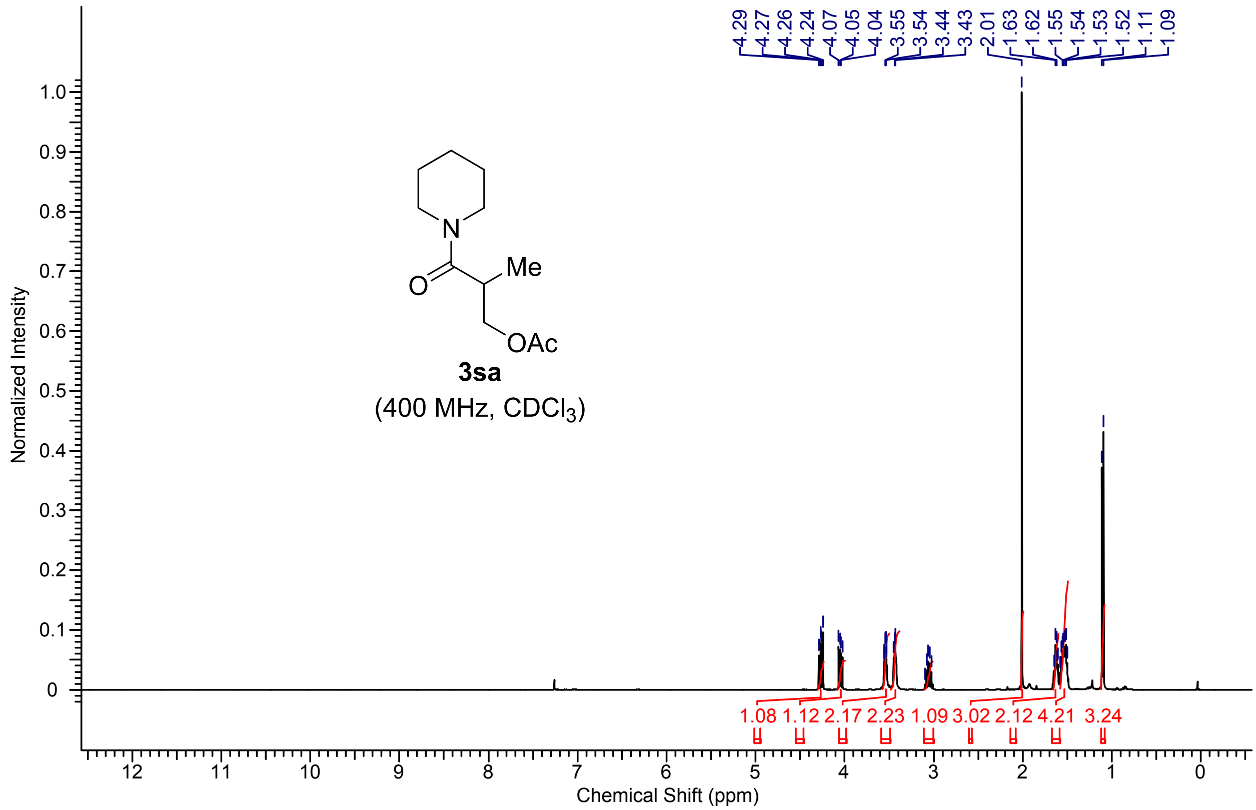

8.92
25.46
63.55
50.79
21.23
172.29
171.12
47.74
27.51
N
Et
Et
O
OAc
3pa
(125 MHz, CDCl3)
Normalized Intensity
Chemical Shift (ppm)

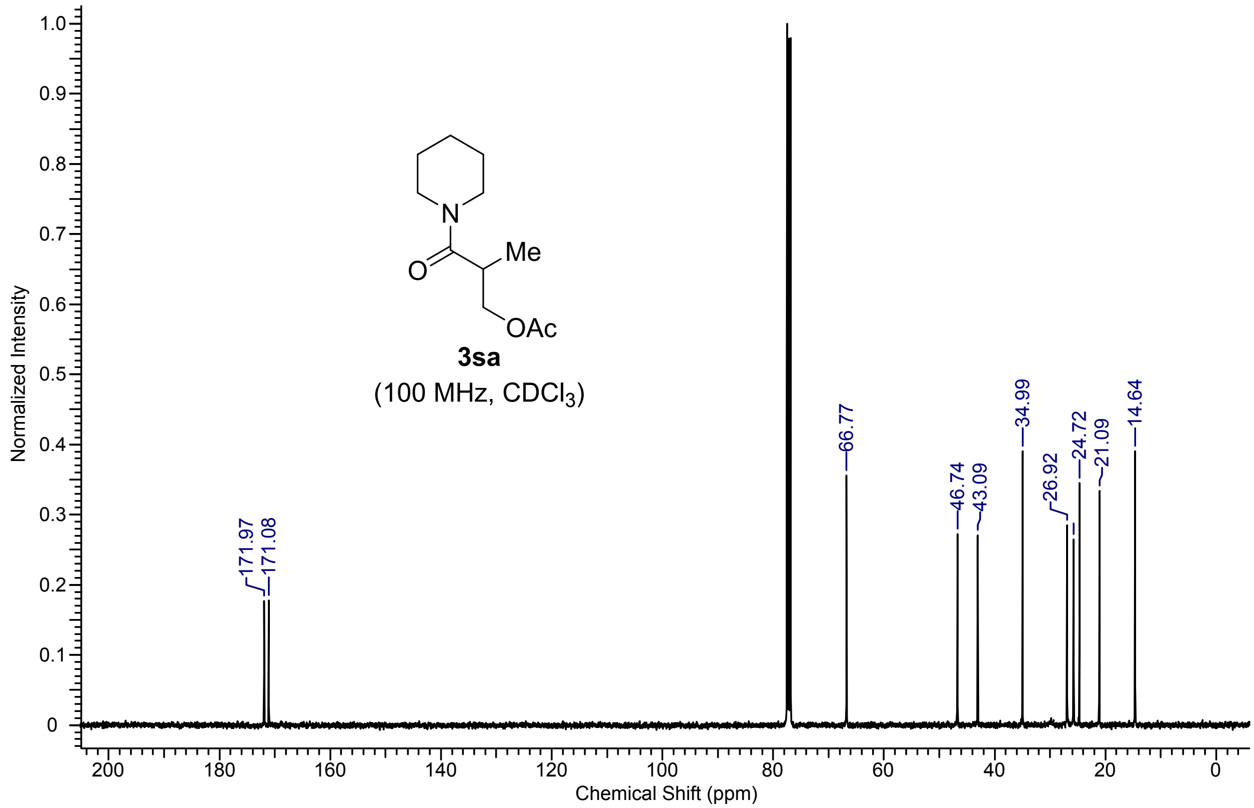

4.34
4.31
4.12
4.10
3.53
2.04
1.86
1.65
1.62
1.52
1.51
1.28
1.24
1.23
0.88
0.87
0.85
Me
N
O
Me
14
OAc
3qa
(400 MHz, CDCl3)
Normalized Intensity
1.00 1.00 4.14
3.16 4.31 1.17 1.06 31.22 3.30
Chemical Shift (ppm)

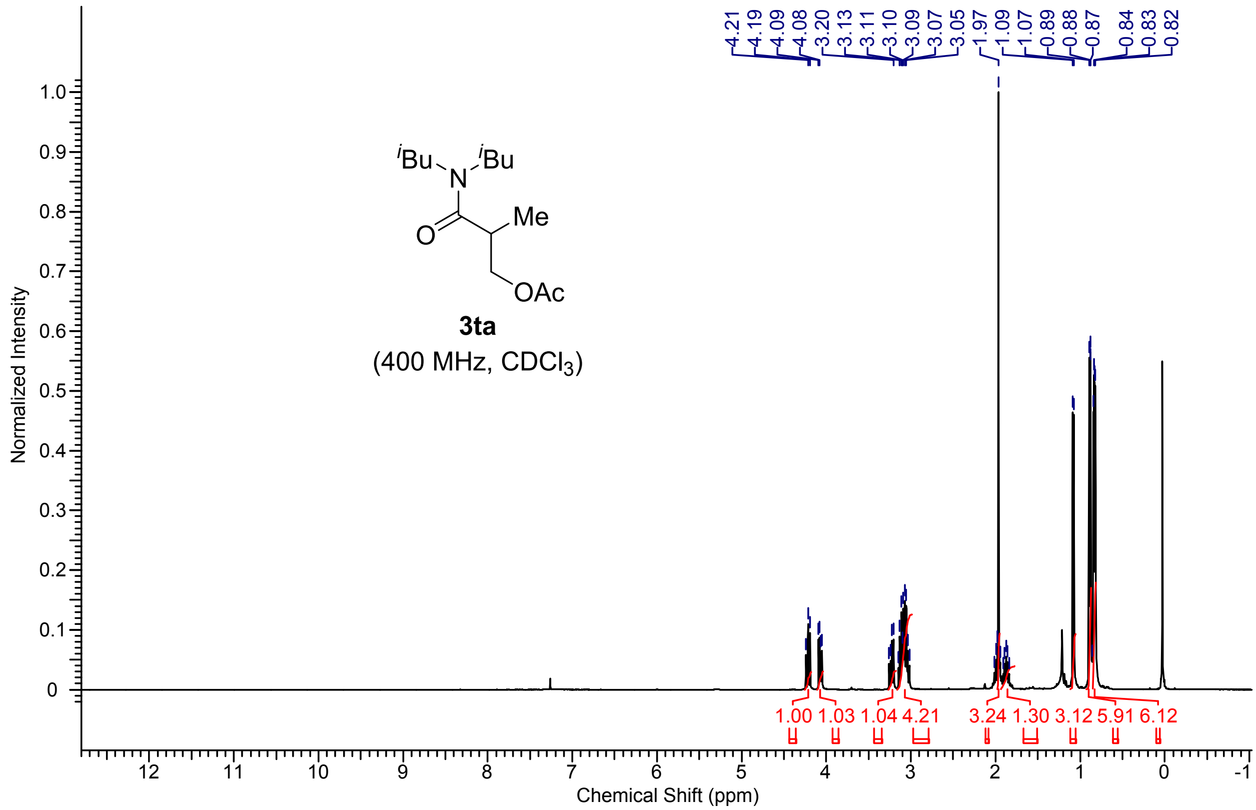

29.85
N
O
Me
Me
14
OAc
3qa
(100 MHz, CDCl3)
173.06
171.29
69.32
47.92
46.66
35.10
32.09
29.80
29.53
24.42
22.86
14.29
Normalized Intensity
Chemical Shift (ppm)


4.30
4.28
4.17
4.14
3.53
3.51
3.49
2.04
1.76
1.76
1.75
1.74
1.73
1.72
1.42
1.40
1.29
Me
Cl
3
N
O
OAc
3ra
(400 MHz, CDCl3)
Normalized Intensity
1.00
0.99
5.85
3.21
3.81
4.21
2.15
2.98
Chemical Shift (ppm)

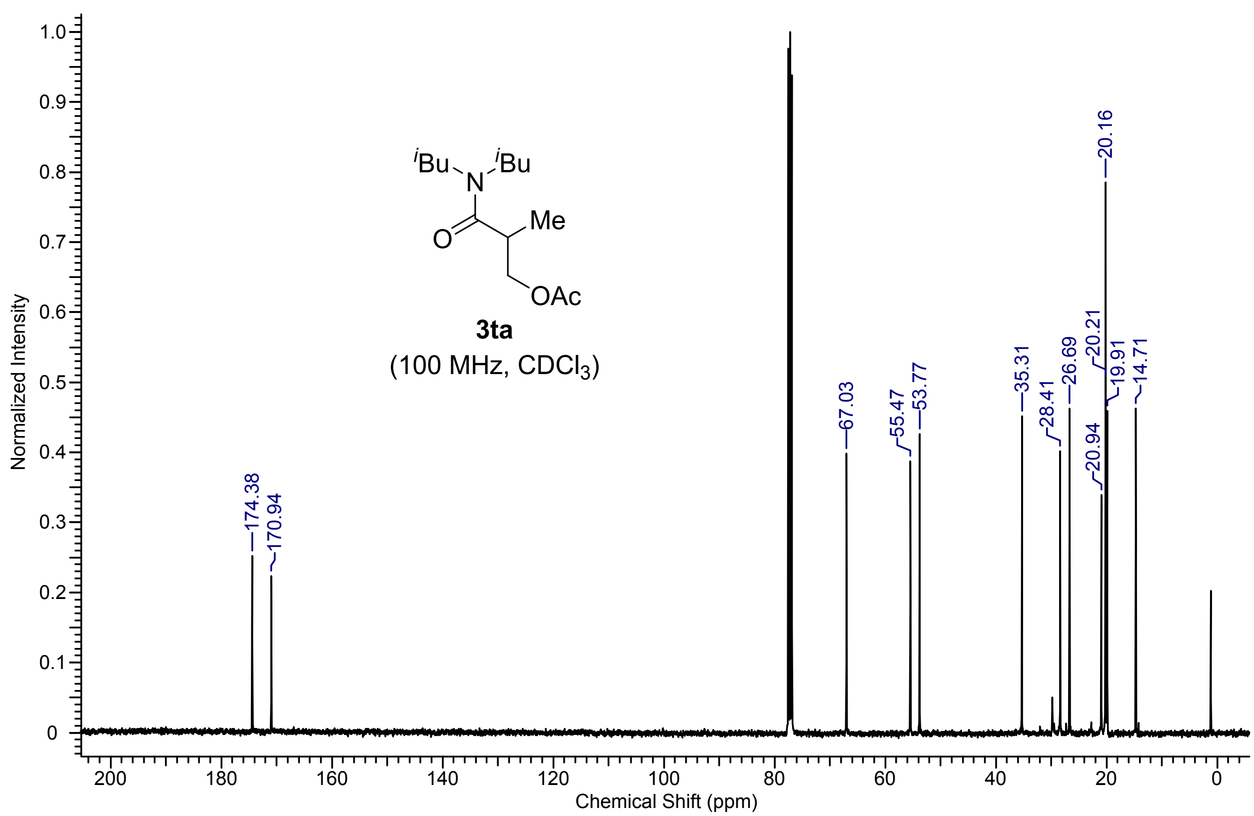

21.12
Me
Cl
3
N
O
OAc
3ra
(100 MHz, CDCl3)
69.02
46.50
44.70
34.43
32.97
21.77
172.53
171.14
47.84
27.30
Normalized Intensity
Chemical Shift (ppm)


4.29 4.27 4.26 4.24 4.07 4.05 4.04 3.55 3.54 3.44 3.43 2.01 1.63 1.62 1.55 1.54 1.53 1.52 1.11 1.09
N
O
Me
OAc
3sa
(400 MHz, $CDCl_3$)
1.08 1.12 2.17 2.23 1.09 3.02 2.12 4.21 3.24
Normalized Intensity
Chemical Shift (ppm)

