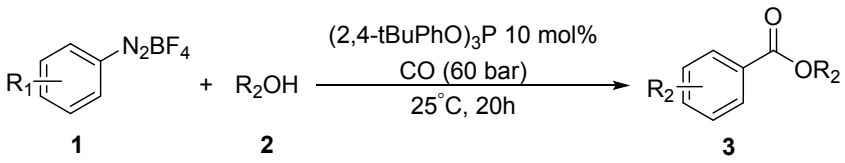
General Information

Unless otherwise noted, materials were purchased from commercial suppliers and used without further purification. Flash column chromatography was performed using 200-300 mesh silica gel. ^{1}H NMR spectra were recorded on 300 or 400 MHz spertrophotometers. chemical shifts are reported in ppm relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard ($CDCl_3$: δ = 7.26 ppm). ^{13}C NMR was recorded at 75 MHz or 100 MHz: chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard ($CDCl_3$: δ = 77.16 ppm). High resolution mass spectra (HR-MS) were recorded on Agilent 6210. The data were given as mass units per charge (m/z).Gas chromatography analysis was performed on an Agilent HP-5890 instrument with a FID detector and HP-5 capillary column (polydimethylsiloxane with 5 % phenyl groups, 30 m, 0.32 mm i.d., 0.25 μm film thickness) using argon as carrier gas.

General procedure for the preparation of aryl diazonium tetrafluoroborates[1,2]

Arylamine (10 mmol) was dissolved in a mixture of 5 mL of distilled water and 3.4 mL of 50% hydrofluoroboric acid. After cooling the reaction mixture to 0 °C using ice bath and the sodium nitrite (0.69 g in 2 mL distilled water) was added dropwise in 5 min interval of time. The resulting mixture was stirred for 1h and the precipitate was collected by filtration and redissolved in minimum amount of acetone. Diethylether was added until precipitation of aryl diazonium tetrafluoroborate, which is filtered, washed several times with diethyl ether and dried under vacuum.

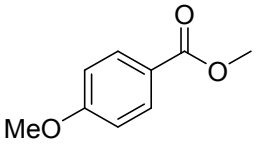
General Procedure



1 **2** **3**

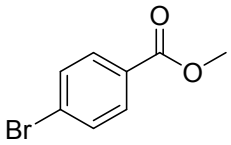
A 4 mL screw-cap vial was charged with tris-(2,4-di-*tert*-butyl-pheny)phosphite (25.9 mg, 10 mol%), 4-methoxybenzene-diazonium tetrafluoroborate(88.8 mg, 0.4 mmol) and an oven-dried stirring bar. The vial was closed by Teflon septum and phenolic cap and connected with atmosphere with a needle. After flashed the vials with argon and vacuum three times, cooled dried Methanol (2 mL) was injected by syringe. The vial was fixed in an alloy plate and put into Paar 4560 series autoclave (500 mL) under argon atmosphere. At room temperature, the autoclave is flushed with carbon monoxide for three times and 60 bar of carbon monoxide was charged. The autoclave was reacted at 25 °C for 20 hours. Afterwards, the pressure was carefully released. After removal of solvent under reduced pressure, pure product was obtained by column chromatography on silica gel.

Methyl 4-methoxybenzoate (3a)[3,4,5]



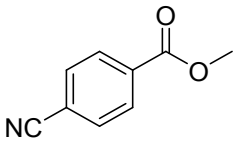
Yellow solid, 71% Yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 – 7.95 (m, 2H), 6.95 – 6.87 (m, 2H), 3.88 (s, 3H), 3.85 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.90, 163.35, 131.61, 122.63, 113.62, 55.44, 51.89. **HRMS** (EI): calcd. for $C_9H_{10}O_3[M]^+$ 166.06245, found 166.06210.

Methyl 4-bromobenzoate (3b)[3,5]



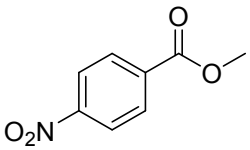
Yellow solid, 74% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.03 – 7.81 (m, 2H), 7.65 – 7.50 (m, 2H), 3.91 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.41, 131.76, 131.16, 129.08, 128.08, 52.34. **HRMS** (EI): calcd. for $C_8H_7O_2Br[M]^+$ 213.96239, found 213.96225.

Methyl 4-cyanobenzoate (3c)[3,5]



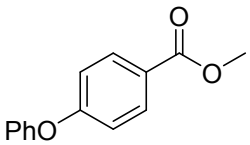
White solid, 63% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.27 – 7.98 (m, 2H), 7.83 – 7.60 (m, 2H), 3.95 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.41, 133.92, 132.22, 130.09, 117.95, 116.39, 52.72. **HRMS** (EI): calcd. for $C_9H_7O_2N[M]^+$ 161.04713, found 161.04684.

Methyl 4-nitrobenzoate (3d)[3,4]



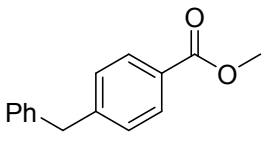
Yellow solid, 64% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.32 – 8.26 (m, 2H), 8.24 – 8.17 (m, 2H), 3.98 (s, 3H). **^{13}C NMR** (75 MHz, CDCl3) δ 165.22, 150.59, 135.53, 130.76, 123.59, 52.88. **HRMS** (EI): calcd. for $C_8H_7O_4N[M]^+$ 181.03696, found 181.03676.

Methyl 4-phenoxybenzoate (3e)[3]



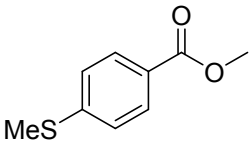
Yellow solid, 78% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.05 – 7.97 (m, 2H), 7.44 – 7.34 (m, 2H), 7.23 – 7.15 (m, 1H), 7.10 – 7.04 (m, 2H), 7.02 – 6.95 (m, 2H), 3.90 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.62, 161.83, 155.63, 131.69, 130.04, 124.51, 124.48, 120.12, 117.29, 52.02. **HRMS** (EI): calcd. for $C_{14}H_{12}O_3[M]^+$ 228.07810, found 228.07806.

Methyl 4-benzylbenzoate (3f)



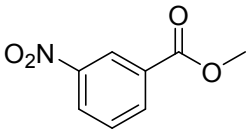
White solid, 73% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.99 – 7.92 (m, 2H), 7.33 – 7.25 (m, 3H), 7.24 – 7.13 (m, 4H), 4.02 (s, 2H), 3.89 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 167.09, 146.55, 129.85, 128.98, 128.64, 128.12, 126.41, 52.04, 41.94. **HRMS**(EI): calcd. for $C_{15}H_{14}O_2[M]^+$ 226.09883, found 226.09918.

Methyl 4-(methylthio)benzoate (3g)



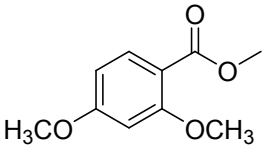
Yellow solid, 69% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.10 – 7.81 (m, 2H), 7.35 – 7.13 (m, 2H), 3.90 (s, 3H), 2.51 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.86, 145.44, 129.88, 126.27, 124.92, 52.03, 14.82. **HRMS** (EI): calcd. for $C_9H_{10}O_2S_1[M]^+$ 182.03960, found 182.03989.

Methyl 3-nitrobenzoate (3h)[3]



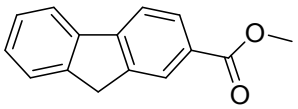
White solid, 56% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.96 – 8.79 (m, 1H), 8.44 – 8.39 (m, 1H), 8.42 – 8.32 (m, 1H), 7.65 (t, *J* = 8.0 Hz, 1H), 3.99 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.00, 148.33, 135.31, 131.91, 129.68, 127.43, 124.65, 52.84. **HRMS** (EI): calcd. for $C_8H_7O_4N_1[M]^+$ 181.03696, found 181.03657.

Methyl 2,4-dimethoxybenzoate (3i)[6]



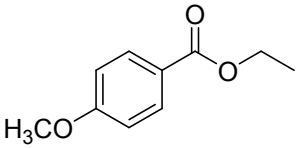
Yellow solid, 34% Yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 – 7.79 (m, 1H), 6.51 – 6.45 (m, 2H), 3.88 (s, 3H), 3.85 (s, 3H), 3.84 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.12, 164.27, 161.33, 133.87, 112.26, 104.53, 98.96, 55.98, 55.49, 51.71. **GC-MS** (EI, 70 eV): m/z = 196.1, 179.1, 165.1, 150.1, 135.1, 122.1, 107.1, 92.1, 77.1, 63.1, 51.1, 41.1. HRMS(EI): calcd. for $C_{10}H_{12}O_4[M]^+$ 196.07301, found 196.07308.

Methyl 9H-fluorene-2-carboxylate (3j)



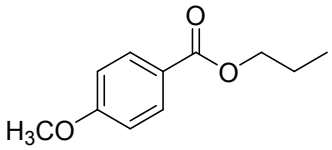
Yellow solid, 34% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.20 (dd, *J* = 1.6, 0.7 Hz, 1H), 8.12 – 8.02 (m, 1H), 7.86 – 7.75 (m, 2H), 7.60 – 7.52 (m, 1H), 7.43 – 7.32 (m, 2H), 3.93 (s, 3H), 3.92 (s, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 167.44, 146.27, 144.36, 143.08, 140.60, 128.70, 128.29, 127.92, 127.02, 126.25, 125.24, 120.79, 119.56, 52.09, 36.83. **HRMS** (EI): calcd. for $C_{15}H_{12}O_2[M]^+$ 224.08318, found 224.08342.

Ethyl 4-methoxybenzoate (3k)[3,5]



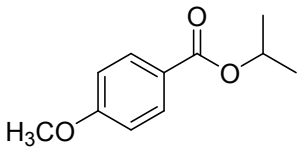
Colorless oil, 56% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.11 – 7.90 (m, 2H), 6.99 – 6.81 (m, 2H), 4.35 (q, *J* = 7.1 Hz, 2H), 3.86 (s, 3H), 1.38 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.44, 163.29, 131.58, 123.01, 113.58, 60.67, 55.45, 14.43. **HRMS** (EI): calcd. for $C_{10}H_{12}O_3[M]^+$ 180.07810, found 180.07822.

Propyl 4-methoxybenzoate (3l)[3]



Compound **3l** was prepared using acetonitrile(1.8 mL) and 1-Propanol(0.2 mL) as solvent. Colorless oil, 42% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.09 – 7.90 (m, 2H), 6.98 – 6.81 (m, 2H), 4.24 (t, *J* = 6.7 Hz, 2H), 3.83 (s, 3H), 1.76 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.39, 163.22, 131.49, 122.93, 113.51, 66.18, 55.34, 22.13, 10.50. **HRMS** (EI): calcd. for $C_{11}H_{14}O_3[M]^+$ 194.09375, found 194.09299.

Isopropyl 4-methoxybenzoate (3m)[3,4,5]



Compound 3m was prepared using acetonitrile(1.8 mL) and Isopropanol (0.2 mL) as solvent. Colorless oil, 36% Yield. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.05 – 7.93 (m, 2H), 6.96 – 6.82 (m, 2H), 5.22 (p, *J* = 6.3 Hz, 1H), 3.84 (s, 3H), 1.35 (s, 3H), 1.33 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.85, 163.16, 131.47, 123.35, 113.46, 67.89, 55.36, 21.98. **HRMS** (EI): calcd. for $C_{11}H_{14}O_3[M]^+$ 194.09375, found 194.09445.

References

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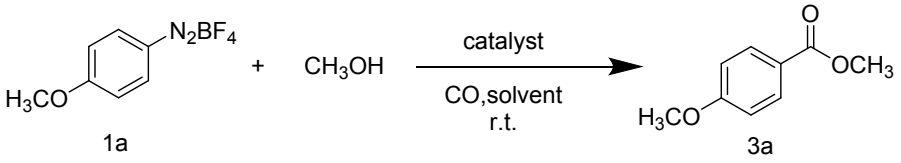
6. Y. Zhu, H. Yan, L. Lu, D. Liu, G. Rong, J. Mao. *J. Org. Chem.,* **2013**, 78, 9898-9905.

