**Electronic supplementary materials** *Mendeleev Commun.*, 2022, **32**, 256–257

# Rhodium-catalyzed regioselective cross-coupling of styrene oxides with arylboronic acids in aqueous γ-valerolactone

**Jie Zhang, Yuhong Chen, Xiaohui Luo and Zhengru Wen**

**Contents**



## General Experimental

Unless otherwise noted, all chemicals were purchased from commercial suppliers (Aladdin) and used without further purification. $^{1}H$ NMR and $^{13}C$ NMR spectra were recorded at ambient temperature on a Bruker AVANCE III 500 spectrometer ($^{1}H$: 500 MHz; $^{13}C$: 125 MHz). Chemical shifts are reported in δ units, parts per million (ppm), and were referenced to $CDCl_3$ (7.26 or 77.0 ppm) as the internal standard. The coupling constants *J* are given in Hz. Column chromatography was performed using EM Silica gel 60 (300-400 mesh). High-resolution mass spectrometry (HRMS) was performed on an Agilent Q-TOF 6540 MS instrument with an ESI source.

## Experimental Procedures, Spectral and Analytical data

### General procedure for the synthesis of 3

A sealed tube equipped with a magnetic stirring bar was charged with styrene oxide **1** (0.2 mmol), arylboronic acid **2** (0.6 mmol, 3.0 equiv), $Rh(PPh_3)_3Cl$ (0.5 mol%), $Na_2CO_3$ (0.2 mmol, 1.0 equiv) and GVL/$H_2O$ (1:1, 2 ml) under $N_2$. The mixture was then heated to 120 °C and stirred for 12 h. Upon reaction completion, the resulting solution was quenched with water and extracted with ethyl acetate. The collected organic extracts were dried over $Na_2SO_4$. The solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography using petroleum ether/ethyl acetate (8:1) as eluent to afford product **3**.

### Characterization data of product 3

*1,2-Diphenylethan-1-ol* (**3a**) [S1]. Colourless oil (34 mg, 85%). $^{1}H$ NMR (500 MHz, $CDCl_3$) δ 7.38-7.26 (m, 7H), 7.25-7.15 (m, 3H), 4.90 (dd, *J* = 8.6, 4.6 Hz, 1H), 3.08-2.97 (m, 2H), 1.98 (s, 1H). $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 143.9, 138.2, 129.5, 128.5, 128.4, 127.6, 126.6, 126.0, 75.4, 46.2.

*1-Phenyl-2-(o-tolyl)ethan-1-ol* (**3b**) [S1]. Colourless oil (30 mg, 71%). $^{1}H$ NMR (500 MHz, $CDCl_3$) δ 7.26-7.34 (m, 5H), 7.16-7.12(m, 4H), 4.86 (dd, *J* = 8.0, 5.5 Hz, 1H), 3.09-2.94 (m, 2H), 2.27 (s, 3H), 2.04 (s, 1H). $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 144.2, 136.8, 136.4, 130.5, 130.3, 128.4, 127.6, 126.8, 126.0, 125.8, 74.5, 43.4, 19.6.

*1-Phenyl-2-(m-tolyl)ethan-1-ol* (**3c**) [S2]. Colourless oil (28 mg, 67%). $^{1}H$ NMR (500 MHz, $CDCl_3$) δ 7.37-7.17 (m, 6H), 7.05-6.98 (m, 3H), 4.86 (dd, *J* = 9.0, 4.5 Hz, 1H), 2.96 (m, 2H), 2.32 (s, 3H), 2.02 (s, 1H).

$^{13}$C NMR (125 MHz, $CDCl_3$) δ 144.0, 138.2, 138.0, 130.3, 128.5, 128.4, 127.6, 127.4, 126.5, 125.9, 75.3, 46.2, 21.4.

*1-Phenyl-2-(p-tolyl)ethan-1-ol* (**3d**) [S3]. Colourless oil (32 mg, 76%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.35-7.21 (m, 5H), 7.09-7.06 (m, 4H), 4.82-4.85 (m, 1H), 3.09-2.83 (m, 2H), 2.31 (s, 3H), 2.01 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 144.0, 136.2, 135.0, 129.4, 129.2, 128.4, 127.6, 126.0, 75.4, 45.7, 21.1.