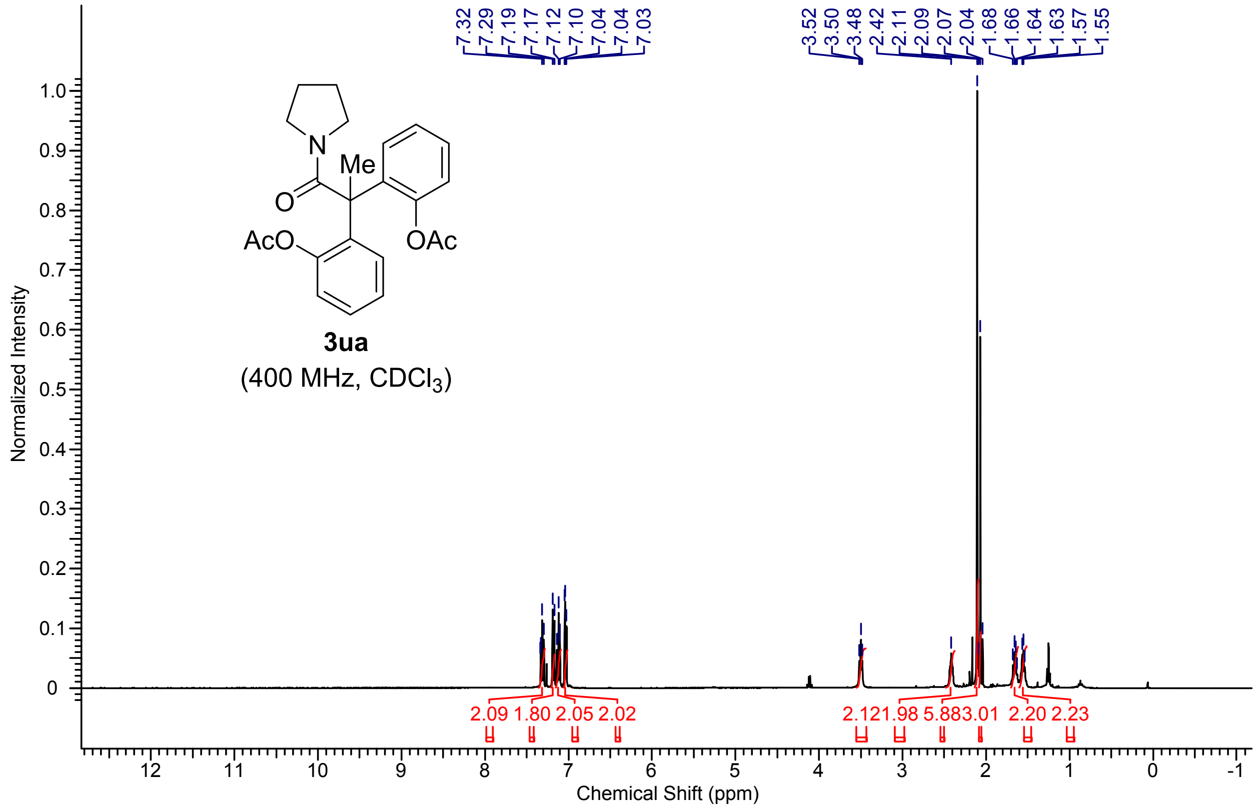

N
O
Me
OAc
3sa
(100 MHz, $CDCl_3$)
171.97 171.08
66.77
46.74 43.09
34.99
26.92 24.72 21.09
14.64
Normalized Intensity
Chemical Shift (ppm)

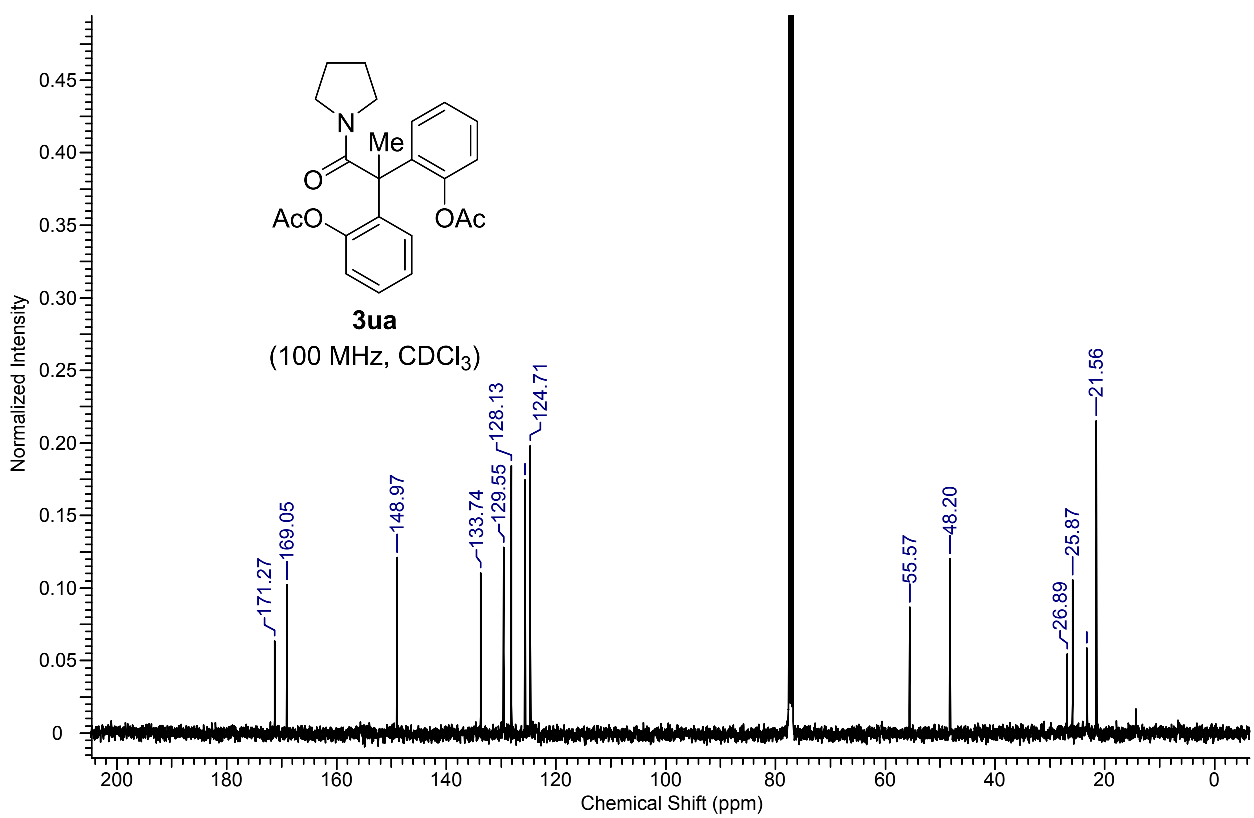


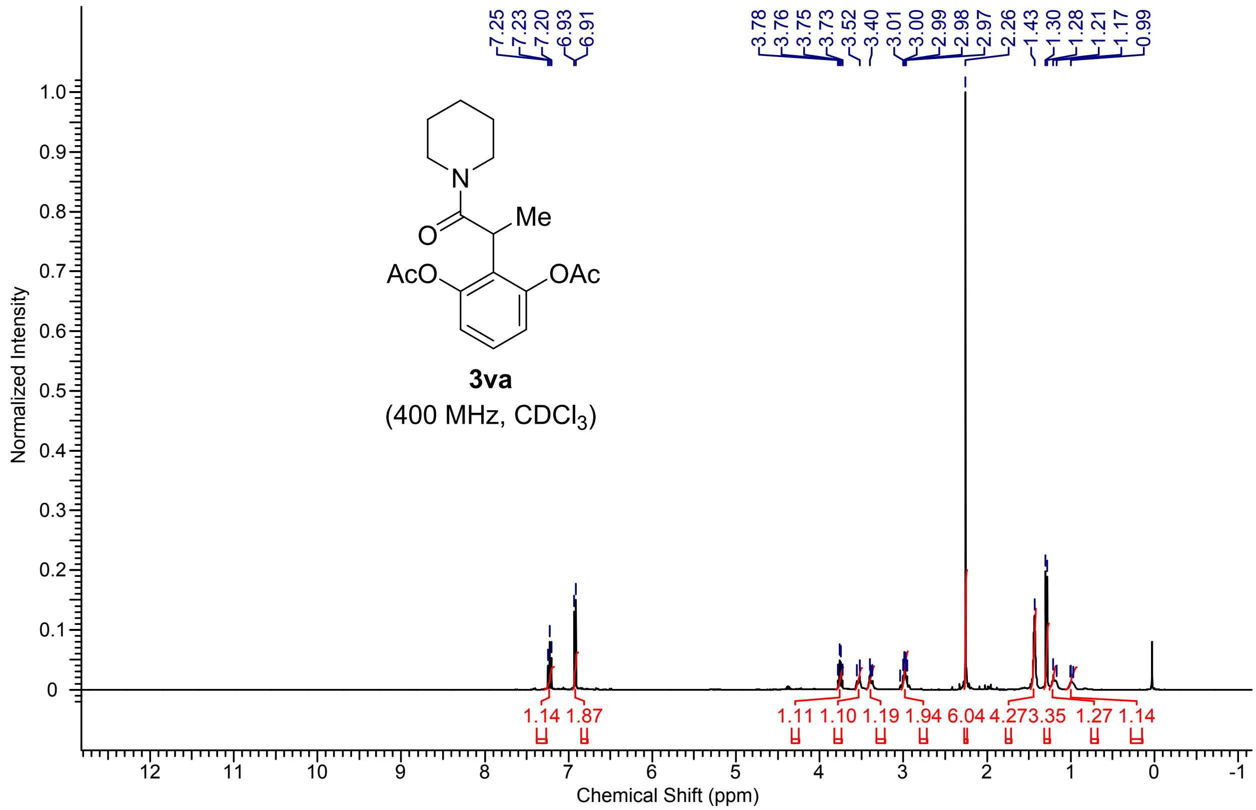
4.21
4.19
4.09
4.08
3.20
3.13
3.11
3.10
3.09
3.07
3.05
1.97
1.09
1.07
0.89
0.88
0.87
0.84
0.83
0.82
iBu
N
iBu
Me
O
OAc
3ta
(400 MHz, CDCl3)
Normalized Intensity
1.00 1.03 1.04 4.21 3.24 1.30 3.12 5.91 6.12
Chemical Shift (ppm)

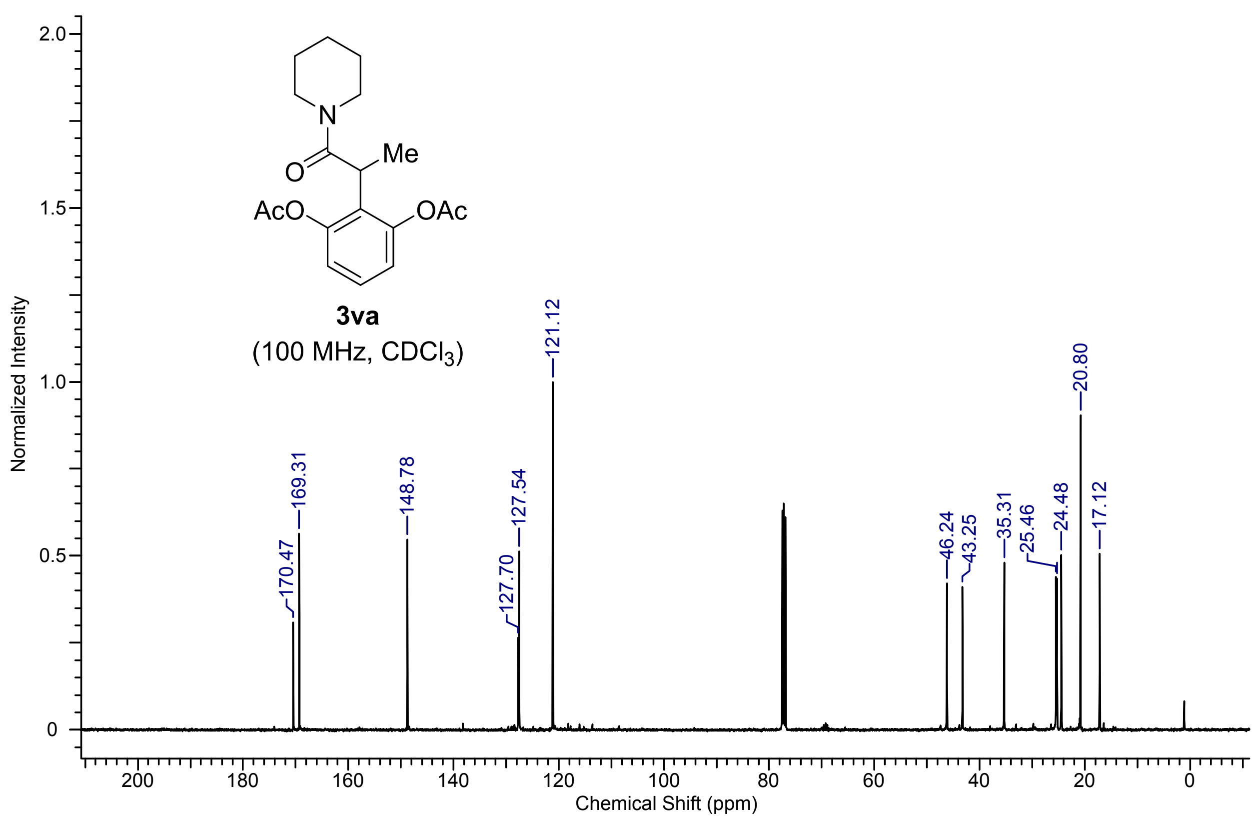


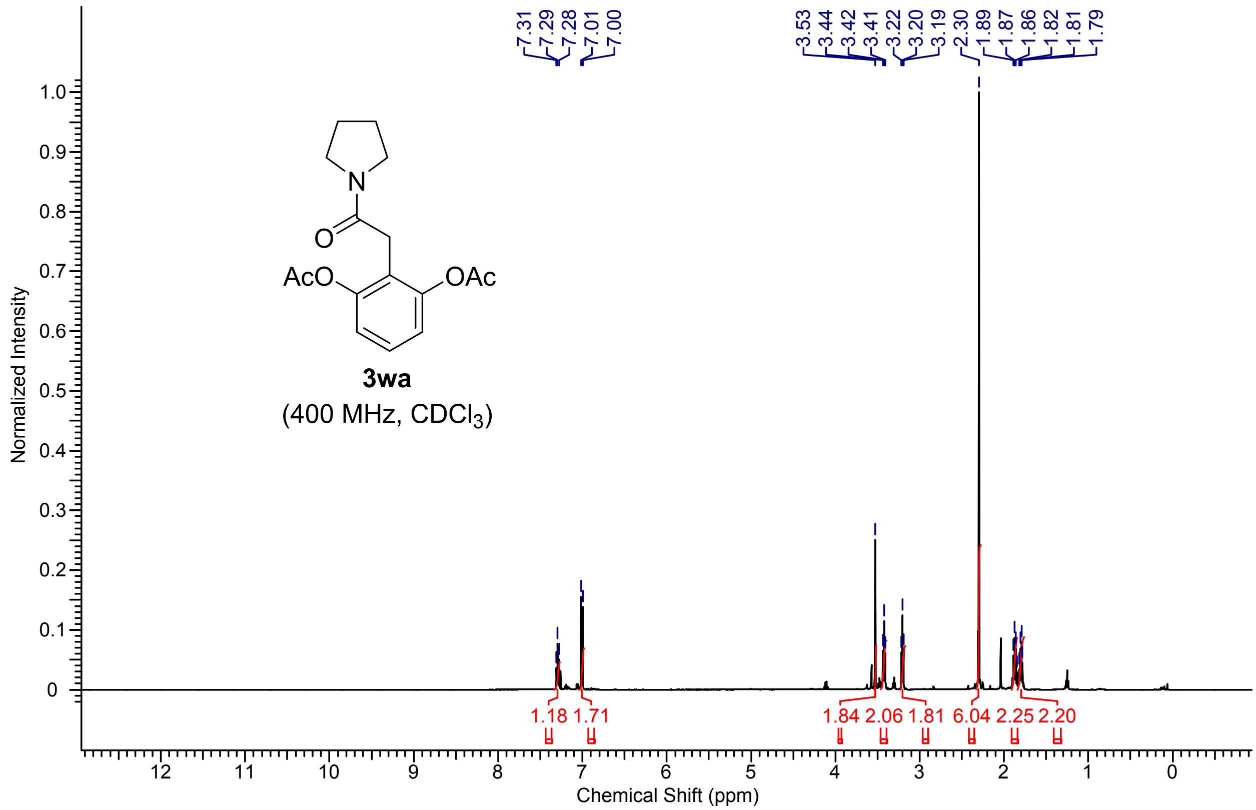
iBu
N
iBu
Me
O
OAc
3ta
(100 MHz, CDCl3)
174.38
170.94
67.03
55.47
53.77
35.31
28.41
26.69
20.94
20.21
20.16
19.91
14.71
Normalized Intensity
Chemical Shift (ppm)