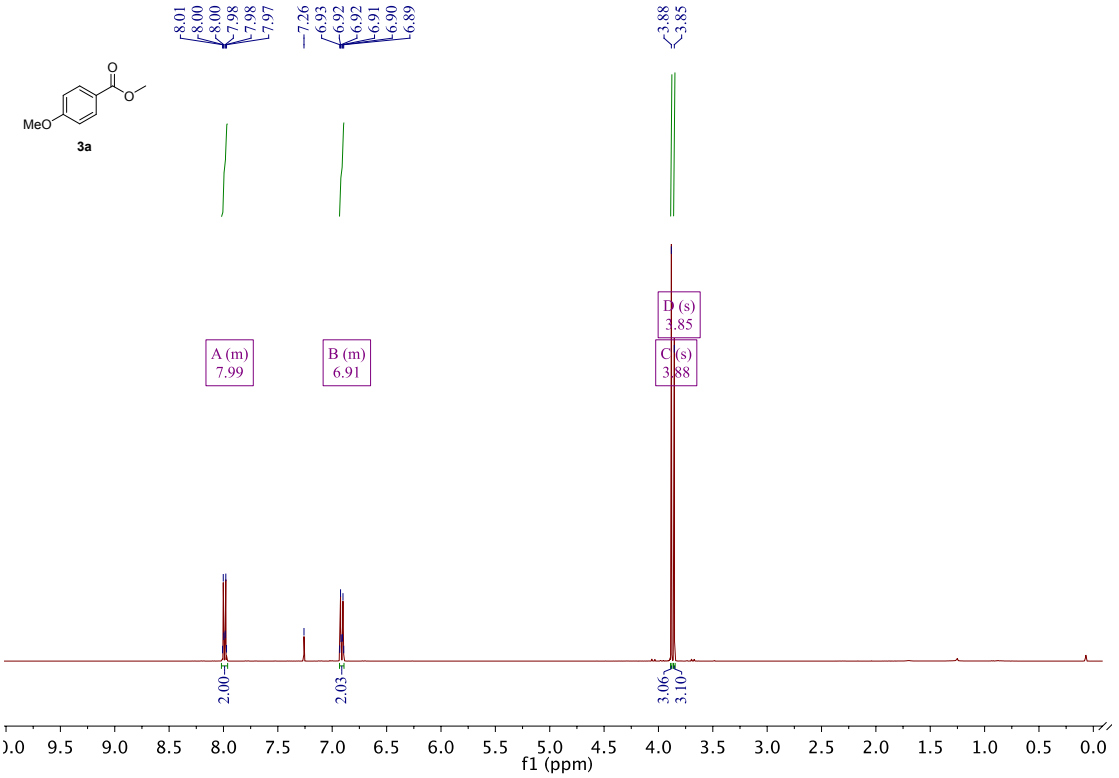
Table S1 Optimization of reaction conditions.[a]

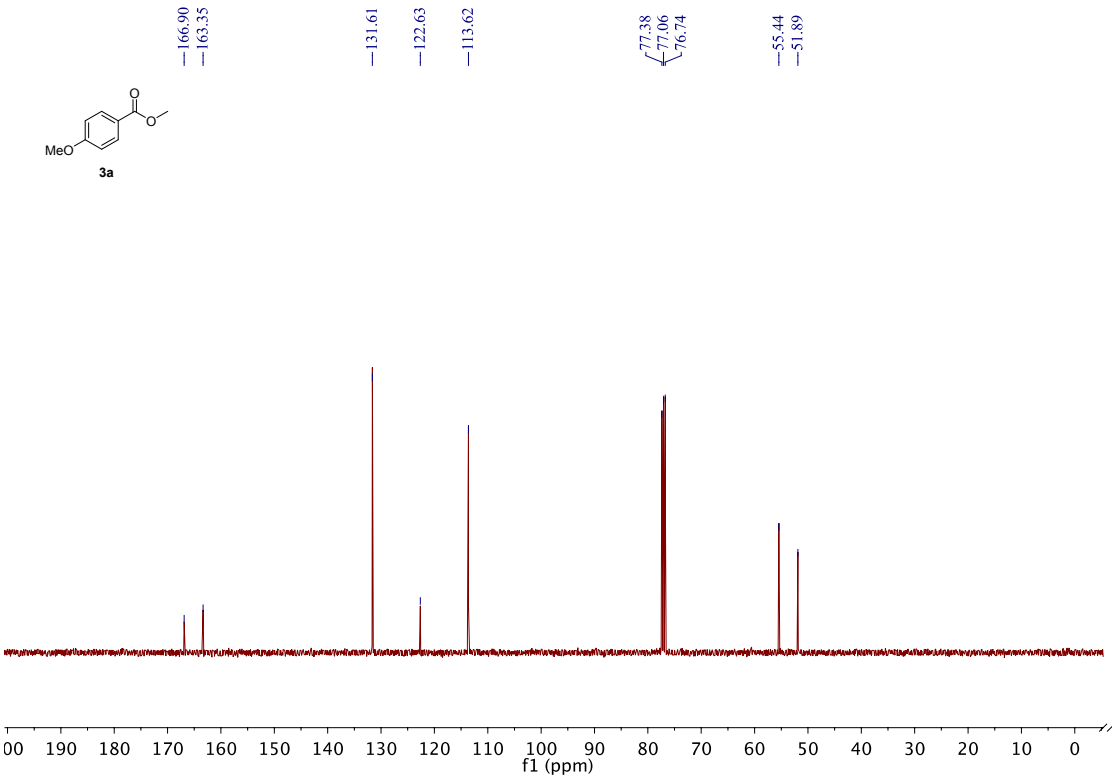


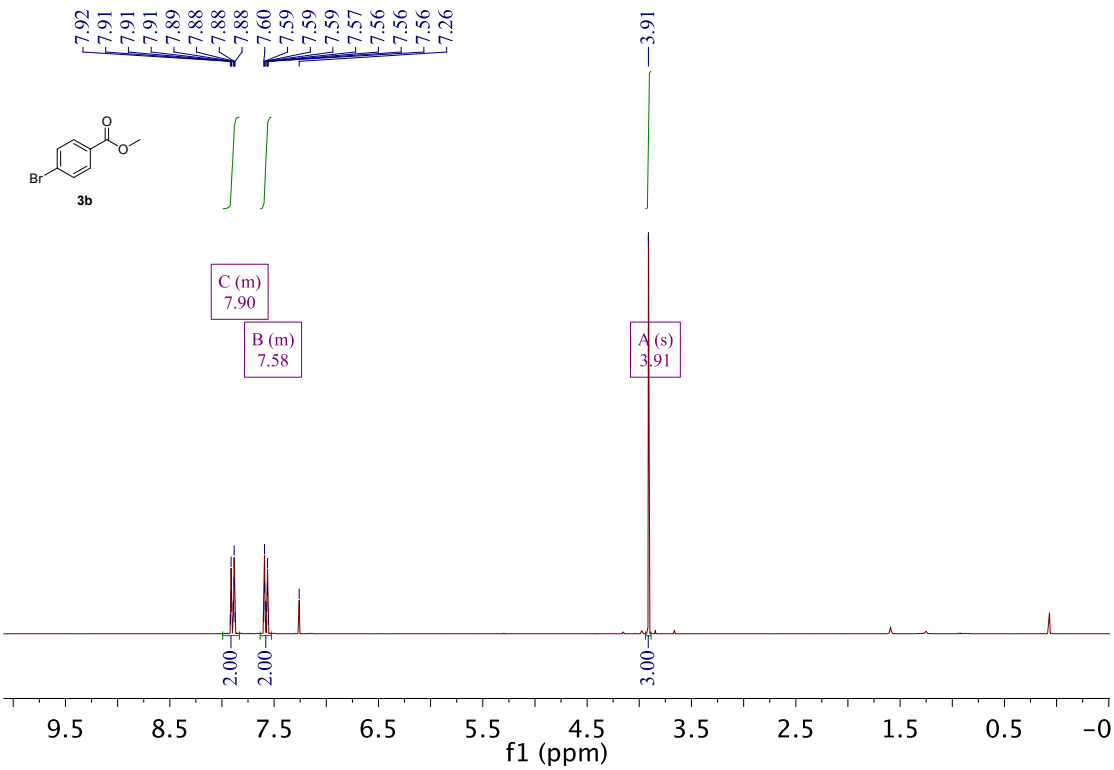
Entry	Catalyst(mol%)	Solvent (0.1M)	Temp.	CO(bar)	Yield%[b]
1[c]	-	Toluene	40	50	NR
2[c]	-	MeOH	40	50	trace
3[c]	-	CH_3CN	40	50	trace
4[c]	-	DMF	r.t.	50	trace
5[c]	-	Dioxane	r.t.	50	nr
6[c]	-	DMSO	r.t.	50	nr
7	-	MeOH	r.t.	50	trace
8	PPh_3 (2)	MeOH	r.t.	50	6
9	PPh_3 (5)	MeOH	r.t.	50	38
10	PPh_3 (10)	MeOH	r.t.	50	58
11	PPh_3 (40)	MeOH	40	50	35
12[c]	PPh_3 (10)	Et_2O	r.t.	50	50
13[c]	PPh_3 (10)	CH_3CN	r.t.	50	29
14[d]	PPh_3 (10)	CH_3CN	r.t.	50	56
15[c]	PPh_3 (10)	THF	r.t.	50	31
16[c]	PPh_3 (10)	Actone	r.t.	50	32
17[c]	PPh_3 (10)	EtOAc	r.t.	50	49
18	PCy_3 (10)	MeOH	r.t.	50	trace
19	dppm (10)	MeOH	r.t.	50	36
20	dppe (10)	MeOH	r.t.	50	21
21	dcype (10)	MeOH	r.t.	50	36
22	dppf (10)	MeOH	r.t.	50	35
23	1,10-Phen (10)	MeOH	r.t.	50	N.R.
24	BINAP (10)	MeOH	r.t.	50	11
25	XantPhos (10)	MeOH	r.t.	50	21
26	Tris(perfluorophenyl)phosphine (10)	MeOH	r.t.	50	30
27	$(MeOC_6H_4)_3P$ (10)	MeOH	r.t.	50	61
28	$(2,4\text{-}tBuPhO)_3P$ (10)	MeOH	r.t.	50	71
29	$[(CH_3)_3C_6H_2]_3P$ (10)	MeOH	r.t.	50	11
30	$(2\text{-}MeOPh)_2PhP$ (10)	MeOH	r.t.	50	45
31[e]	$(2,4\text{-}tBuPhO)_3P$ (10)	MeOH	r.t.	50	62
32	$(2,4\text{-}tBuPhO)_3P$ (10)	MeOH	r.t.	60	73(71)
33	$[(MeO)_3C_6H_2]_3P$ (10)	MeOH	r.t.	60	34
34	$[(CH3)_3C_6H_2]_3P$ (10)	MeOH	r.t.	60	9
35	$(2\text{-}MeOPh)_2PhP$ (10)	MeOH	r.t.	60	19

a Conditions: 1a (0.2 mmol, 0.1M), CH_3OH(2 mL), catalyst(10 mol%), CO(50 bar). Reaction time is 20 hours for every case. N.R. is no reaction; trace represented <5% yield. b Determined by GC analysis using hexadecane as an internal standard. Values in parentheses indicate isolated yields. c CH_3OH(100 eq.). d CH_3OH(200 eq.). e MeOH(1 mL, 0.2M of 1a). 1,10-Phen = 1,10-Phenanthroline