*2-(4-tert-Butylphenyl)-1-phenylethan-1-ol* (**3e**) [S2]. Colourless oil (31 mg, 62%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.39-7.22 (m, 7H), 7.15 (d, *J* = 8.0 Hz, 2H), 4.87 (dd, *J* = 9.0, 4.0 Hz, 1H), 2.96 (ddd, *J* = 43.5, 13.5, 4.0 Hz, 2H), 2.01 (s, 1H), 1.31 (s, 9H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 149.5, 144.1, 135.1, 129.2, 128.4, 127.6, 125.9, 125.5, 75.3, 45.7, 34.5, 31.4.

*2-([1,1'-Biphenyl]-4-yl)-1-phenylethan-1-ol* (**3f**) [S2]. Light yellow amorphous solid (35 mg, 64%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.57-7.49 (m, 4H), 7.43-7.18 (m, 10H), 4.91 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.06-3.00 (m, 2H), 2.09 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 142.9, 140.8, 140.5, 138.0, 129.5, 128.8, 128.5, 127.3, 127.1, 127.1, 126.6, 126.4, 75.1, 46.0.

*2-(4-Chlorophenyl)-1-phenylethan-1-ol* (**3g**) [S3]. Colourless oil (32 mg, 69%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.34-7.27 (m, 5H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 4.83 (t, *J* = 6.5 Hz, 1H), 2.97 (d, *J* = 6.5 Hz, 2H), 2.04 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 143.6, 136.5, 132.4, 130.9, 128.5, 128.5, 127.8, 125.9, 75.3, 45.2.

*1-Phenyl-2-(4-trifluoromethylphenyl)ethan-1-ol* (**3h**) [S3]. Colourless oil (32 mg, 60%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0Hz, 2H), 7.39-7.17 (m, 7H), 4.89-4.86(m, 1H), 3.14-2.95 (m, 2H), 2.08 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 143.5, 142.3, 123.0, 129.0 (q, $J_{C-F}$=32.0 Hz), 128.6, 127.9, 126.0, 125.2 (q, $J_{C-F}$ = 3.7 Hz), 124.3 (q, $J_{C-F}$ = 270.0 Hz), 75.2, 45.6. $^{19}$F NMR (376 MHz, $CDCl_3$) δ -62.8.

*2-Phenyl-1-(o-tolyl)ethan-1-ol* (**3i**) [S1]. Colourless oil (31 mg, 73%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.52(d, *J* =8.0 Hz, 1H), 7.09-7.30(m, 8H), 5.08(q, *J* =4.0 Hz, 1H), 3.00-2.88 (m, 2H), 2.25(s, 3H), 1.95 (s,1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 142.1, 138.4, 134.4, 130.3, 129.5, 128.5, 127.3, 126.6, 126.4, 125.3, 71.8, 45.1, 19.0.

*2-Phenyl-1-(m-tolyl)ethan-1-ol* (**3j**) [S1]. Colourless oil (32 mg, 75%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.0 Hz, 2H), 7.26-7.17 (m, 5H), 7.14 (d, *J* = 7.5 Hz, 1H), 7.09 (d, *J* = 7.0 Hz, 1H), 4.85 (q, *J* = 4.5 Hz 1H), 3.05-2.91 (m, 2H), 2.35 (s, 3H), 1.97 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 143.9, 138.6, 138.1, 129.5, 128.5, 128.4, 128.3, 126.6, 123.0, 75.4, 46.1, 21.5.

*2-Phenyl-1-(p-tolyl)ethan-1-ol* (**3k**) [S1]. Light yellow oil (30 mg, 71%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.30-7.13 (m, 9H), 4.82-4.85 (m, 1H), 3.02-2.94 (m, 2H), 2.34 (s, 3H), 1.97 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 140.9, 138.2, 137.2, 129.5, 129.1, 128.5, 126.5, 125.9, 75.2, 46.0, 21.1.