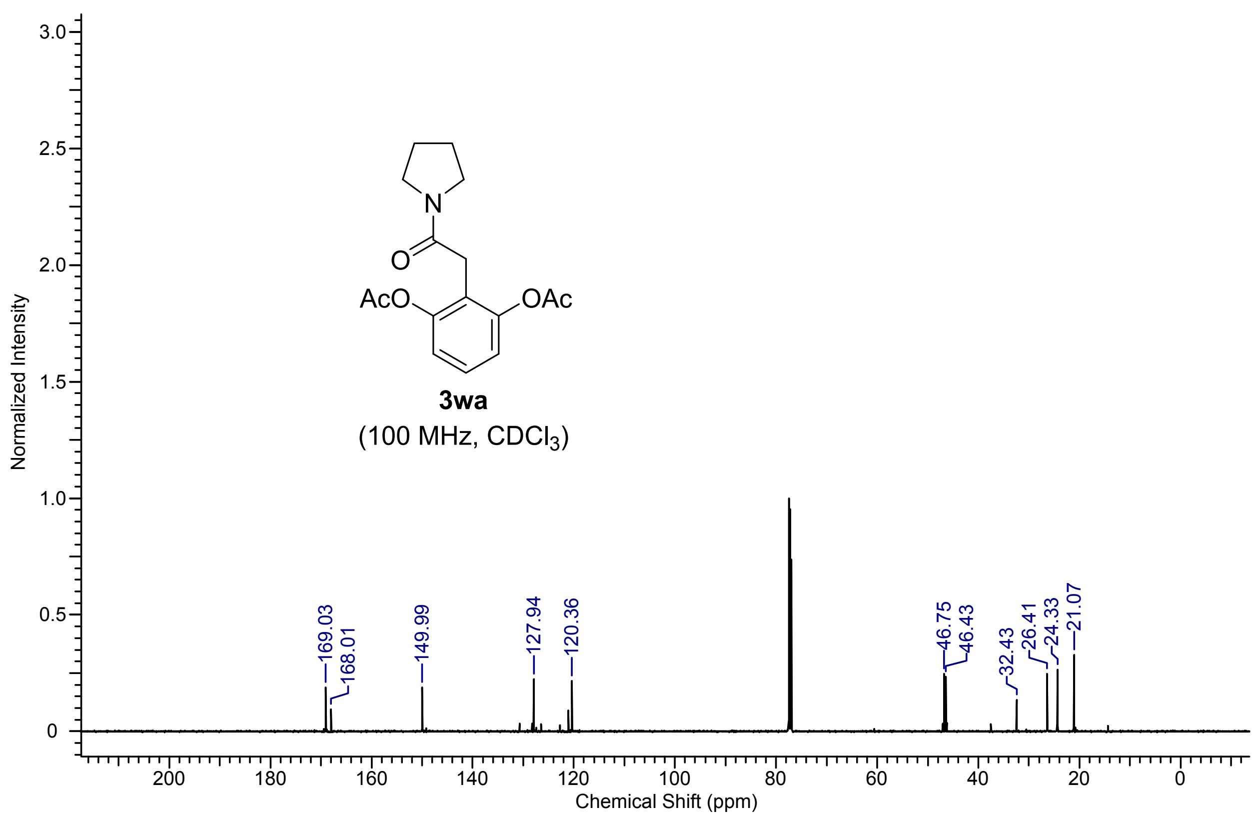

7.32
7.29
7.19
7.17
7.12
7.10
7.04
7.04
7.03
3.52
3.50
3.48
2.42
2.11
2.09
2.07
2.04
1.68
1.66
1.64
1.63
1.57
1.55
Me
N
O
AcO
OAc
3ua
(400 MHz, CDCl3)
Normalized Intensity
2.09
1.80
2.05
2.02
2.12
1.98
5.88
3.01
2.20
2.23
Chemical Shift (ppm)

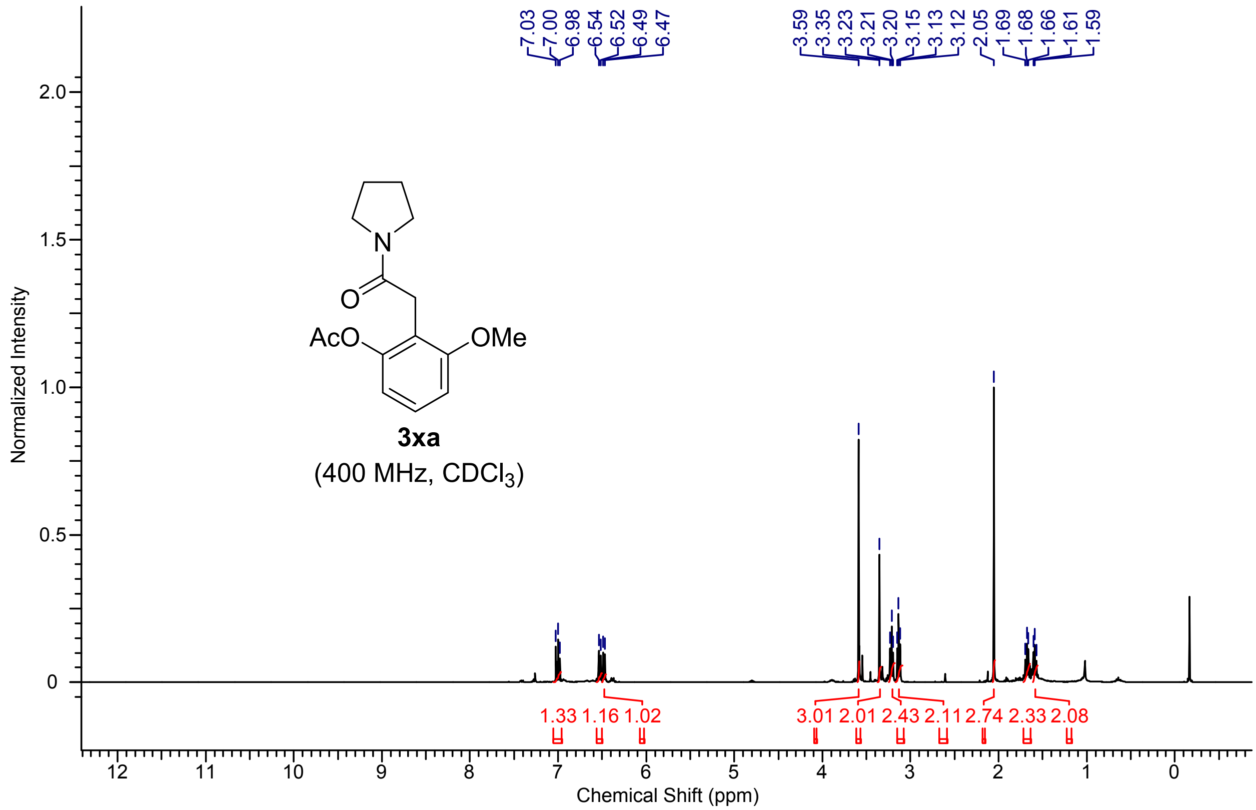

N
Me
O
AcO
OAc
3ua
(100 MHz, CDCl3)
Normalized Intensity
171.27
169.05
148.97
133.74
129.55
128.13
124.71
55.57
48.20
26.89
25.87
21.56
Chemical Shift (ppm)

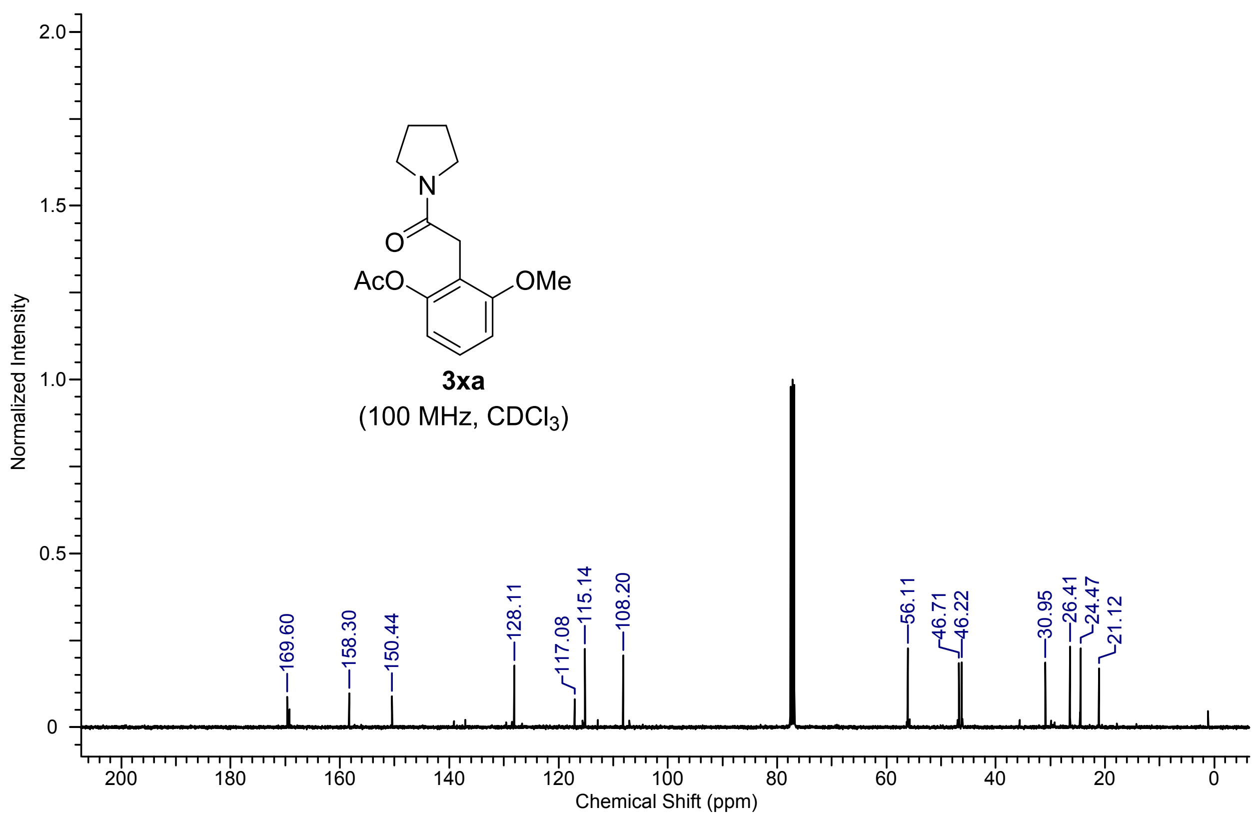

7.25
7.23
7.20
6.93
6.91
3.78
3.76
3.75
3.73
3.52
3.40
3.01
3.00
2.99
2.98
2.97
2.26
1.43
1.30
1.28
1.21
1.17
0.99
N
Me
O
AcO
OAc
3va
(400 MHz, $CDCl_3$)
Normalized Intensity
1.14 1.87
1.11 1.10 1.19 1.94 6.04 4.27 3.35 1.27 1.14
Chemical Shift (ppm)

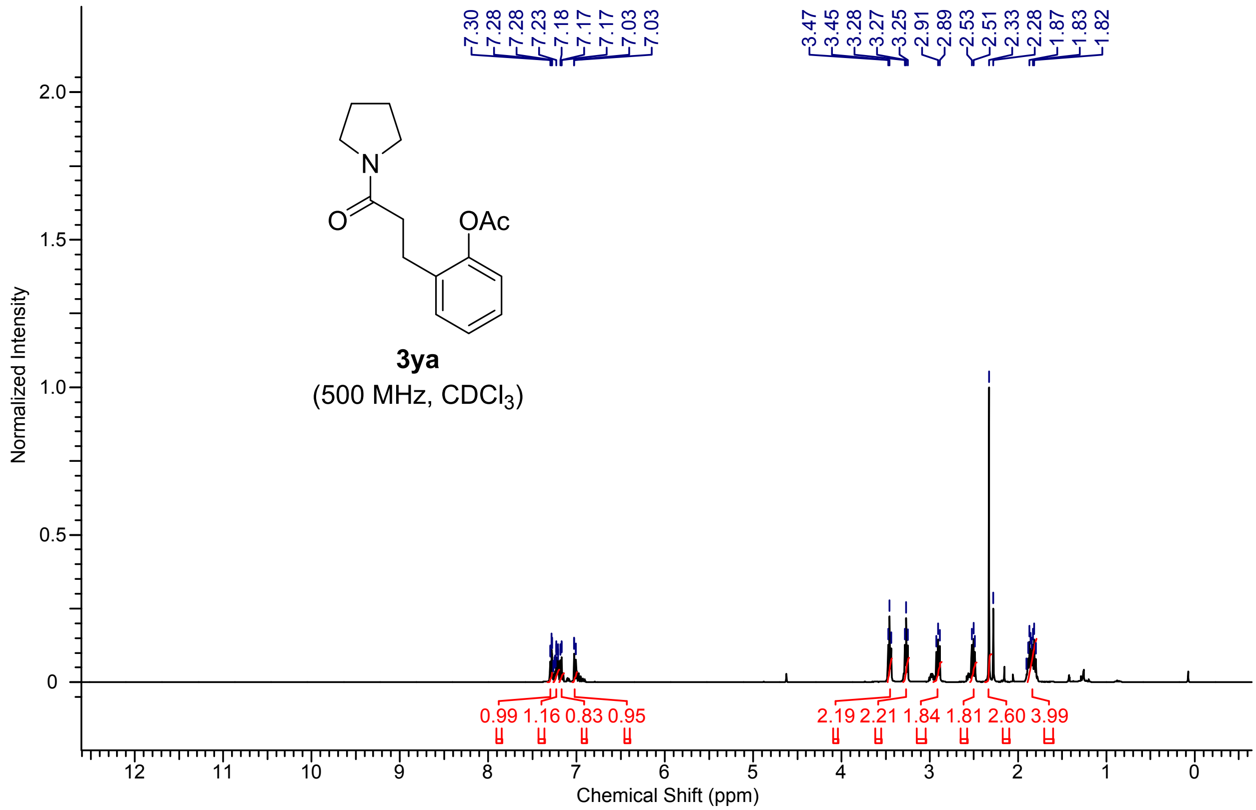

N
Me
O
AcO
OAc
3va
(100 MHz, $CDCl_3$)
Normalized Intensity
170.47
169.31
148.78
127.70
127.54
121.12
46.24
43.25
35.31
25.46
24.48
20.80
17.12
Chemical Shift (ppm)