NMR Spectra of products: ^{1}H, ^{13}C NMR



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7.57
7.56
7.56
7.56
7.26
3.91
Br
O
O
3b
C (m)
7.90
B (m)
7.58
A (s)
3.91
2.00
2.00
3.00
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7.5
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5.5
4.5
3.5
2.5
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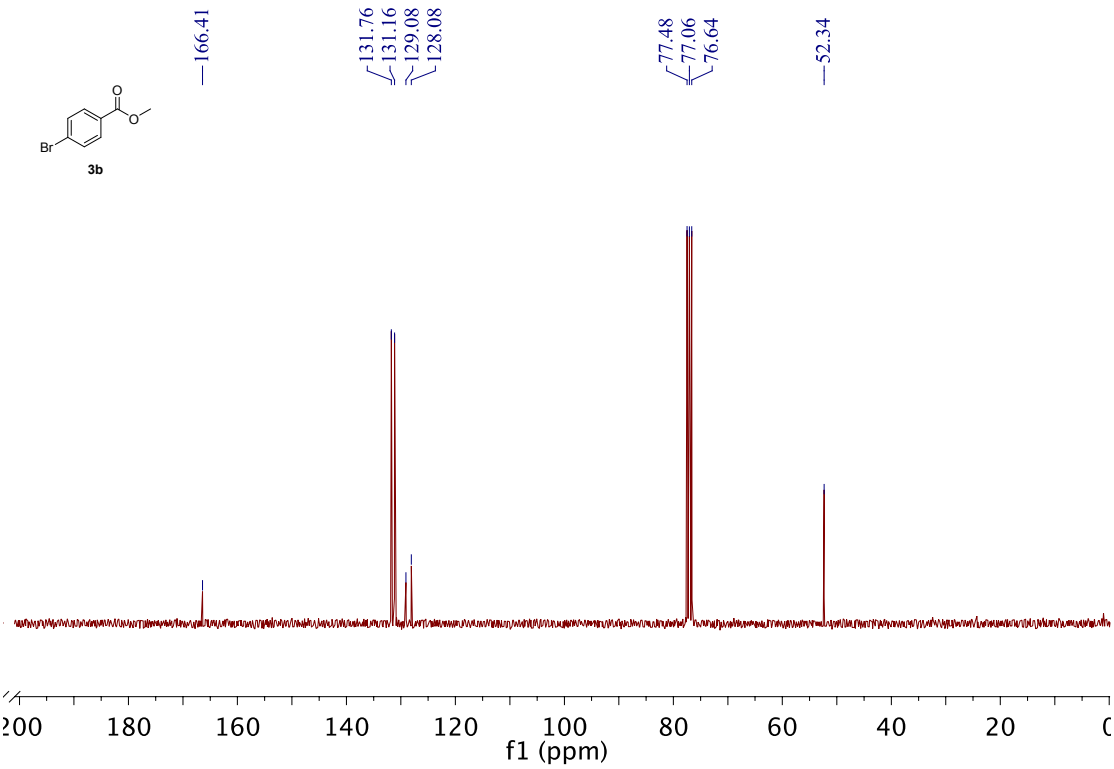
0.5
f1 (ppm)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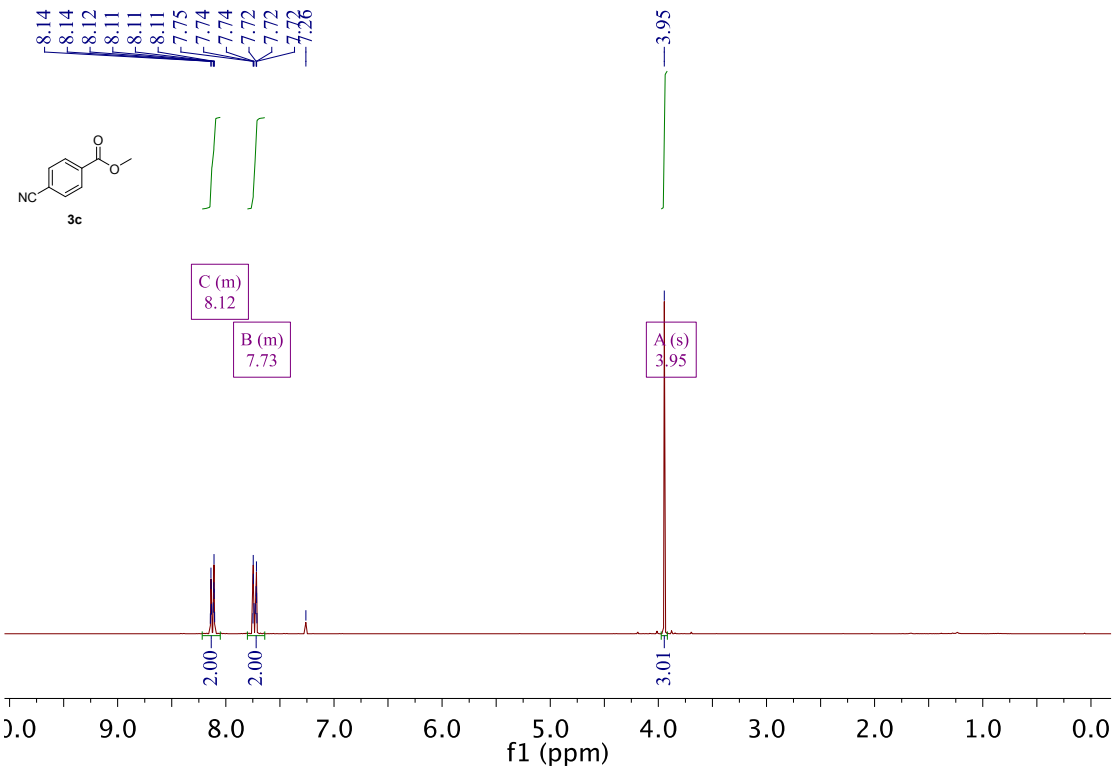
f1 (ppm)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3.95
NC
O
O
3c
C (m)
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B (m)
7.73
A (s)
3.95
2.00
2.00
3.01
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5.0
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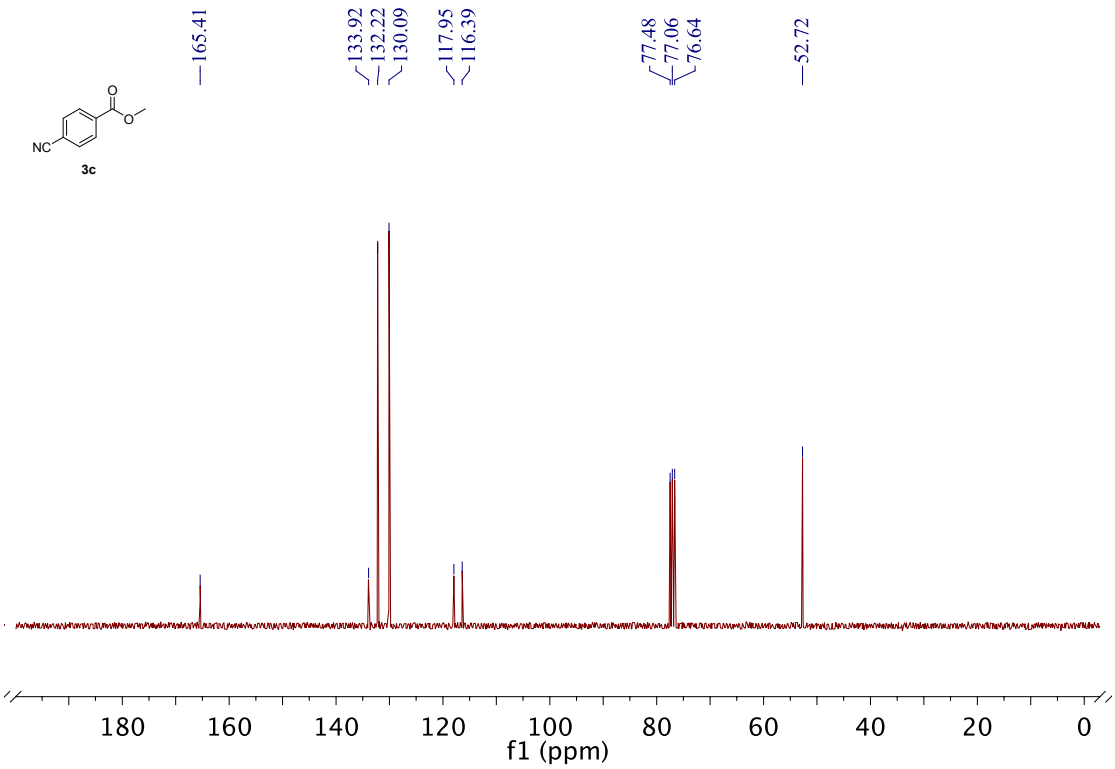
f1 (ppm)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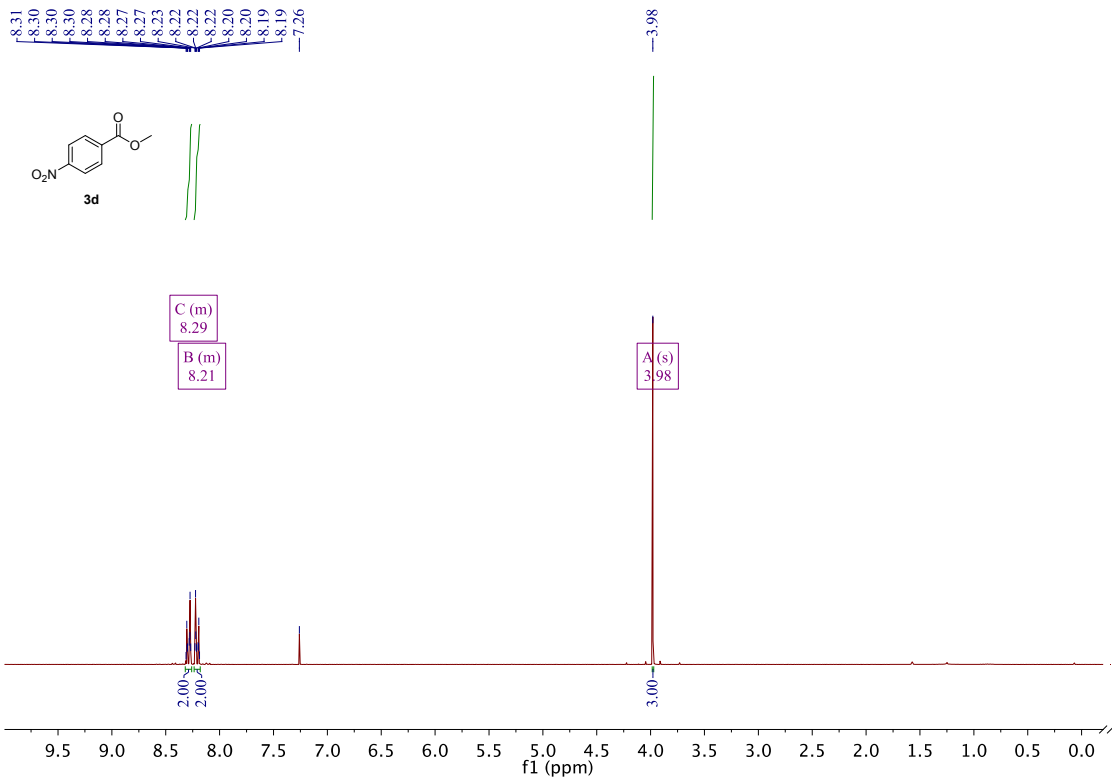
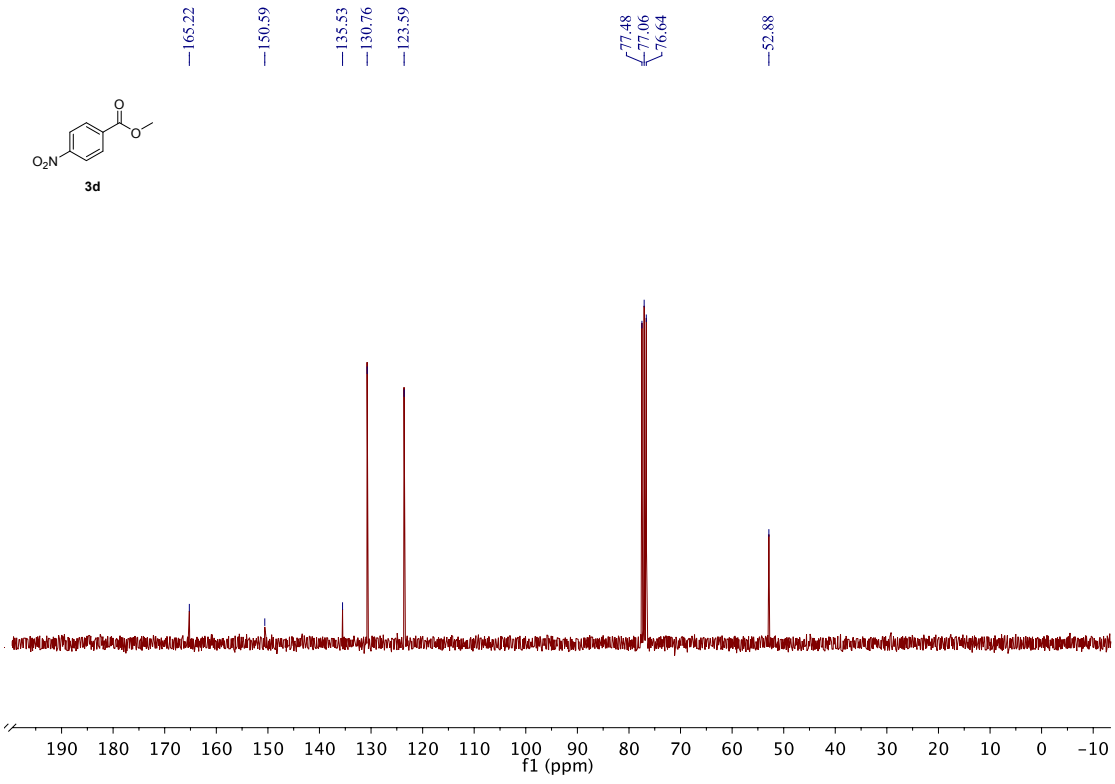
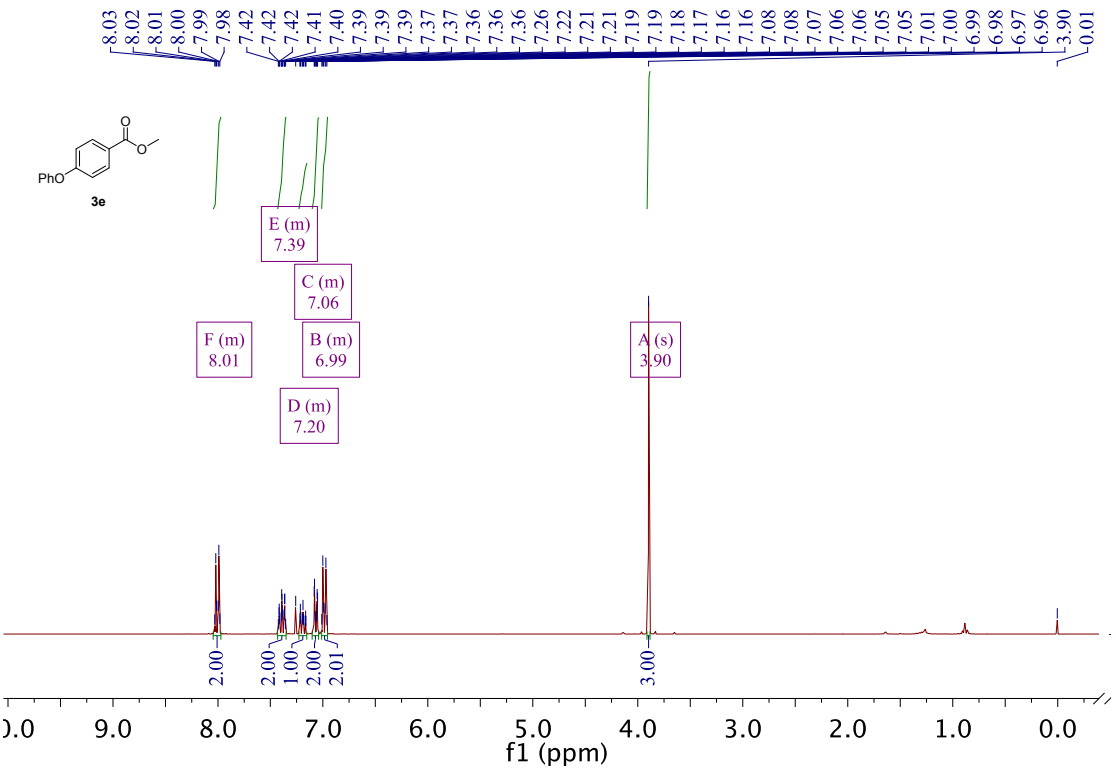
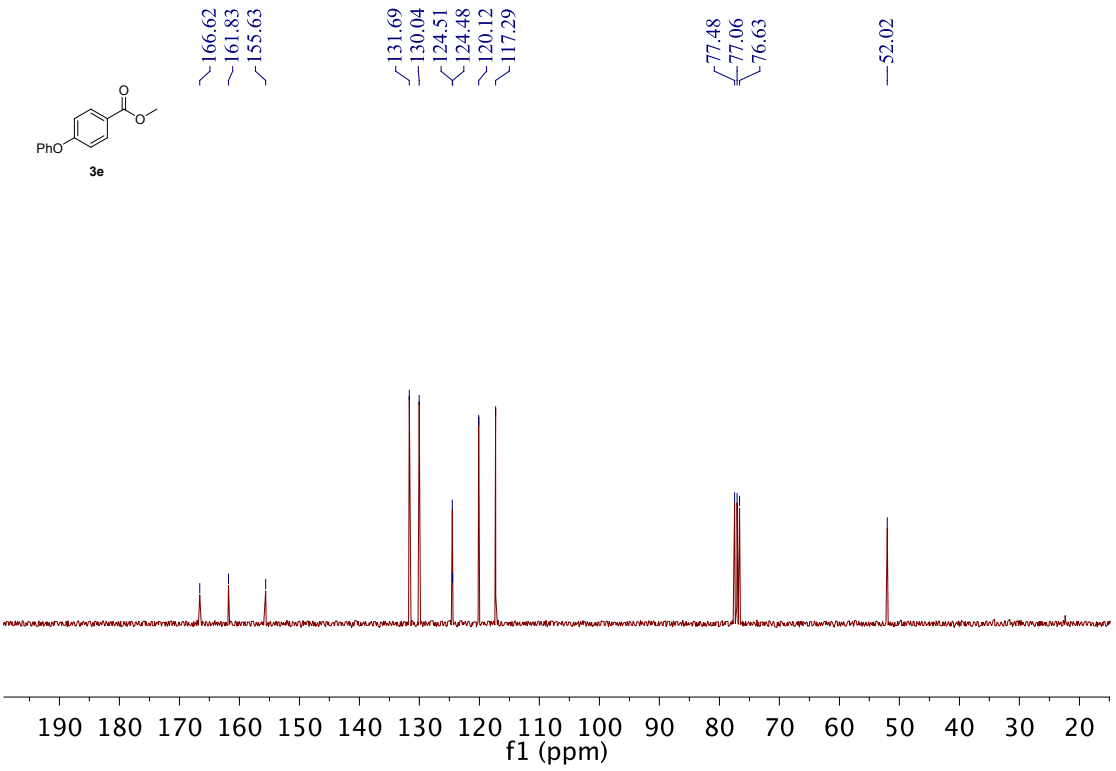
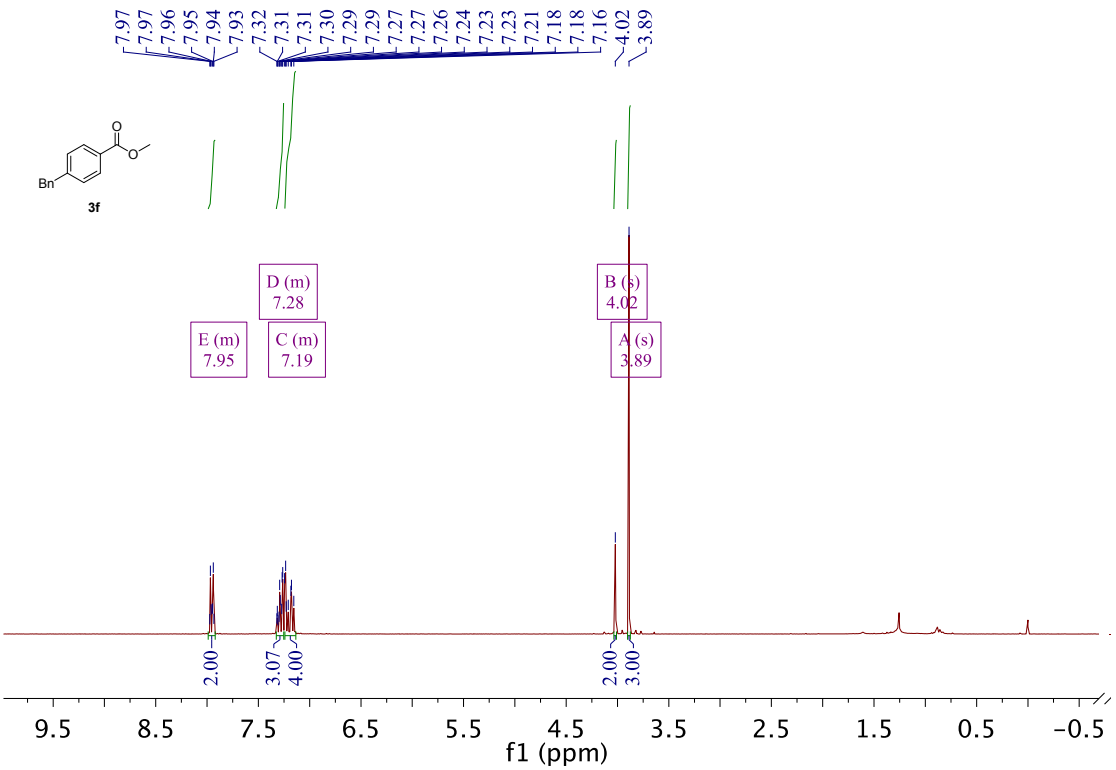
f1 (ppm)