*1-(4-tert-Butylphenyl)-2-phenylethan-1-ol* (**3l**) [S1]. Colourless oil (36 mg, 70%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.37(d, *J* = 8.0 Hz, 2H), 7.31-7.28 (m, 4H), 7.24-7.21 (m, 3H), 4.86-4.84 (m, 1H), 3.05-2.94 (m, 2H), 1.96 (s, 1H), 1.32 (s, 9H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 150.6, 140.9, 138.4, 129.5, 128.5, 126.5, 125.6, 125.3, 75.1, 45.9, 34.5, 31.4.

*1-(4-Chlorophenyl)-2-phenylethan-1-ol* (**3m**) [S1]. Light yellow amorphous solid (30 mg, 64%). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.31-7.28 (m, 4H), 7.25-7.22 (m, 3H), 7.16-7.15 (m, 2H), 4.85 (dd, *J* = 8.5, 5.5 Hz, 1H), 3.01-2.91 (m, 2H), 2.05 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 142.3, 137.5, 133.2, 129.5, 128.6, 128.5, 127.3, 126.8, 74.6, 46.1.

*1-(4-Methoxyphenyl)-2-phenylethan-1-ol* (**3n**) [S1]. Colourless oil (31 mg, 68%). $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.30-7.15 (m, 7H), 6.86 (d, *J* = 8.5 Hz, 2H), 4.82 (t, *J* = 7.0 Hz, 1H), 3.79 (s, 3H), 2.99 (d, *J* = 6.0Hz, 2H), 1.99 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 159.1, 138.2, 136.1, 129.5, 128.5, 127.2, 126.5, 113.8, 75.0, 55.3, 46.0.

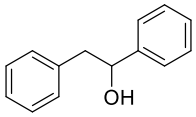
*1-(4-Methylthiophenyl)-2-phenylethan-1-ol* (**3o**). Light yellow oil (30 mg, 62%). $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.30-7.20 (m, 7H), 7.16 (d, *J* = 7.5 Hz, 2H), 4.82 (t, *J* = 6.0 Hz, 1H), 3.07 (ddd, *J* = 28.0, 13.5, 5.0Hz, 2H), 2.46 (s, 3H), 2.04 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 140.8, 137.9, 137.6, 129.5, 128.5, 126.7, 126.6, 126.5, 74.9, 46.0, 16.0. HRMS m/z (ESI) calcd for $C_{15}H_{16}NaOS$ $(M+Na)^+$ 267.0814, found 267.0826.

*1-(Naphthalen-2-yl)-2-phenylethan-1-ol* (**3p**) [S1]. White amorphous solid (37 mg, 74%). $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.82-7.74(m, 4H), 7.46-7.43 (m, 3H), 7.17-7.28 (m, 5H), 5.00 (dd, *J* = 8.0, 5.0 Hz, 1H), 3.12-3.01 (m, 2H), 2.17 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 141.3, 138.1, 133.4, 133.1, 129.6, 128.6, 128.2, 128.0, 127.7, 126.7, 126.2, 125.9, 124.7, 124.2, 75.4, 46.0.

## References


[S1] C. Zhou, Z. Wang, *Synthesis* 2005, **10**, 1649.
[S2] D. K. Nielsen, A. G. Doyle, *Angew. Chem., Int. Ed.*, 2011, **50**, 6056.
[S3] A. Potrząsaj, M. Musiejuk, W. Chaładaj, M. Giedyk, D. Gryko, *J. Am. Chem. Soc.*, 2021, **143**, 9368.

**Copies of $^{1}H$ and $^{13}C$ NMR spectra**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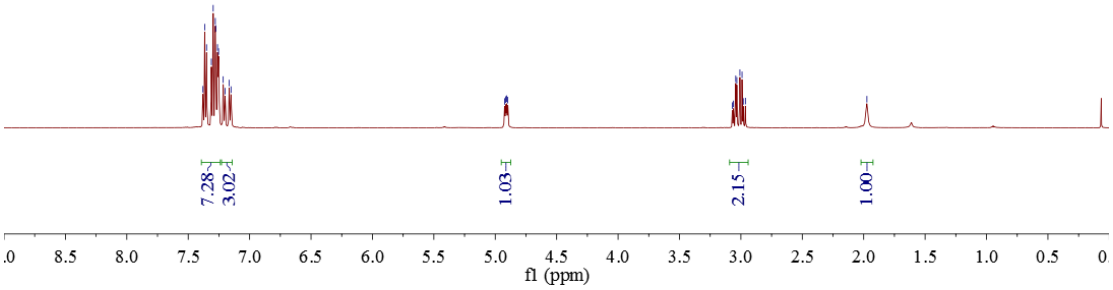