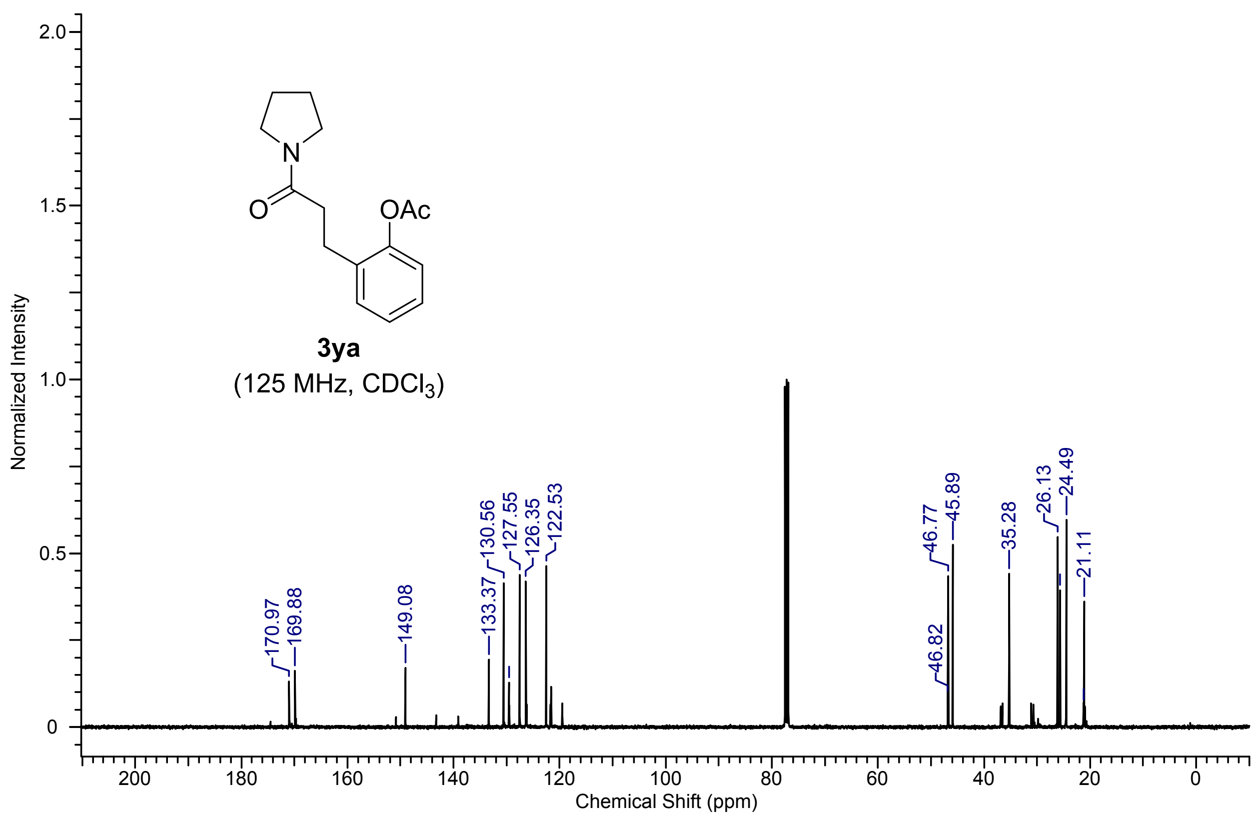

7.31
7.29
7.28
7.01
7.00
3.53
3.44
3.42
3.41
3.22
3.20
3.19
2.30
1.89
1.87
1.86
1.82
1.81
1.79
N
O
AcO
OAc
3wa
(400 MHz, $CDCl_3$)
1.18
1.71
1.84
2.06
1.81
6.04
2.25
2.20
Normalized Intensity
Chemical Shift (ppm)

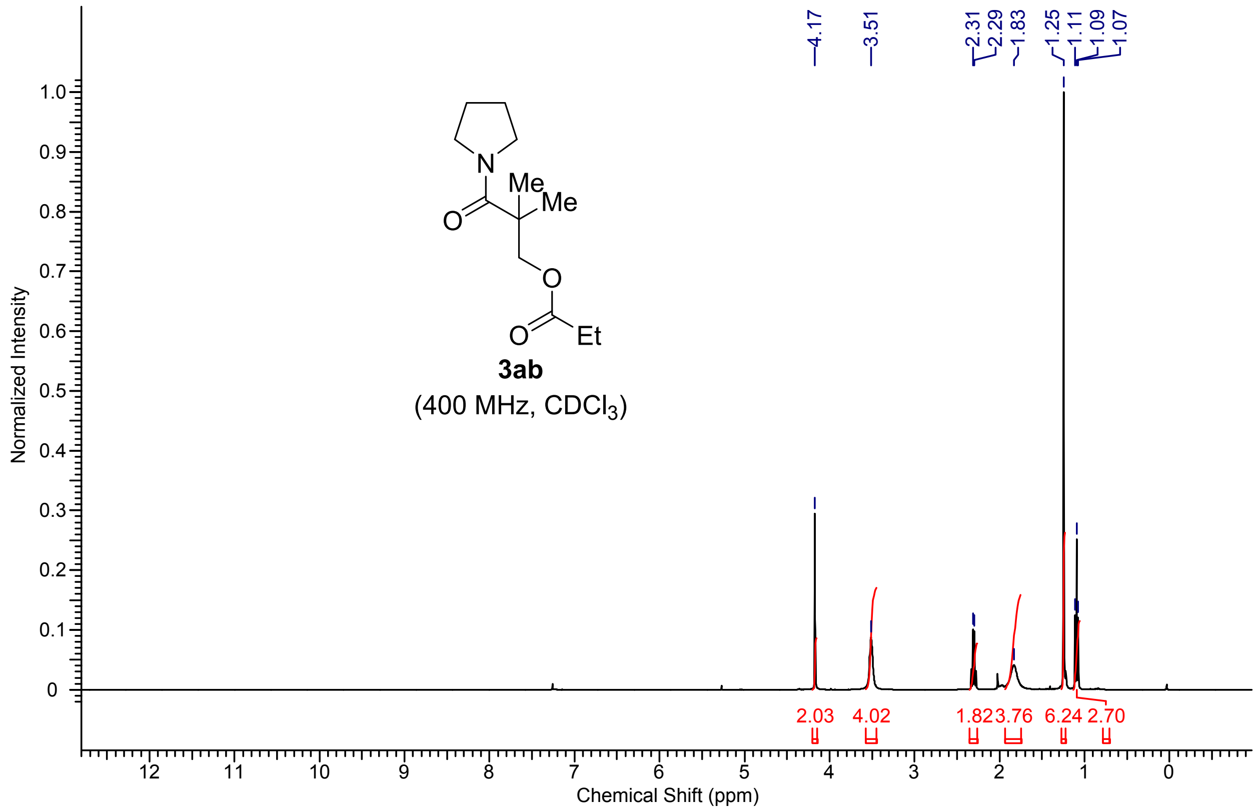

N
O
AcO
OAc
3wa
(100 MHz, $CDCl_3$)
169.03
168.01
149.99
127.94
120.36
46.75
46.43
32.43
26.41
24.33
21.07
Normalized Intensity
Chemical Shift (ppm)


7.03
7.00
6.98
6.54
6.52
6.49
6.47
3.59
3.35
3.23
3.21
3.20
3.15
3.13
3.12
2.05
1.69
1.68
1.66
1.61
1.59
N
O
AcO
OMe
3xa
(400 MHz, $CDCl_3$)
1.33 1.16 1.02
3.01 2.01 2.43 2.11 2.74 2.33 2.08
Normalized Intensity
Chemical Shift (ppm)

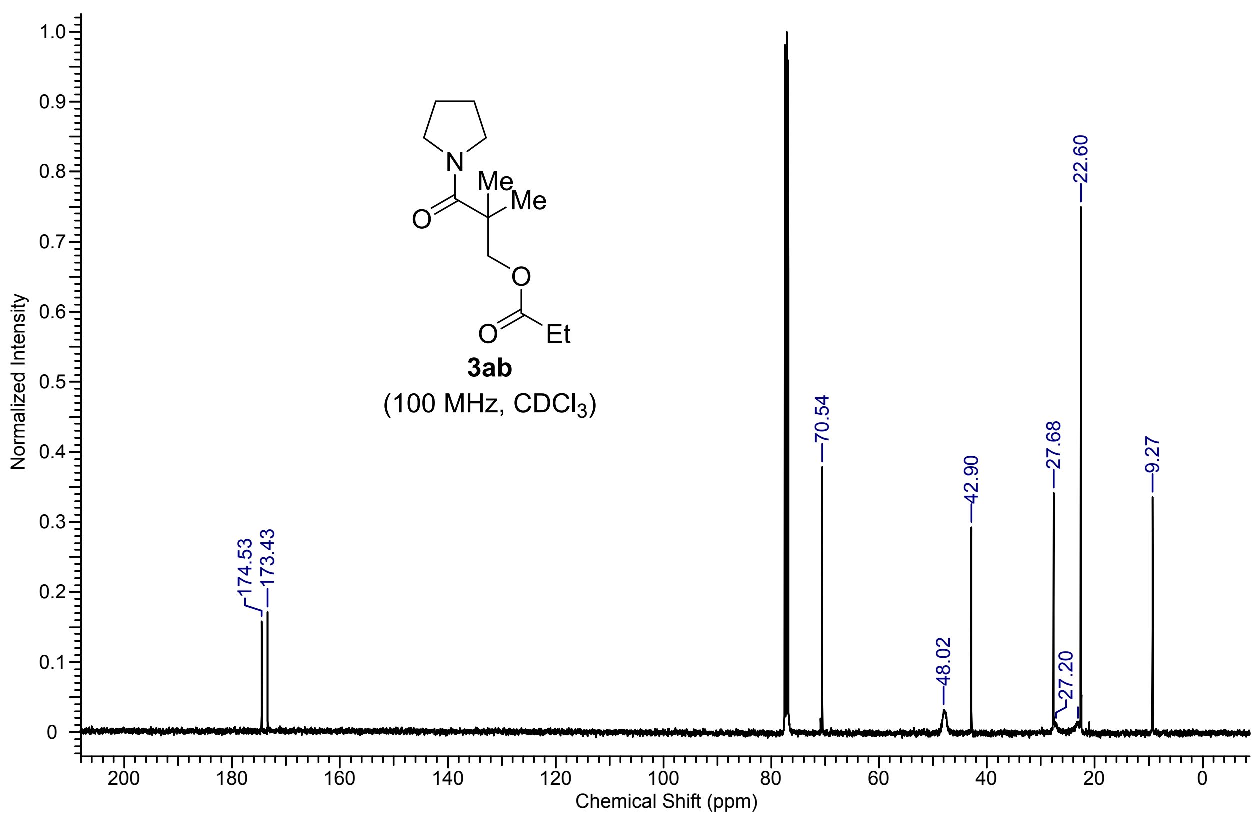

N
O
AcO
OMe
3xa
(100 MHz, $CDCl_3$)
169.60
158.30
150.44
128.11
117.08
115.14
108.20
56.11
46.71
46.22
30.95
26.41
24.47
21.12
Normalized Intensity
Chemical Shift (ppm)


7.30
7.28
7.28
7.23
7.18
7.17
7.17
7.03
7.03
3.47
3.45
3.28
3.27
3.25
2.91
2.89
2.53
2.51
2.33
2.28
1.87
1.83
1.82
OAc
N
O
3ya
(500 MHz, $CDCl_3$)
0.99 1.16 0.83 0.95
2.19 2.21 1.84 1.81 2.60 3.99
Normalized Intensity
Chemical Shift (ppm)

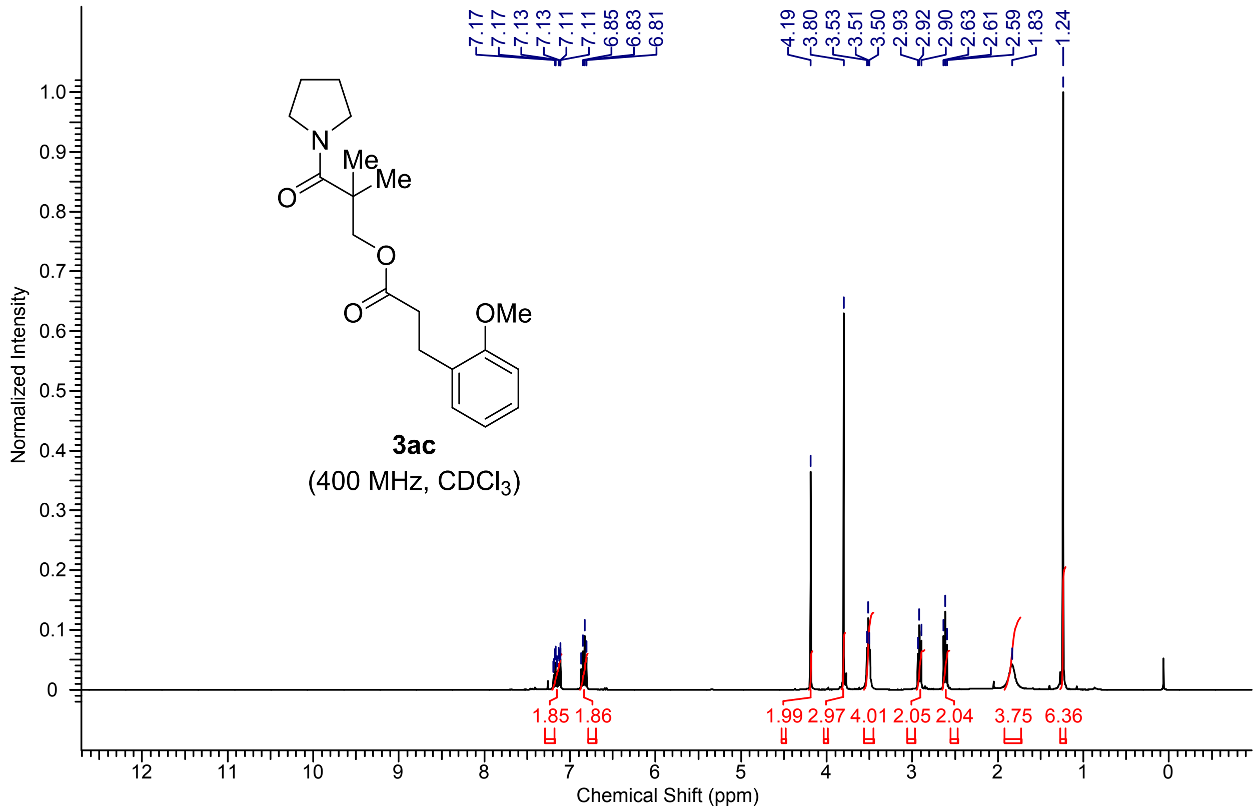

N
O
OAc
3ya
(125 MHz, $CDCl_3$)
170.97
169.88
149.08
133.37
130.56
127.55
126.35
122.53
46.82
46.77
45.89
35.28
26.13
24.49
21.11
Normalized Intensity
Chemical Shift (ppm)