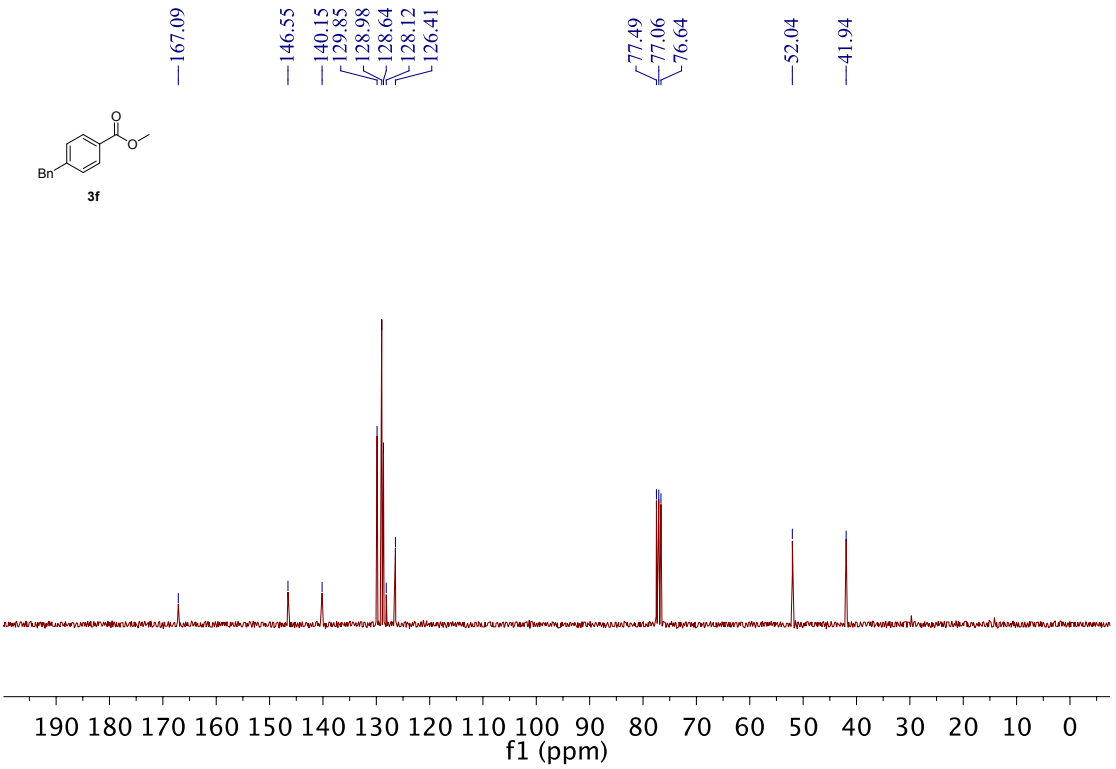

8.31
8.30
8.30
8.30
8.28
8.28
8.27
8.27
8.23
8.22
8.22
8.22
8.20
8.20
8.19
8.19
7.26
3.98
3d
C (m)
8.29
B (m)
8.21
A (s)
3.98
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