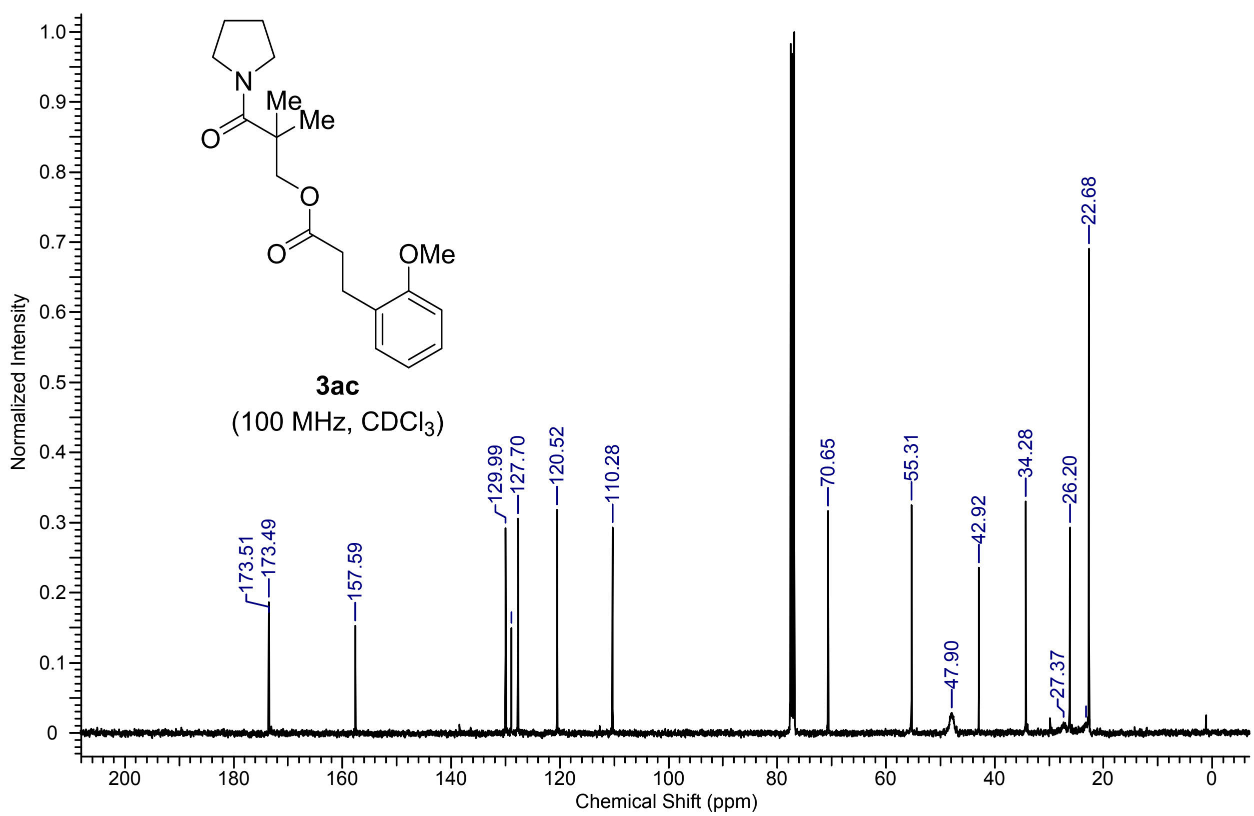
4.17
3.51
2.31
2.29
1.83
1.25
1.11
1.09
1.07
Me
Me
N
O
O
O
Et
3ab
(400 MHz, CDCl3)
Normalized Intensity
2.03
4.02
1.82
3.76
6.24
2.70
Chemical Shift (ppm)



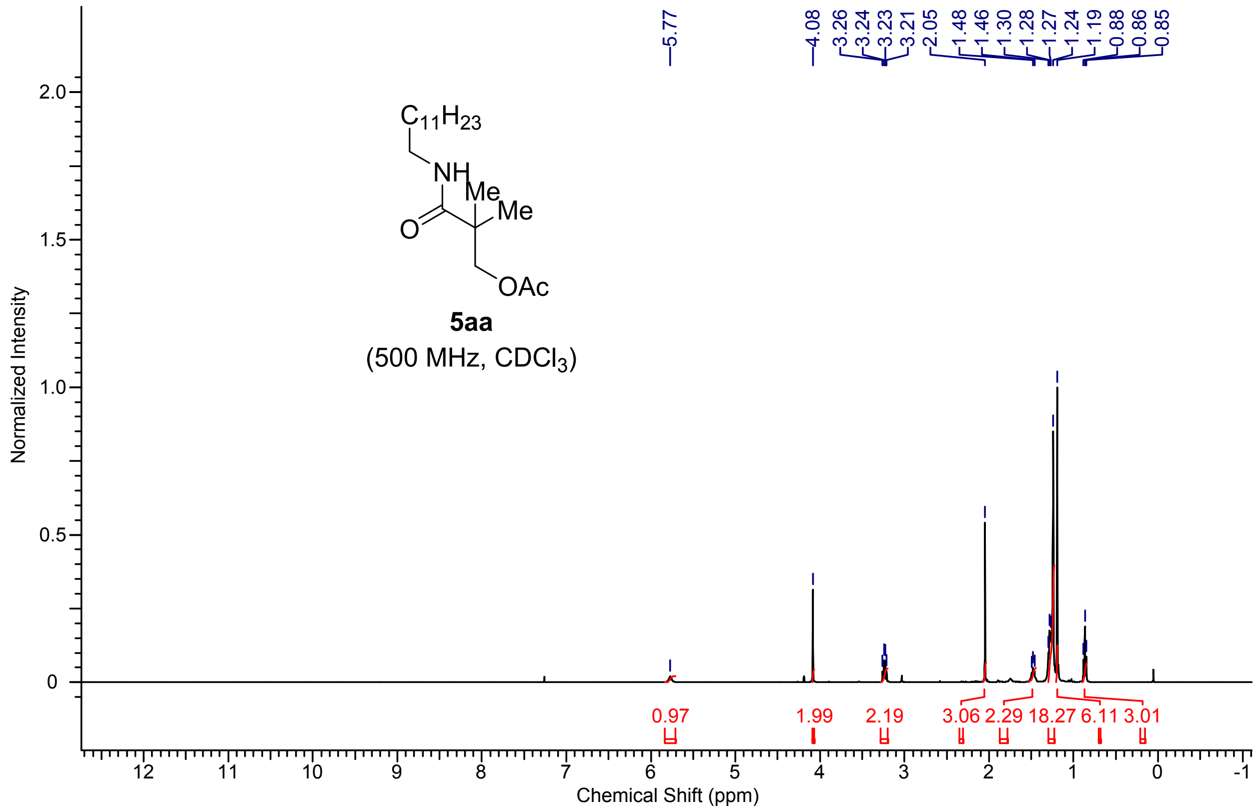
N
Me
Me
O
O
O
Et
3ab
(100 MHz, CDCl3)
Normalized Intensity
174.53
173.43
70.54
48.02
42.90
27.68
27.20
22.60
9.27
Chemical Shift (ppm)

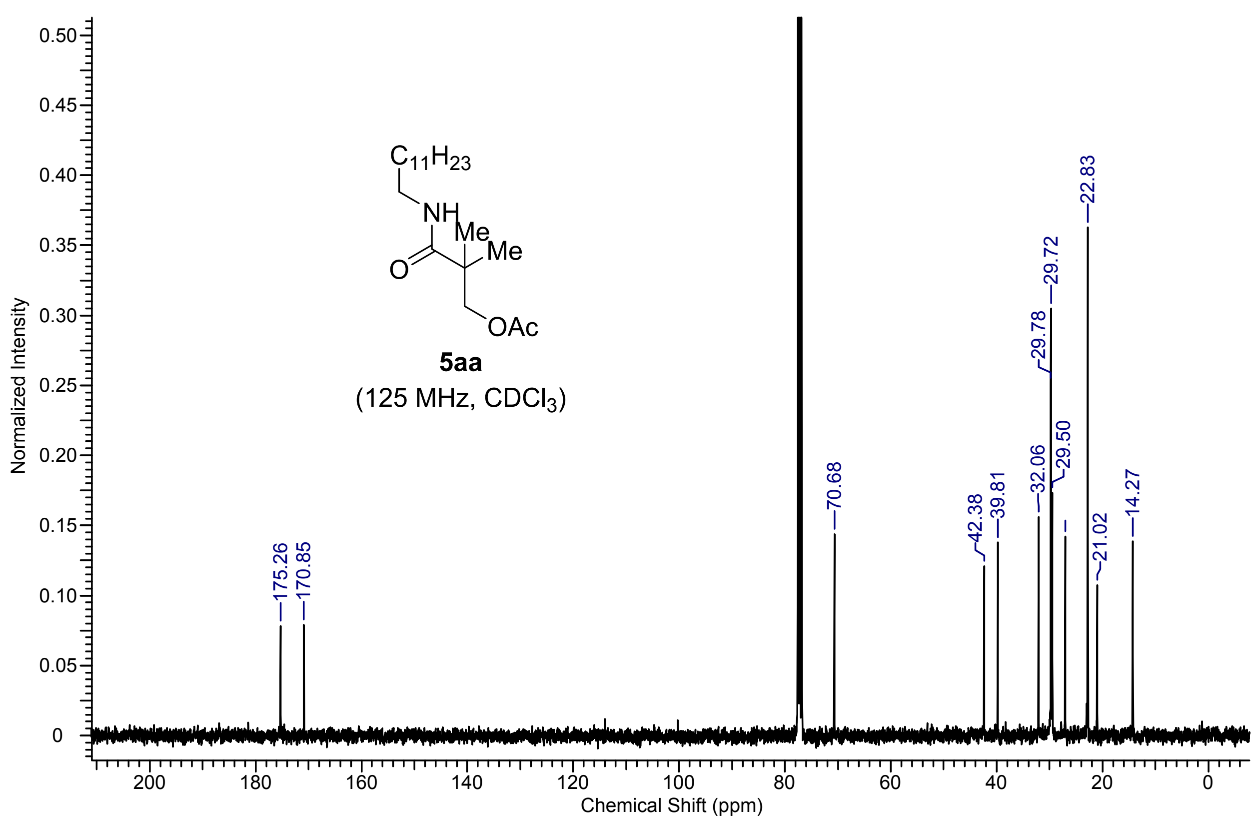


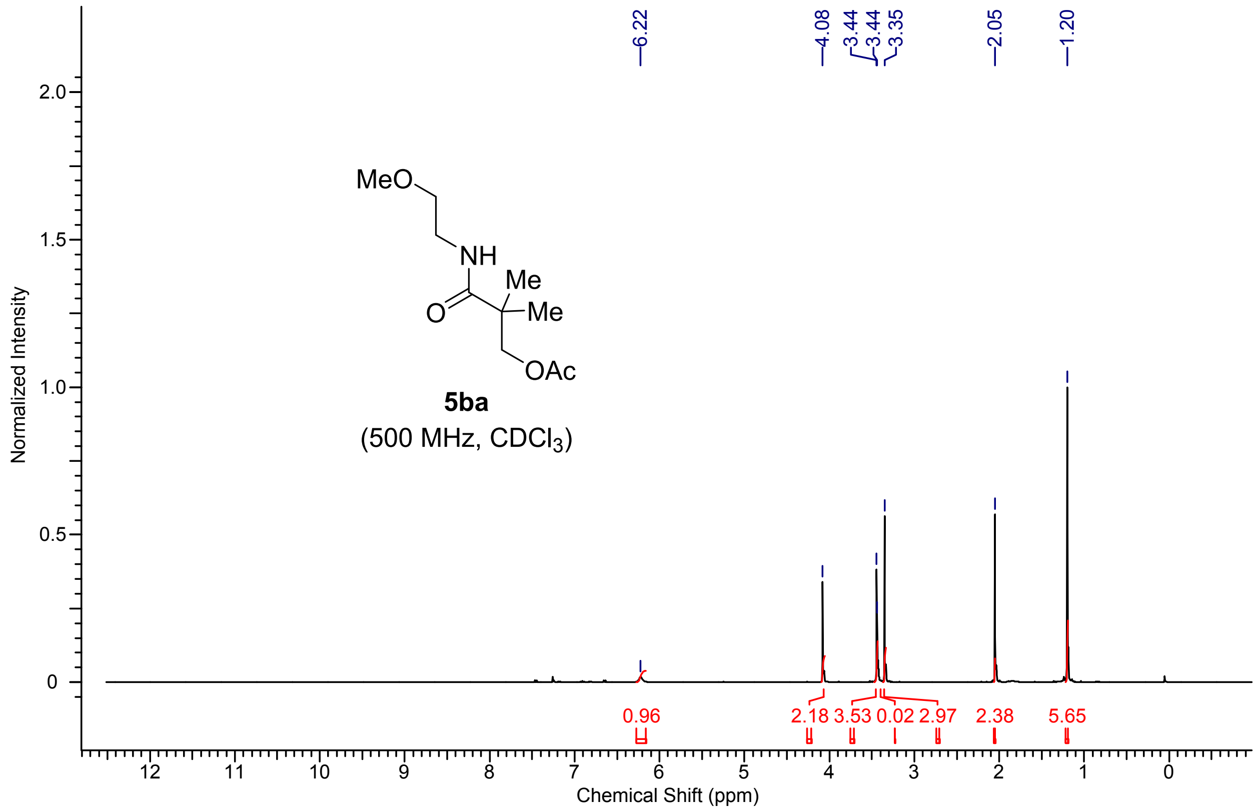
7.17
7.17
7.13
7.13
7.11
7.11
6.85
6.83
6.81
4.19
3.80
3.53
3.51
3.50
2.93
2.92
2.90
2.63
2.61
2.59
1.83
1.24
Me
Me
N
O
O
O
OMe
3ac
(400 MHz, $CDCl_3$)
1.85
1.86
1.99
2.97
4.01
2.05
2.04
3.75
6.36
Normalized Intensity
Chemical Shift (ppm)