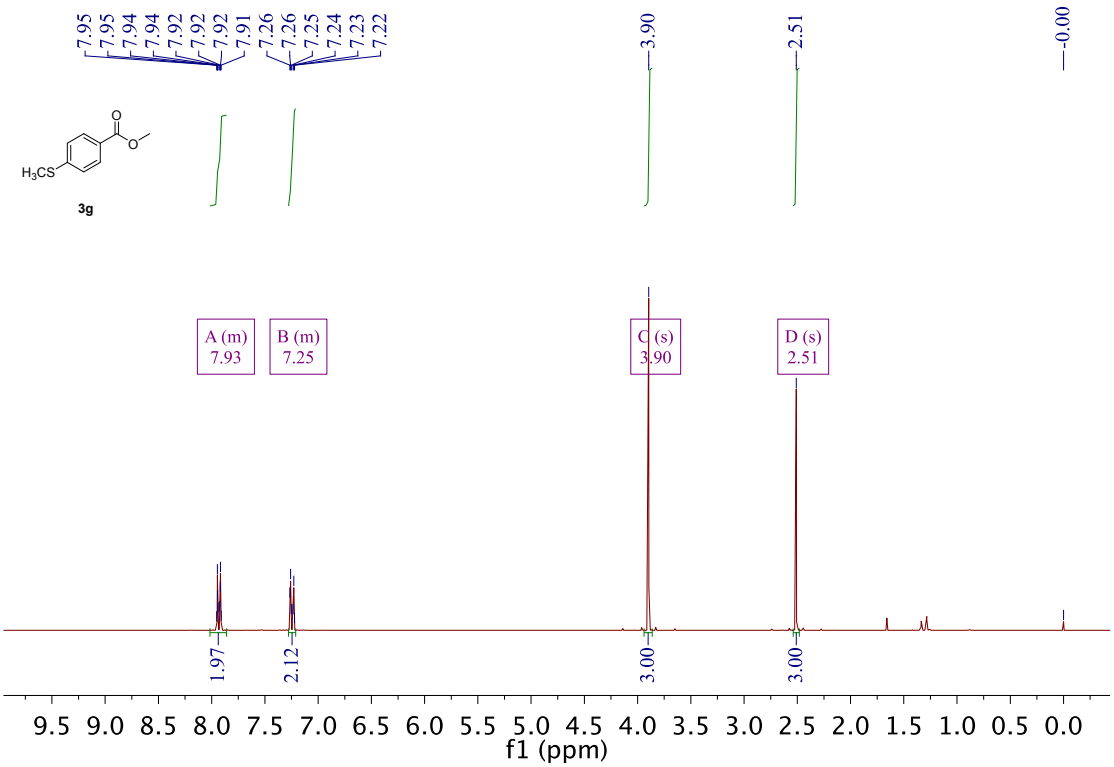

165.22
150.59
135.53
130.76
123.59
77.48
77.06
76.64
52.88
3d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

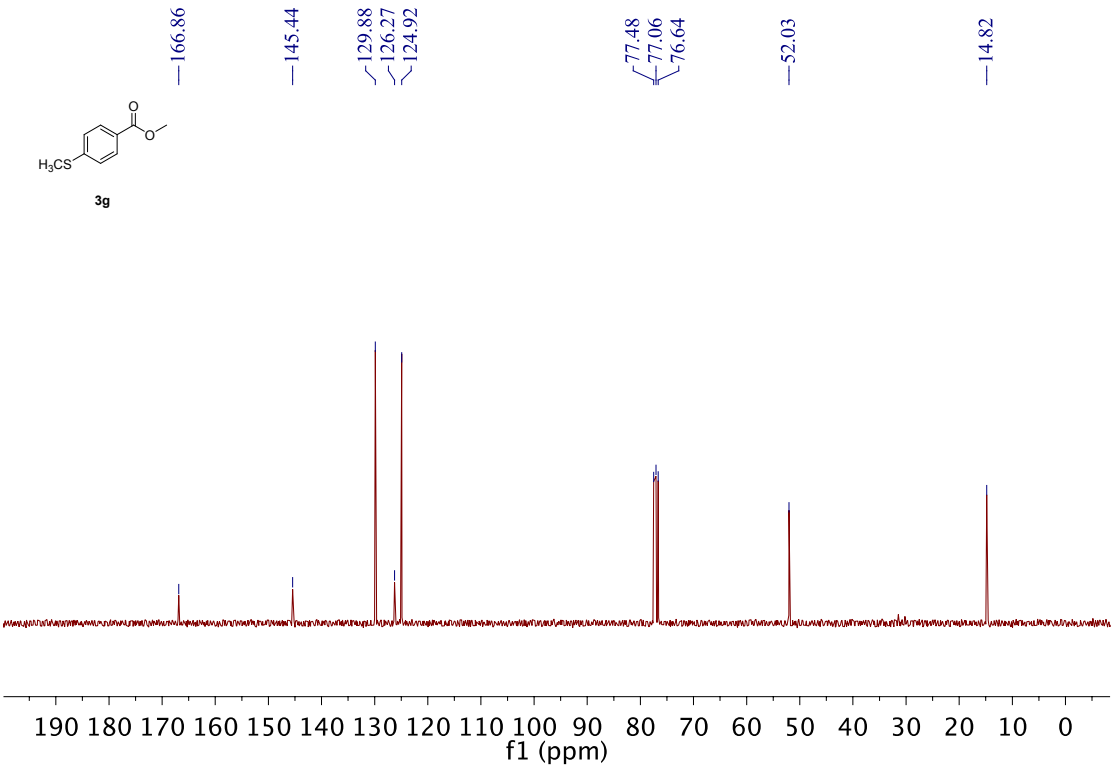

8.03 8.02 8.01 8.00 7.99 7.98 7.42 7.42 7.42 7.41 7.40 7.39 7.39 7.39 7.37 7.37 7.36 7.36 7.36 7.26 7.22 7.21 7.21 7.19 7.19 7.18 7.17 7.16 7.16 7.08 7.08 7.07 7.06 7.06 7.05 7.05 7.01 7.00 6.99 6.98 6.97 6.96 3.90 0.01
PhO
O
O
3e
E (m) 7.39
C (m) 7.06
F (m) 8.01
B (m) 6.99
A (s) 3.90
D (m) 7.20
2.00 2.00 1.00 2.00 2.01 3.00
0.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

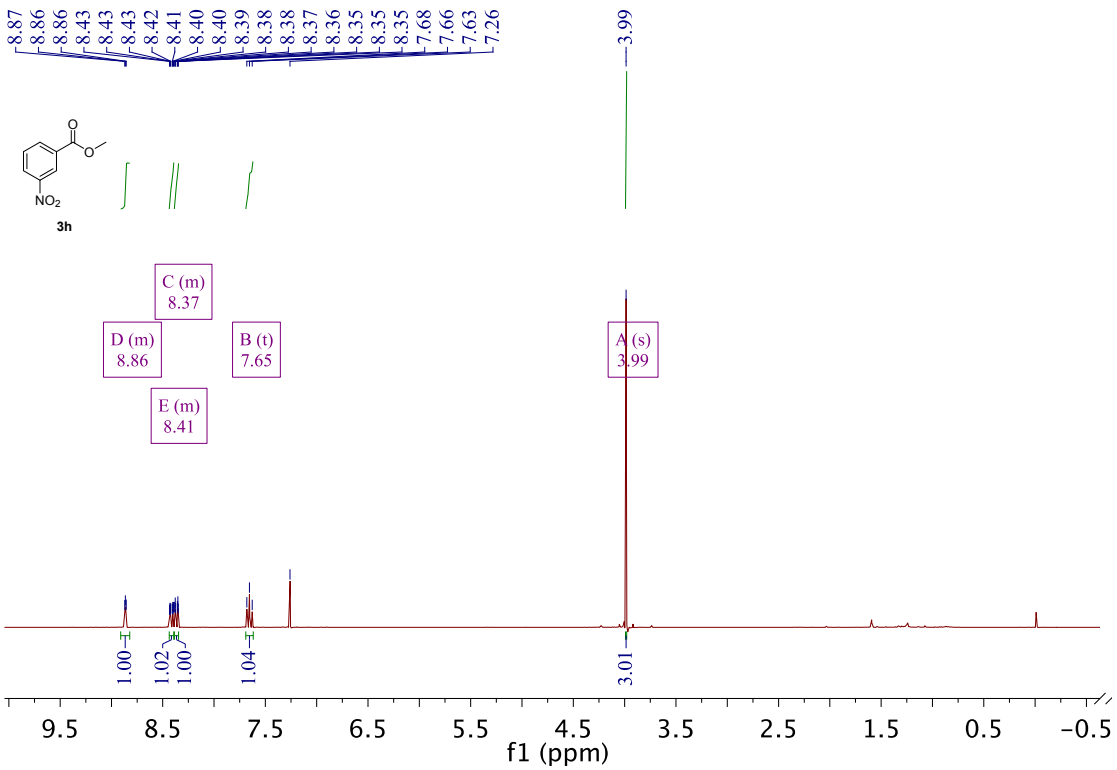

166.62 161.83 155.63 131.69 130.04 124.51 124.48 120.12 117.29 77.48 77.06 76.63 52.02
PhO
O
O
3e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

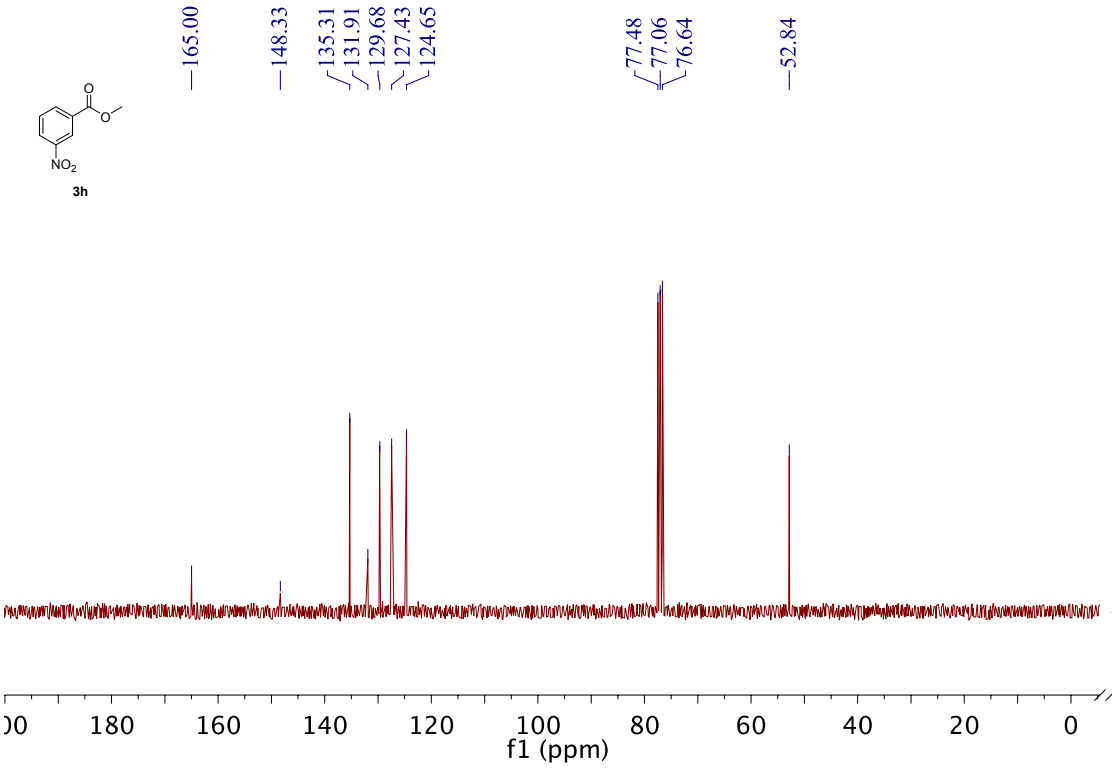

7.97
7.97
7.96
7.95
7.94
7.93
7.32
7.31
7.31
7.30
7.29
7.29
7.27
7.27
7.26
7.24
7.23
7.23
7.21
7.18
7.16
4.02
3.89
Bn
3f
D (m)
7.28
B (s)
4.02
E (m)
7.95
C (m)
7.19
A (s)
3.89
2.00
3.07
4.00
2.00
3.00
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