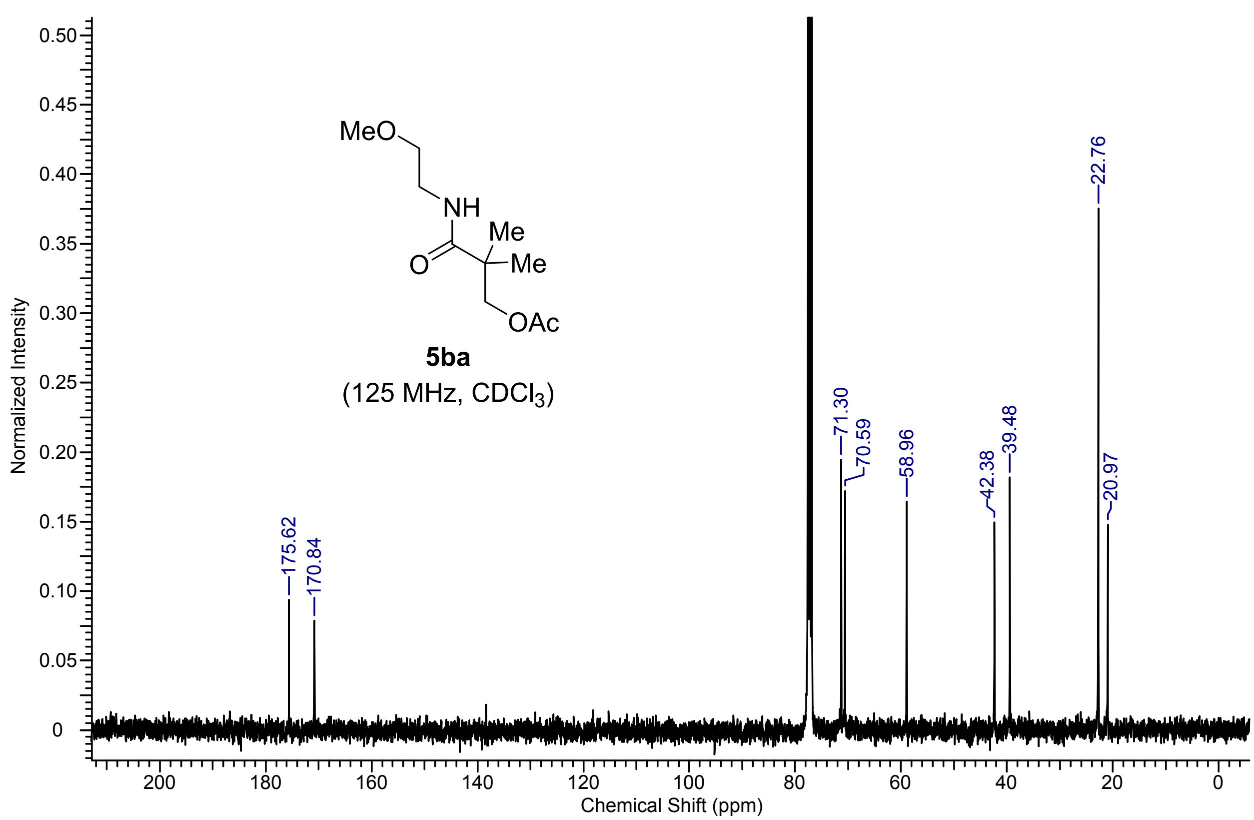


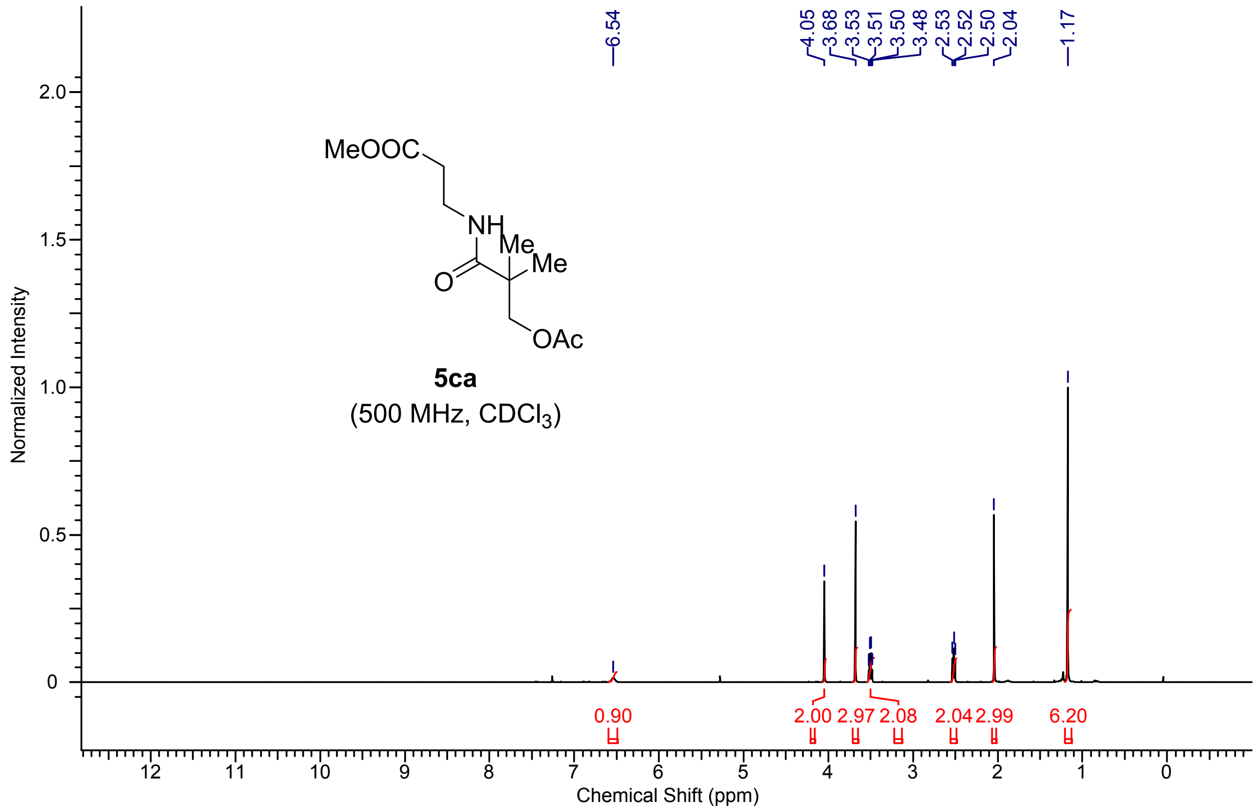
173.51
173.49
157.59
129.99
127.70
120.52
110.28
70.65
55.31
47.90
42.92
34.28
27.37
26.20
22.68
Me
Me
N
O
O
O
OMe
3ac
(100 MHz, $CDCl_3$)
Normalized Intensity
Chemical Shift (ppm)

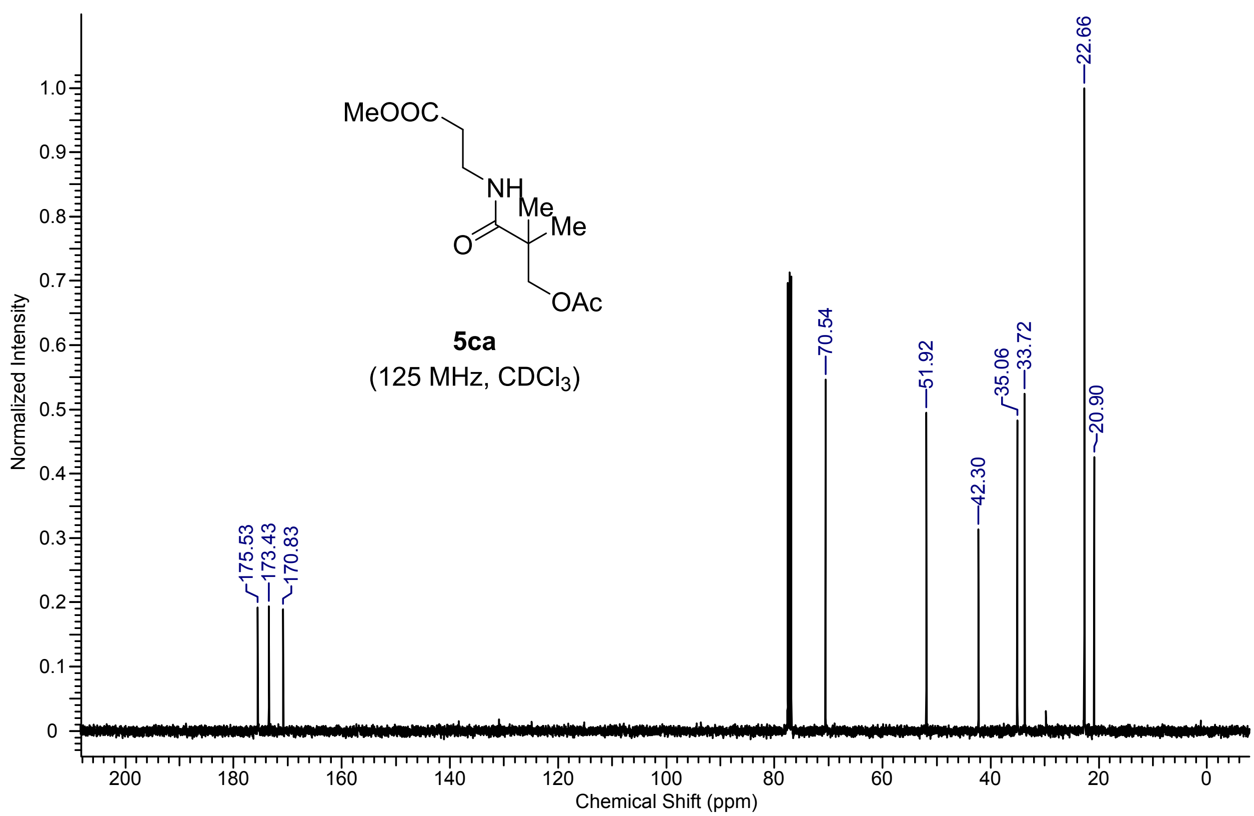

5.77
4.08
3.26
3.24
3.23
3.21
2.05
1.48
1.46
1.30
1.28
1.27
1.24
1.19
0.88
0.86
0.85
C11H23
NH
Me
Me
O
OAc
5aa
(500 MHz, CDCl3)
0.97
1.99
2.19
3.06
2.29
18.27
6.11
3.01
Normalized Intensity
Chemical Shift (ppm)

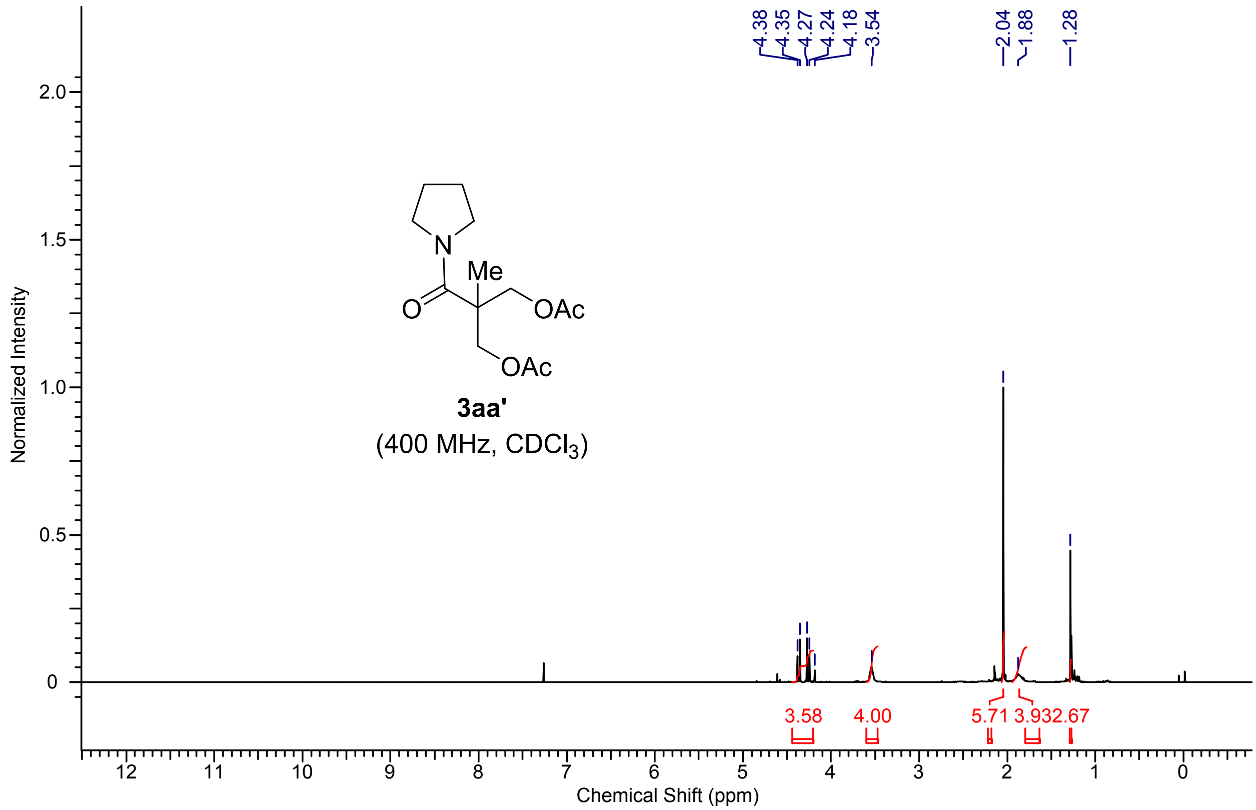

C11H23
NH
Me
Me
O
OAc
5aa
(125 MHz, CDCl3)
175.26
170.85
70.68
42.38
39.81
32.06
29.78
29.72
29.50
22.83
21.02
14.27
Normalized Intensity
Chemical Shift (ppm)

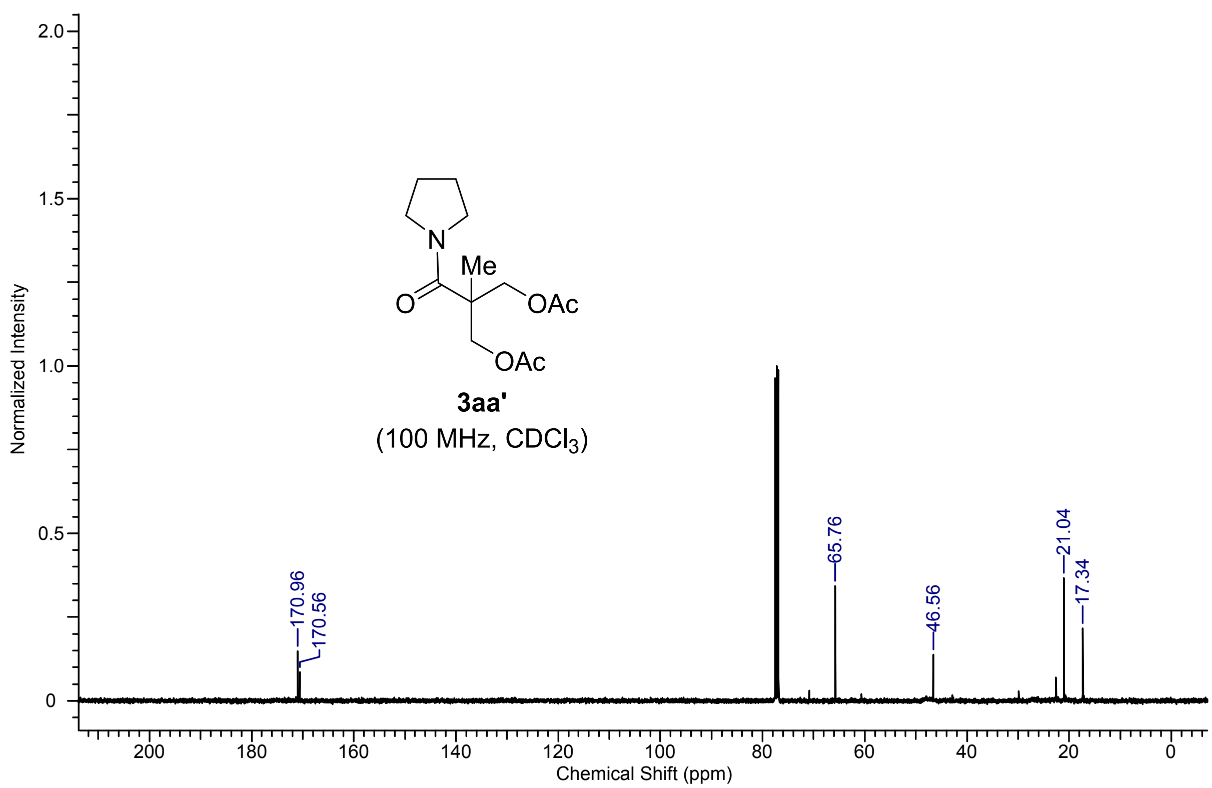

6.22
4.08
3.44
3.44
3.35
2.05
1.20
MeO
NH
Me
O
Me
OAc
5ba
(500 MHz, CDCl3)
0.96
2.18
3.53
0.02
2.97
2.38
5.65
Normalized Intensity
Chemical Shift (ppm)

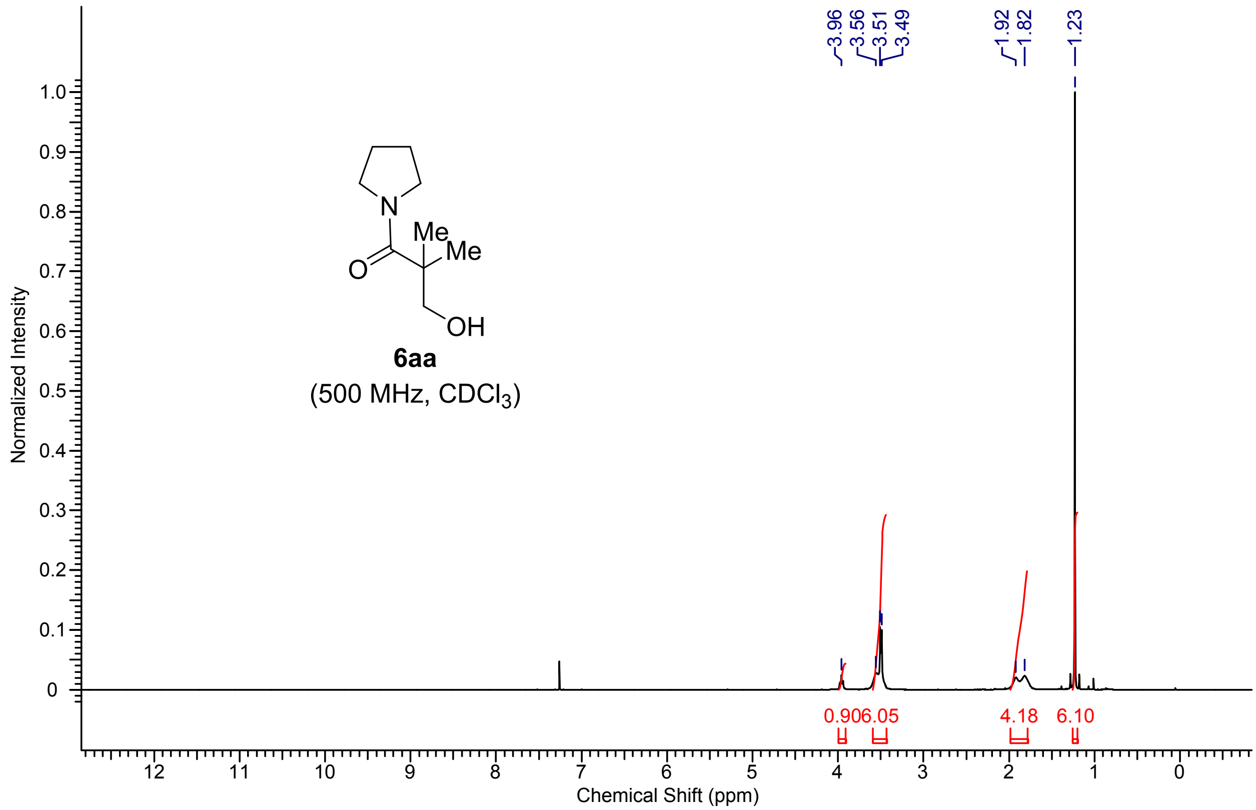

MeO
NH
Me
O
Me
OAc
5ba
(125 MHz, CDCl3)
175.62
170.84
71.30
70.59
58.96
42.38
39.48
22.76
20.97
Normalized Intensity
Chemical Shift (ppm)


6.54
4.05
3.68
3.53
3.51
3.50
3.48
2.53
2.52
2.50
2.04
1.17
MeOOC
NH
Me
Me
O
OAc
5ca
(500 MHz, CDCl3)
0.90
2.00
2.97
2.08
2.04
2.99
6.20
Normalized Intensity
Chemical Shift (ppm)

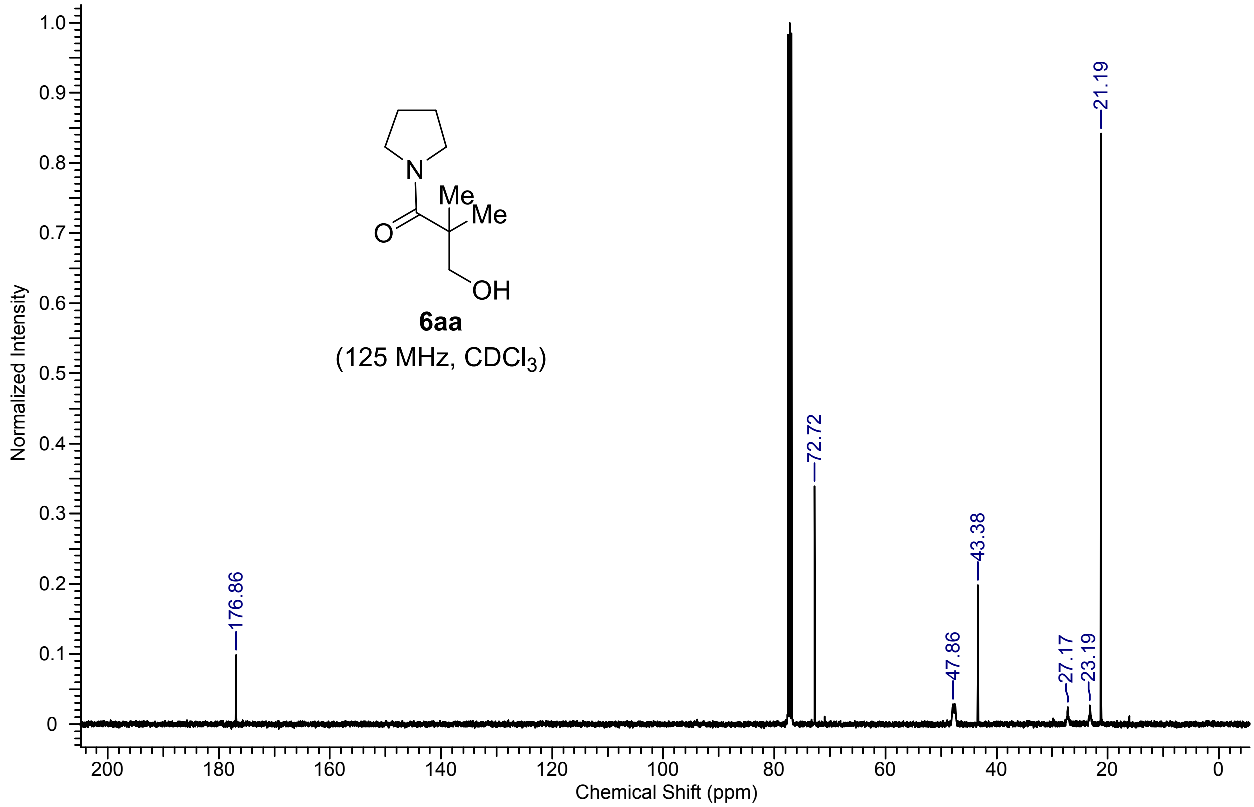

22.66
70.54
51.92
35.06
33.72
20.90
42.30
175.53
173.43
170.83
MeOOC
NH
Me
Me
O
OAc
5ca
(125 MHz, CDCl3)
Normalized Intensity
Chemical Shift (ppm)


4.38
4.35
4.27
4.24
4.18
3.54
2.04
1.88
1.28
Me
OAc
OAc
O
N
3aa'
(400 MHz, CDCl3)
3.58
4.00
5.71
3.93
2.67
Normalized Intensity
Chemical Shift (ppm)

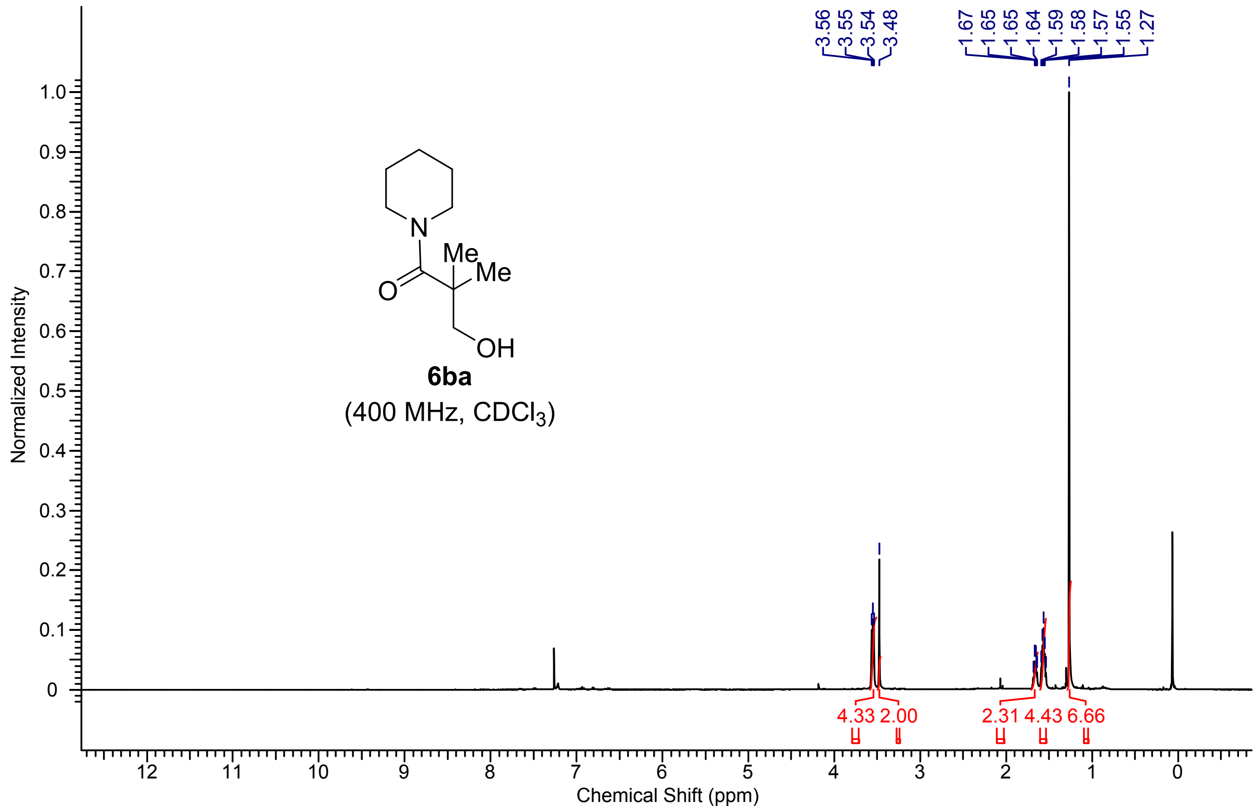

Me
OAc
OAc
O
N
3aa'
(100 MHz, CDCl3)
170.96
170.56
65.76
46.56
21.04
17.34
Normalized Intensity
Chemical Shift (ppm)



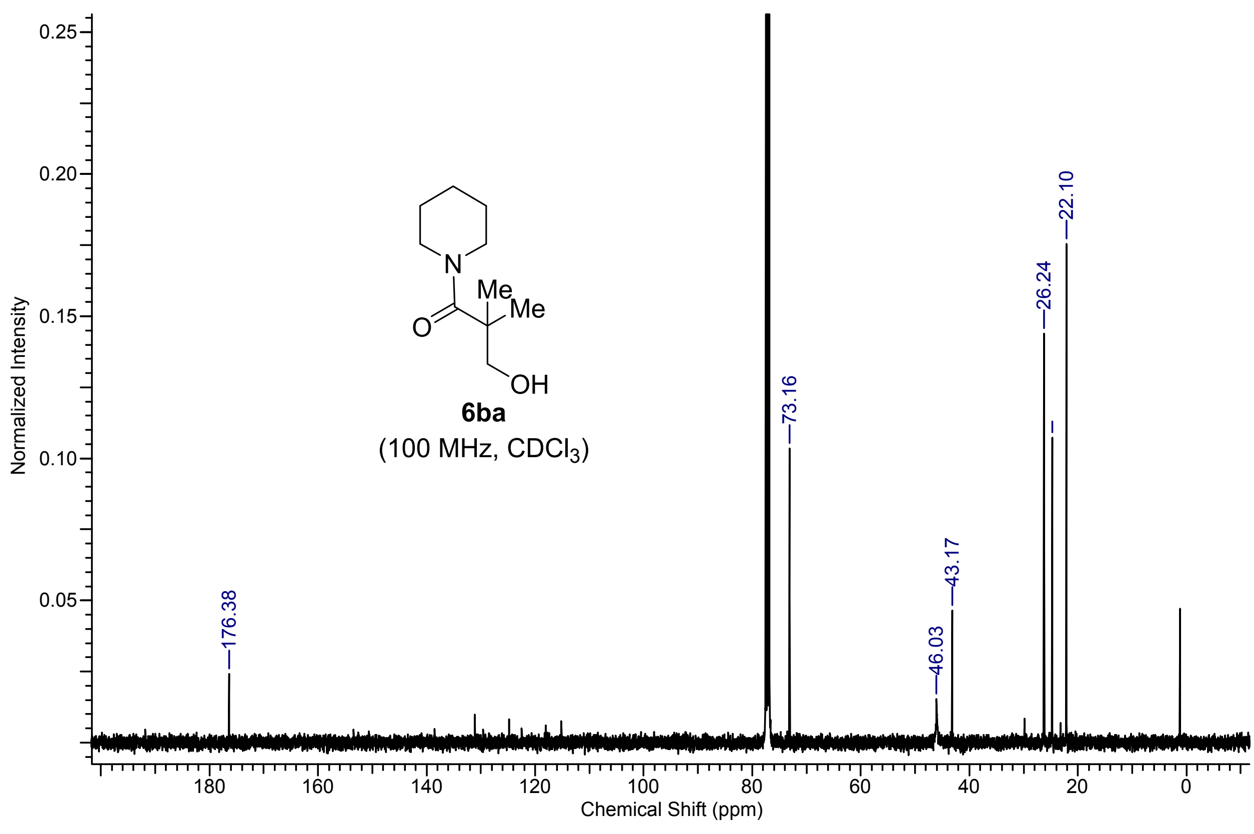
3.96
3.56
3.51
3.49
1.92
1.82
1.23
Me
Me
N
O
OH
6aa
(500 MHz, CDCl3)
Normalized Intensity
0.90
6.05
4.18
6.10
Chemical Shift (ppm)