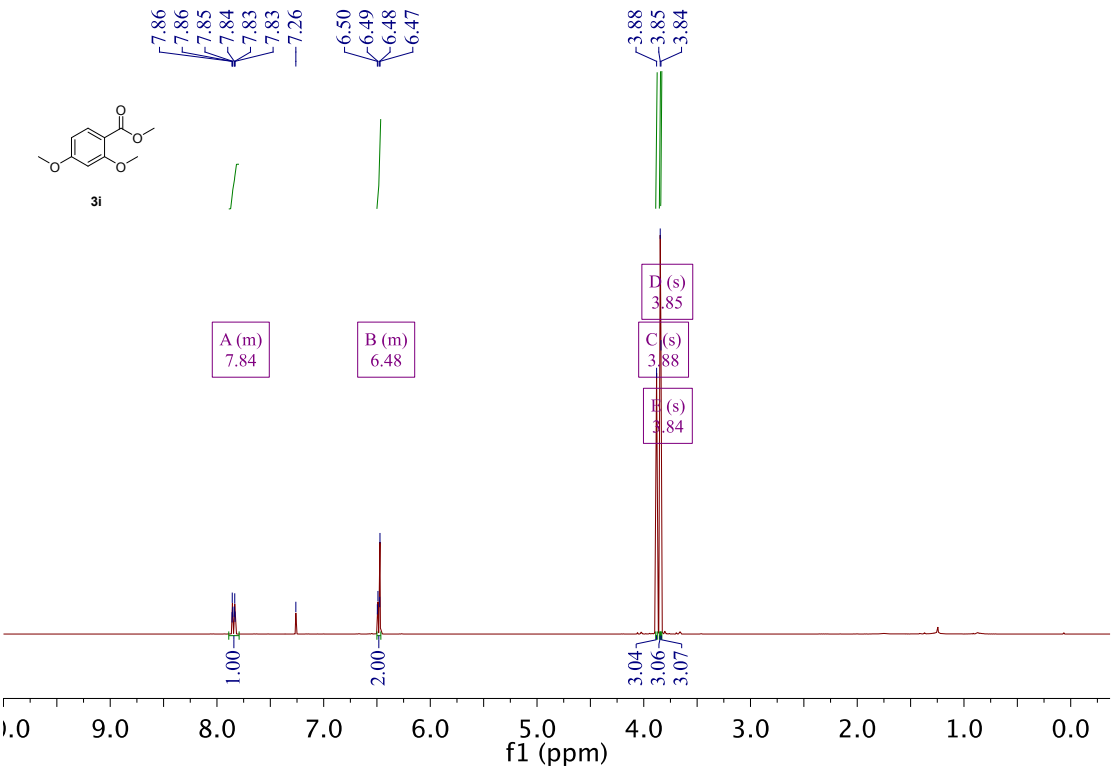

167.09
146.55
140.15
129.85
128.98
128.64
128.12
126.41
77.49
77.06
76.64
52.04
41.94
Bn
3f
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

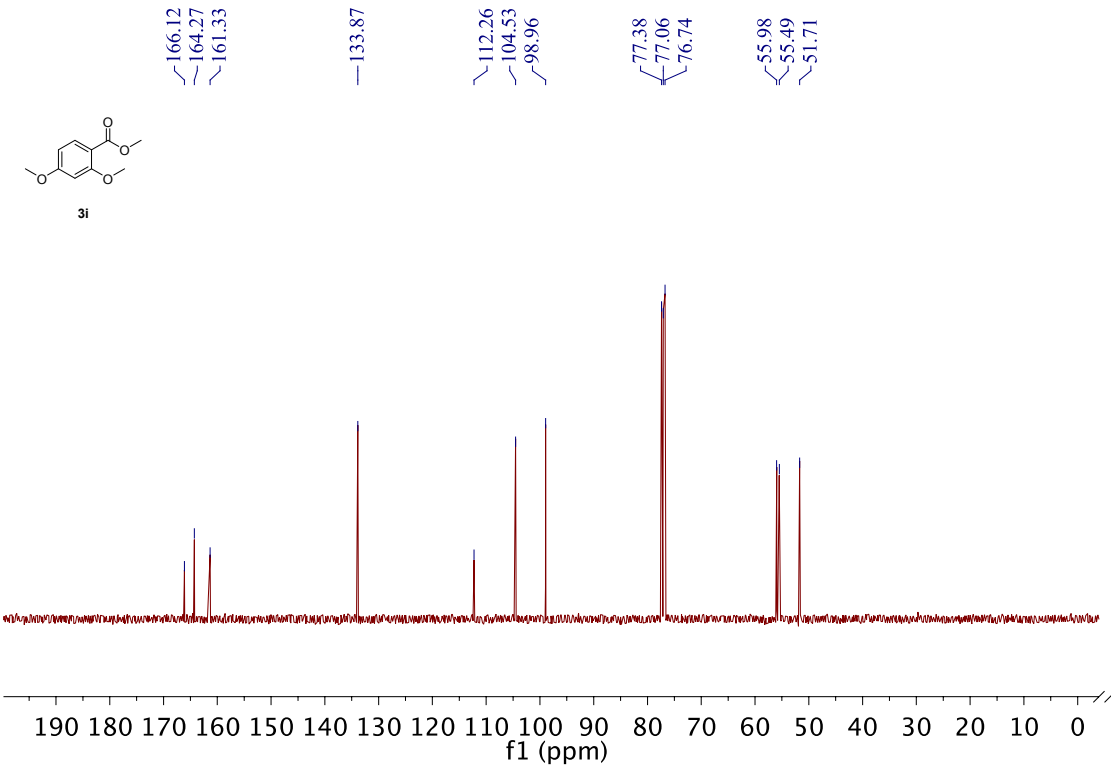

7.95
7.95
7.94
7.94
7.92
7.92
7.92
7.91
7.26
7.26
7.25
7.24
7.23
7.22
3.90
2.51
0.00
O
O
H3CS
3g
A (m)
7.93
B (m)
7.25
C (s)
3.90
D (s)
2.51
1.97
2.12
3.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

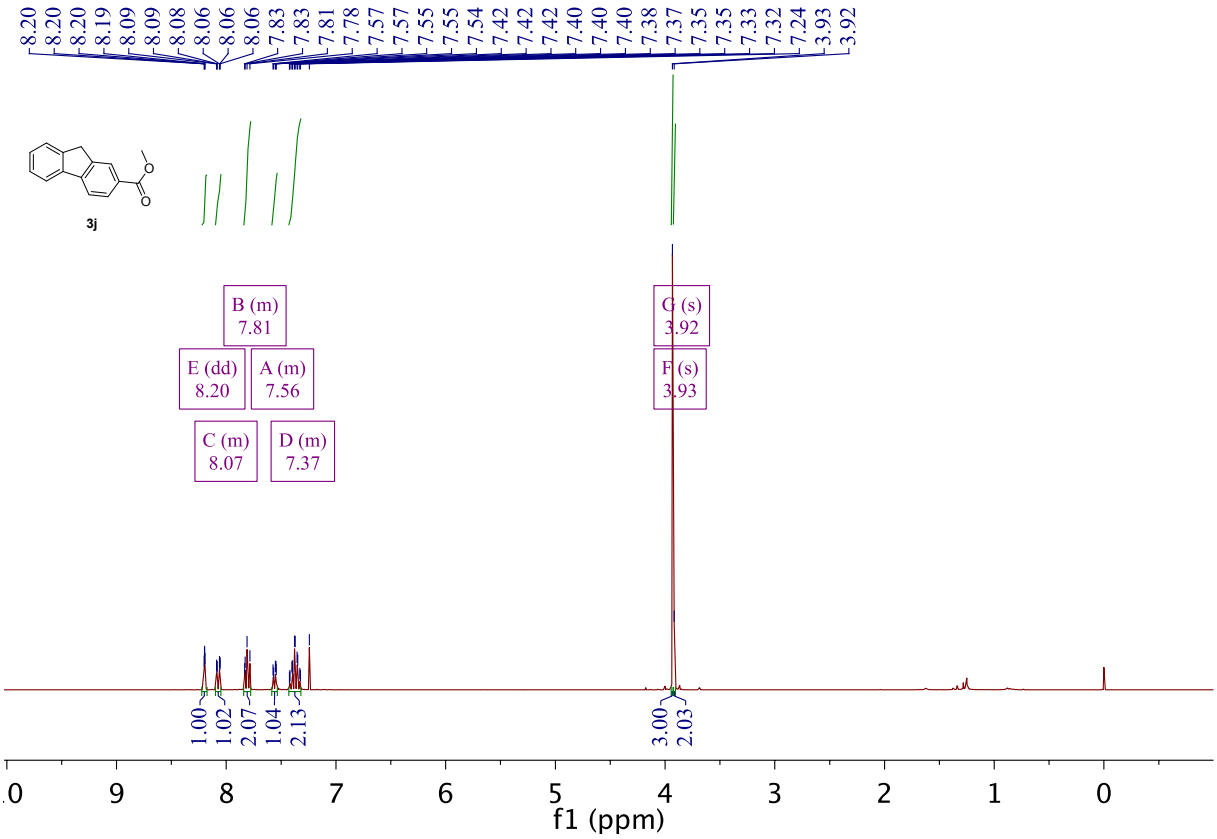

166.86
145.44
129.88
126.27
124.92
77.48
77.06
76.64
52.03
14.82
O
O
H3CS
3g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