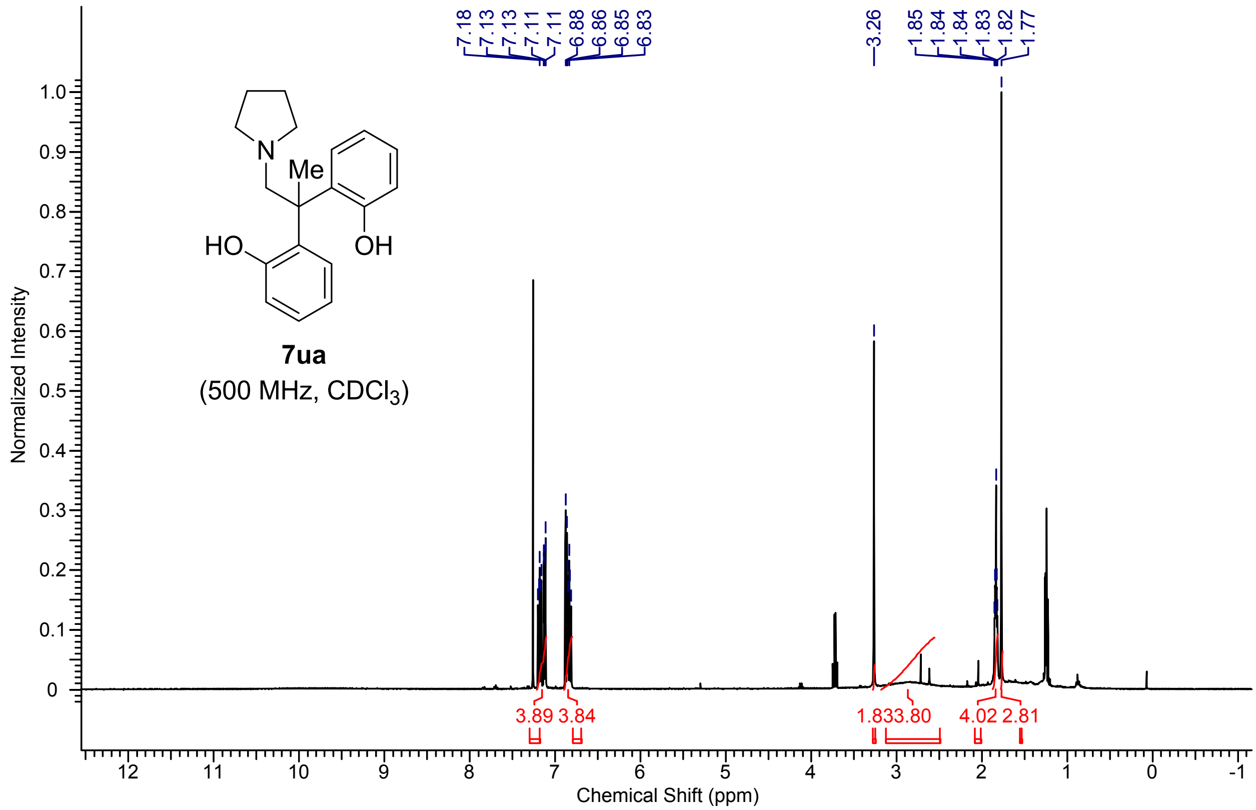
Me
Me
N
O
OH
6aa
(125 MHz, CDCl3)
Normalized Intensity
176.86
72.72
47.86
43.38
27.17
23.19
21.19
Chemical Shift (ppm)

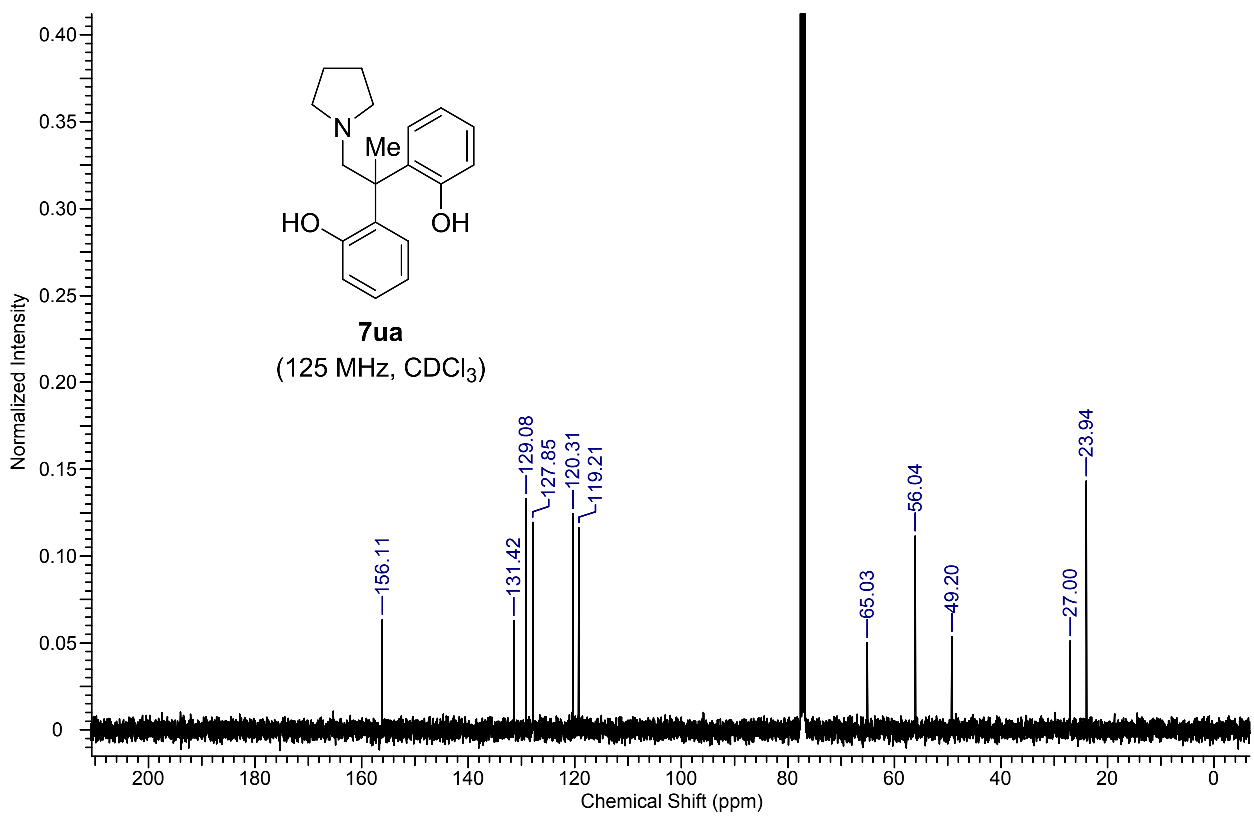


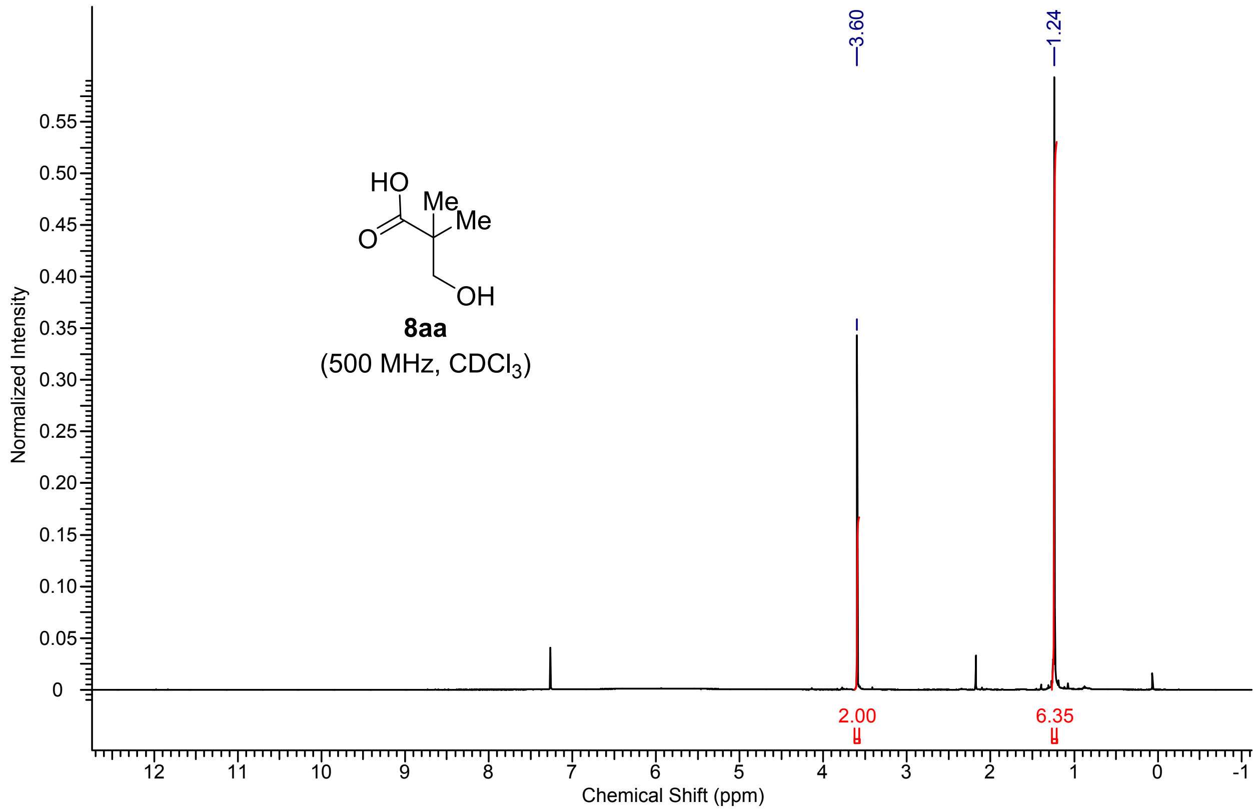
3.56
3.55
3.54
3.48
1.67
1.65
1.65
1.64
1.59
1.58
1.57
1.55
1.27
Me
Me
O
N
OH
6ba
(400 MHz, $CDCl_3$)
Normalized Intensity
4.33
2.00
2.31
4.43
6.66
Chemical Shift (ppm)

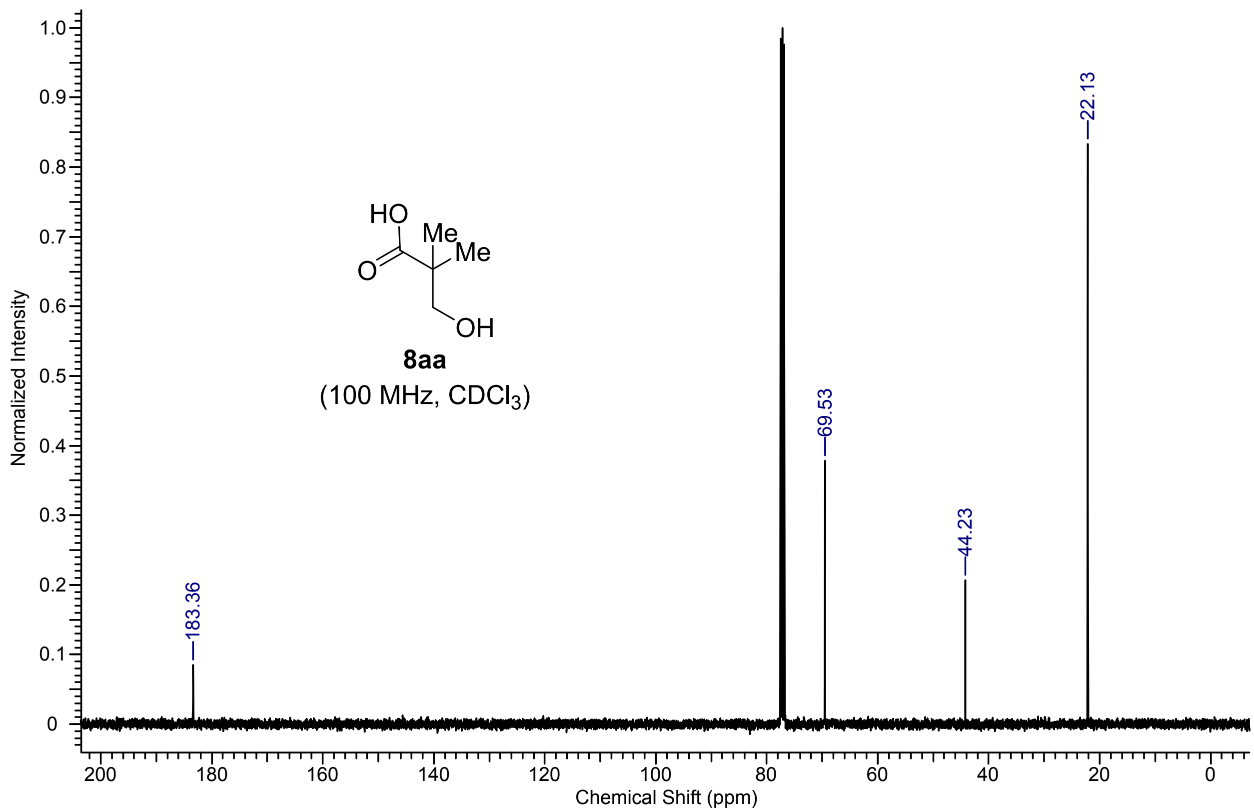


176.38
73.16
46.03
43.17
26.24
22.10
Me
Me
O
N
OH
6ba
(100 MHz, $CDCl_3$)
Normalized Intensity
Chemical Shift (ppm)


7ua
(500 MHz, $CDCl_3$)
Normalized Intensity
Chemical Shift (ppm)
7.18 7.13 7.13 7.11 7.11 6.88 6.86 6.85 6.83
3.26
1.85 1.84 1.84 1.83 1.82 1.77
3.89 3.84 1.83 3.80 4.02 2.81


7ua
(125 MHz, $CDCl_3$)
Normalized Intensity
Chemical Shift (ppm)
156.11
131.42 129.08 127.85 120.31 119.21
65.03 56.04 49.20 27.00 23.94


HO
Me
Me
O
OH
8aa
(500 MHz, $CDCl_3$)
3.60
1.24
2.00
6.35
Normalized Intensity
Chemical Shift (ppm)


HO
Me
Me
O
OH
8aa
(100 MHz, $CDCl_3$)
183.36
69.53
44.23
22.13
Normalized Intensity
Chemical Shift (ppm)