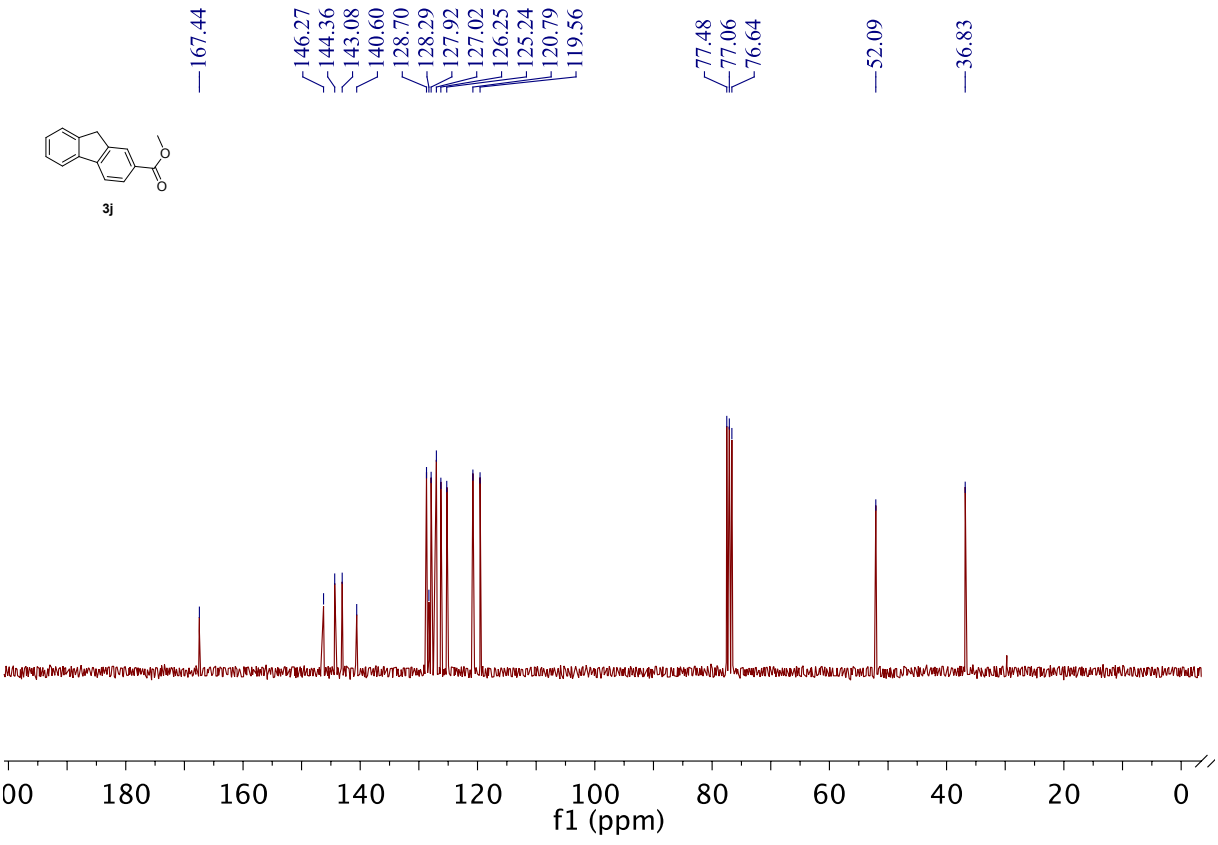

8.87
8.86
8.86
8.43
8.43
8.43
8.42
8.41
8.40
8.40
8.39
8.38
8.38
8.37
8.36
8.35
8.35
8.35
7.68
7.66
7.63
7.26
3.99
O
O
NO2
3h
C (m)
8.37
D (m)
8.86
B (t)
7.65
A (s)
3.99
E (m)
8.41
1.00
1.02
1.00
1.04
3.01
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


165.00
148.33
135.31
131.91
129.68
127.43
124.65
77.48
77.06
76.64
52.84
O
O
NO2
3h
00
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


7.86
7.86
7.85
7.84
7.83
7.83
7.26
6.50
6.49
6.48
6.47
3.88
3.85
3.84
3i
A (m)
7.84
B (m)
6.48
D (s)
3.85
C (s)
3.88
E (s)
3.84
1.00
2.00
3.04
3.06
3.07
f1 (ppm)


166.12
164.27
161.33
133.87
112.26
104.53
98.96
77.38
77.06
76.74
55.98
55.49
51.71
3i
f1 (ppm)


8.20
8.20
8.20
8.19
8.09
8.09
8.08
8.06
8.06
8.06
7.83
7.83
7.81
7.78
7.57
7.57
7.55
7.55
7.54
7.42
7.42
7.42
7.40
7.40
7.40
7.38
7.37
7.35
7.35
7.33
7.32
7.24
3.93
3.92
3j
B (m)
7.81
E (dd)
8.20
A (m)
7.56
C (m)
8.07
D (m)
7.37
G (s)
3.92
F (s)
3.93
1.00
1.02
2.07
1.04
2.13
3.00
2.03
f1 (ppm)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146.27
144.36
143.08
140.60
128.70
128.29
127.92
127.02
126.25
125.24
120.79
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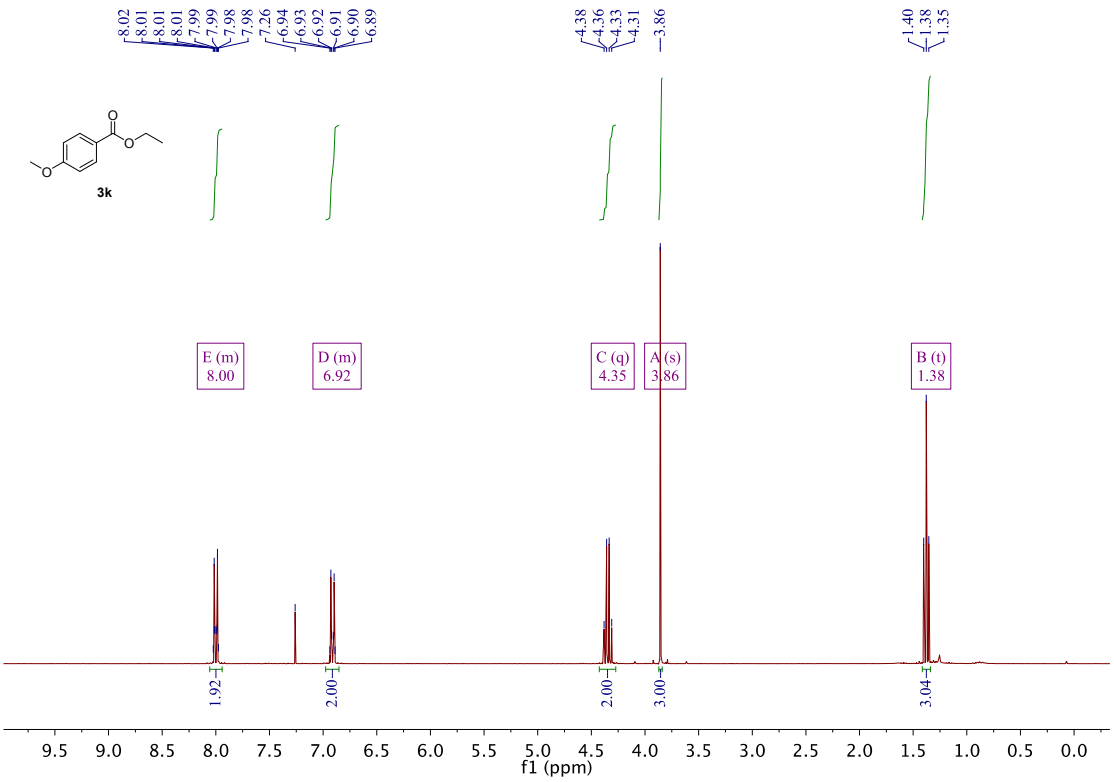
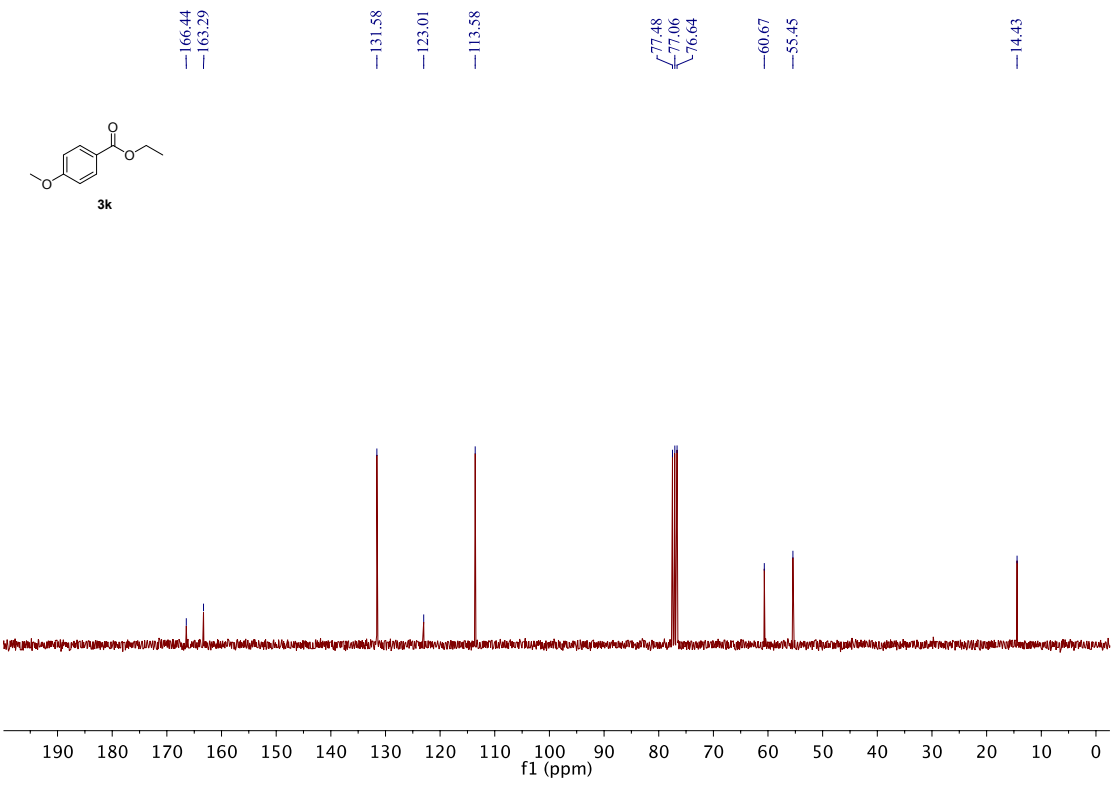
76.64
52.09
36.83
3j
f1 (ppm)


8.02
8.01
8.01
8.01
7.99
7.99
7.98
7.98
7.26
6.94
6.93
6.92
6.91
6.90
6.89
4.38
4.36
4.33
4.31
3.86
1.40
1.38
1.35
3k
E (m)
8.00
D (m)
6.92
C (q)
4.35
A (s)
3.86
B (t)
1.38
1.92
2.00
2.00
3.00
3.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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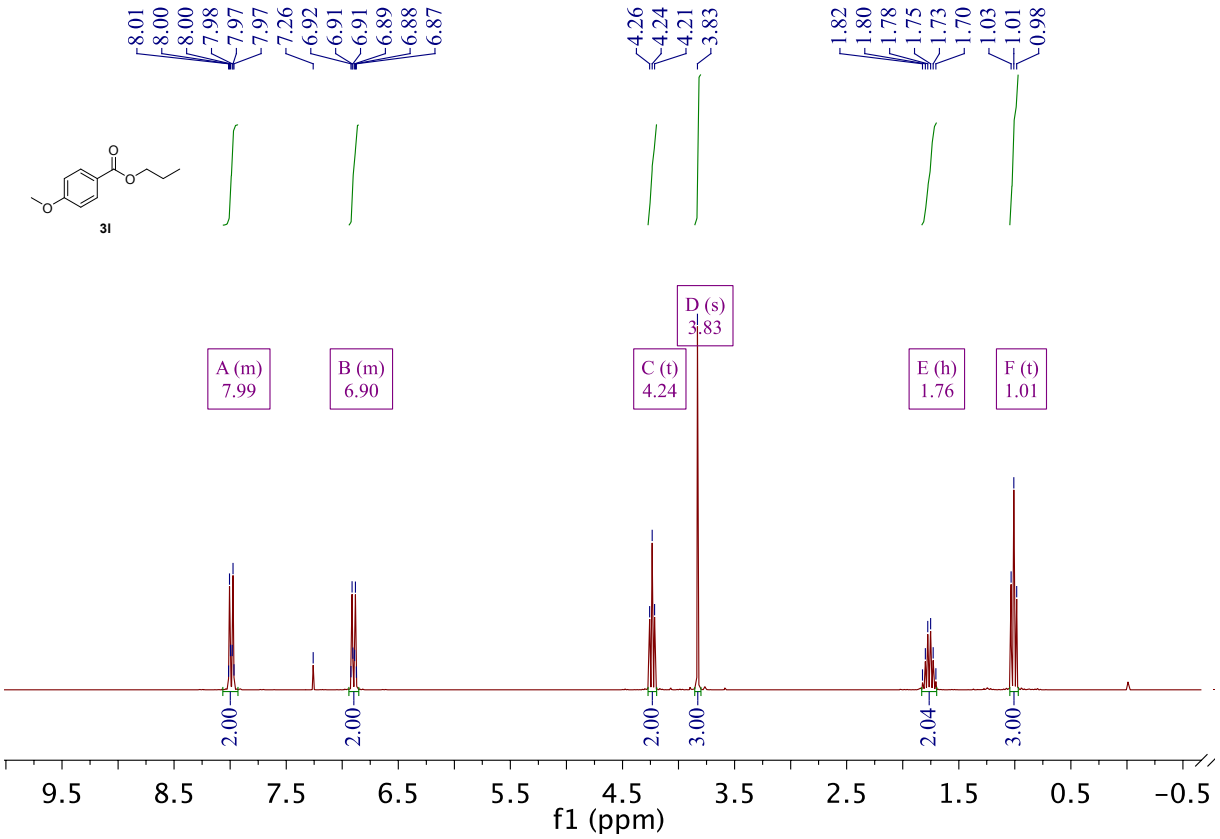
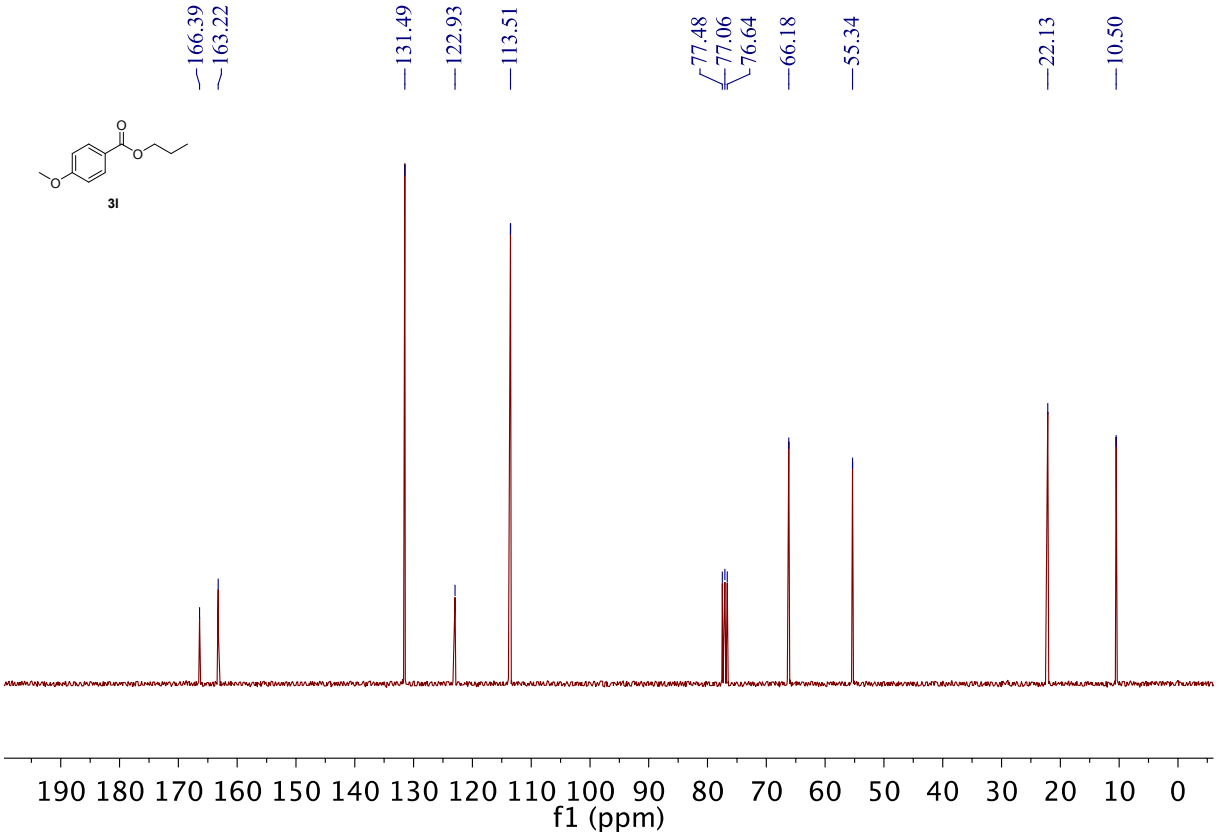
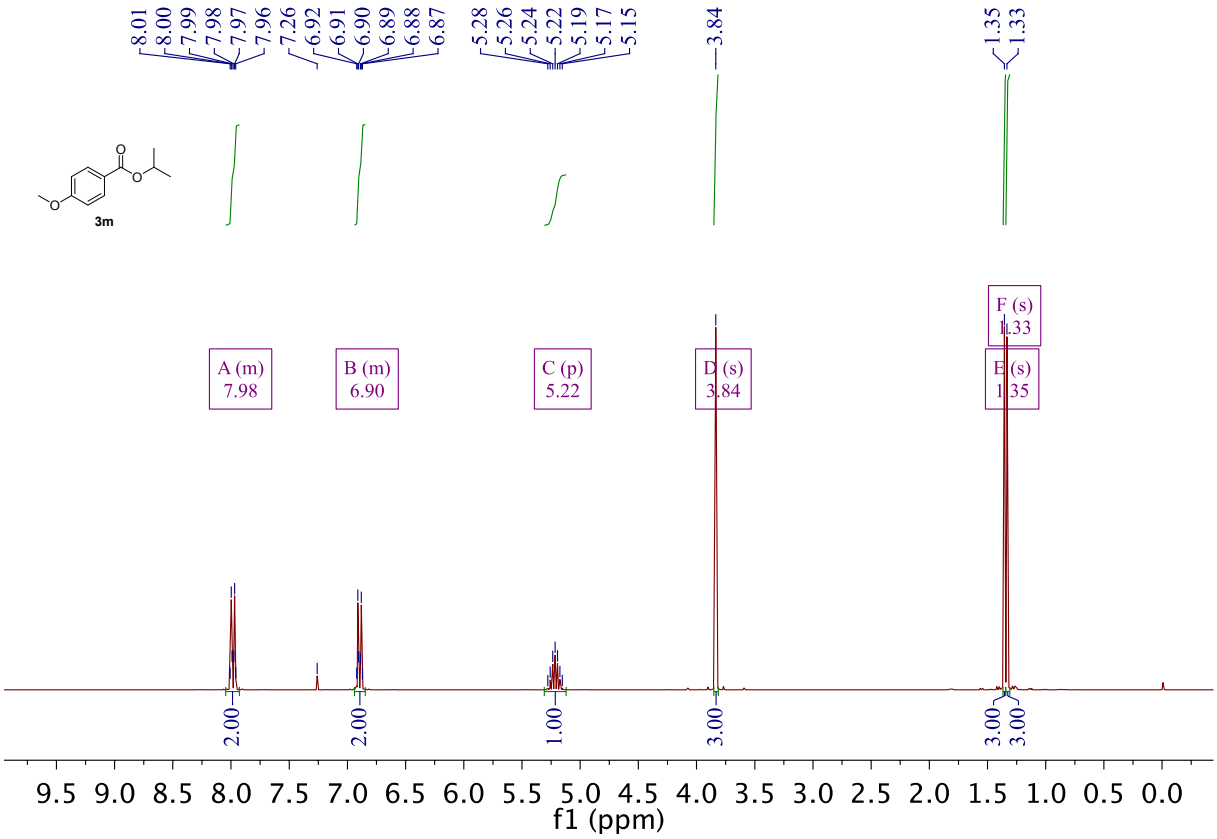
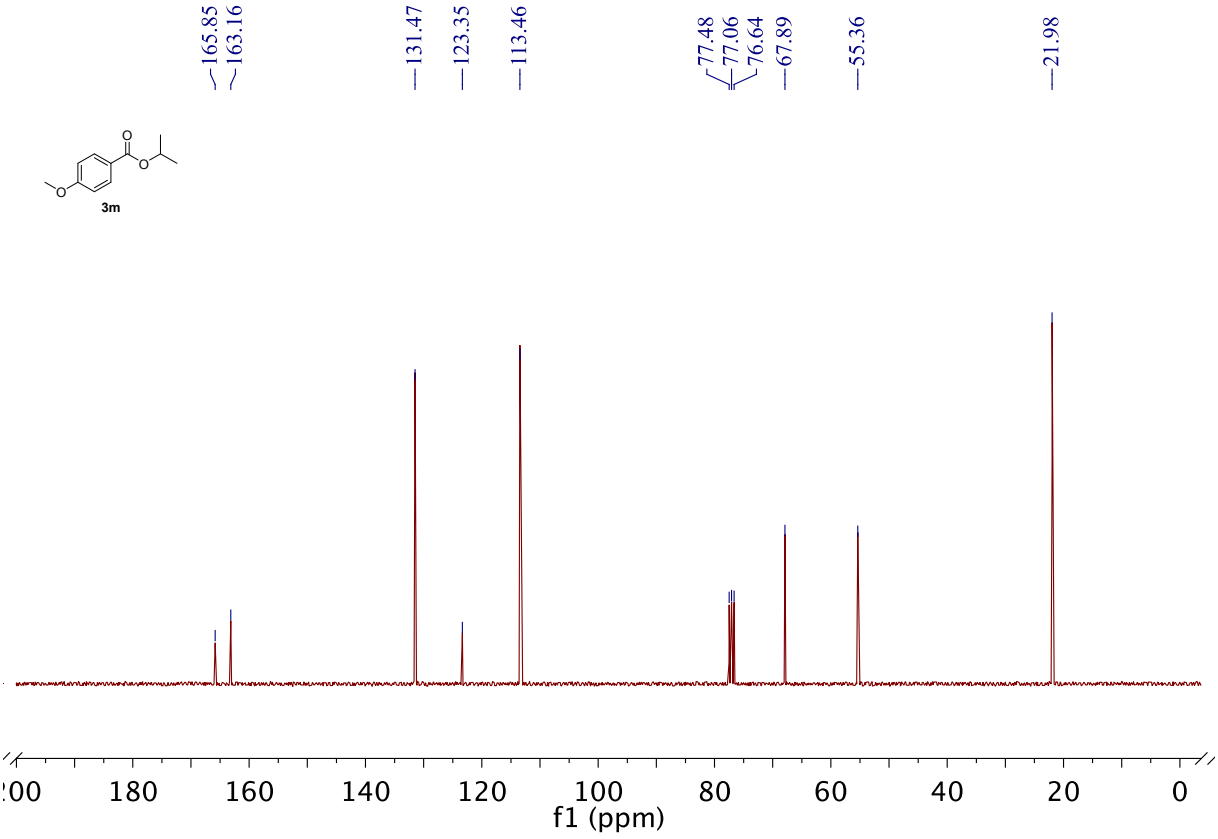
163.29
131.58
123.01
113.58
77.48
77.06
76.64
60.67
55.45
14.43
3k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.01
8.00
8.00
7.98
7.97
7.97
7.26
6.92
6.91
6.91
6.89
6.88
6.87
4.26
4.24
4.21
3.83
1.82
1.80
1.78
1.75
1.73
1.70
1.03
1.01
0.98
3l
A (m)
7.99
B (m)
6.90
C (t)
4.24
D (s)
3.83
E (h)
1.76
F (t)
1.01
2.00
2.00
2.00
3.00
2.04
3.00
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


166.39
163.22
131.49
122.93
113.51
77.48
77.06
76.64
66.18
55.34
22.13
10.50
3l
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.01
8.00
7.99
7.98
7.97
7.96
7.26
6.92
6.91
6.90
6.89
6.88
6.87
5.28
5.26
5.24
5.22
5.19
5.17
5.15
3.84
1.35
1.33
3m
A (m)
7.98
B (m)
6.90
C (p)
5.22
D (s)
3.84
F (s)
1.33
E (s)
1.35
2.00
2.00
1.00
3.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


165.85
163.16
131.47
123.35
113.46
77.48
77.06
76.64
67.89
55.36
21.98
3m
180
160
140
120
100
80
60
40
20
0
f1 (ppm)