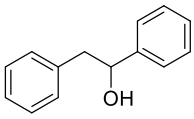


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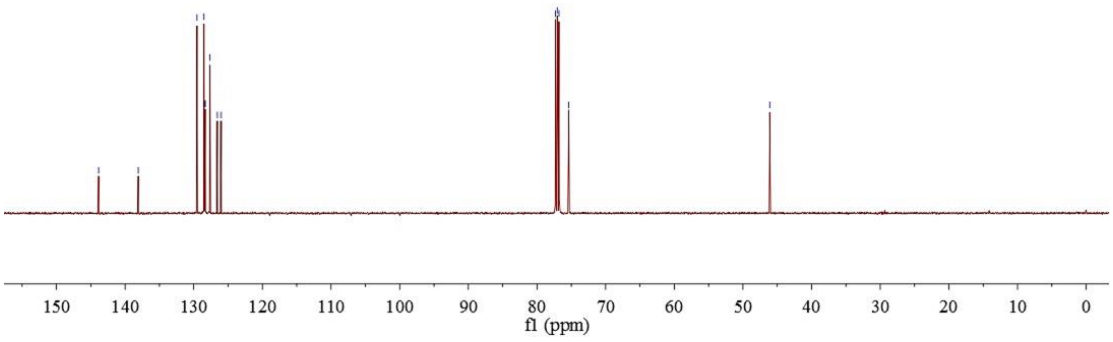

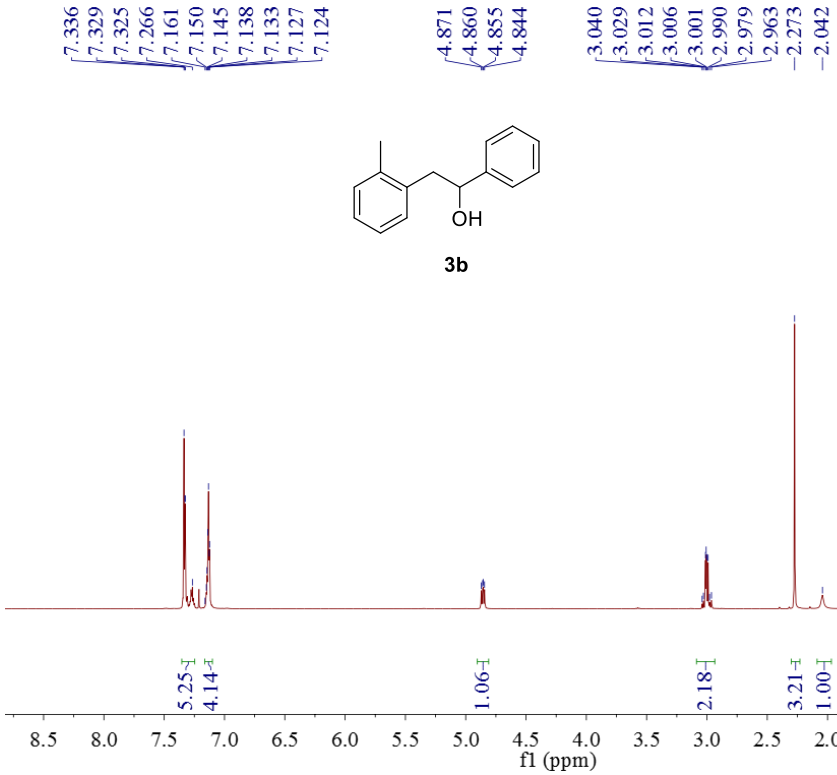
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2.042
OH
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4.14
1.06
2.18
3.21
1.00
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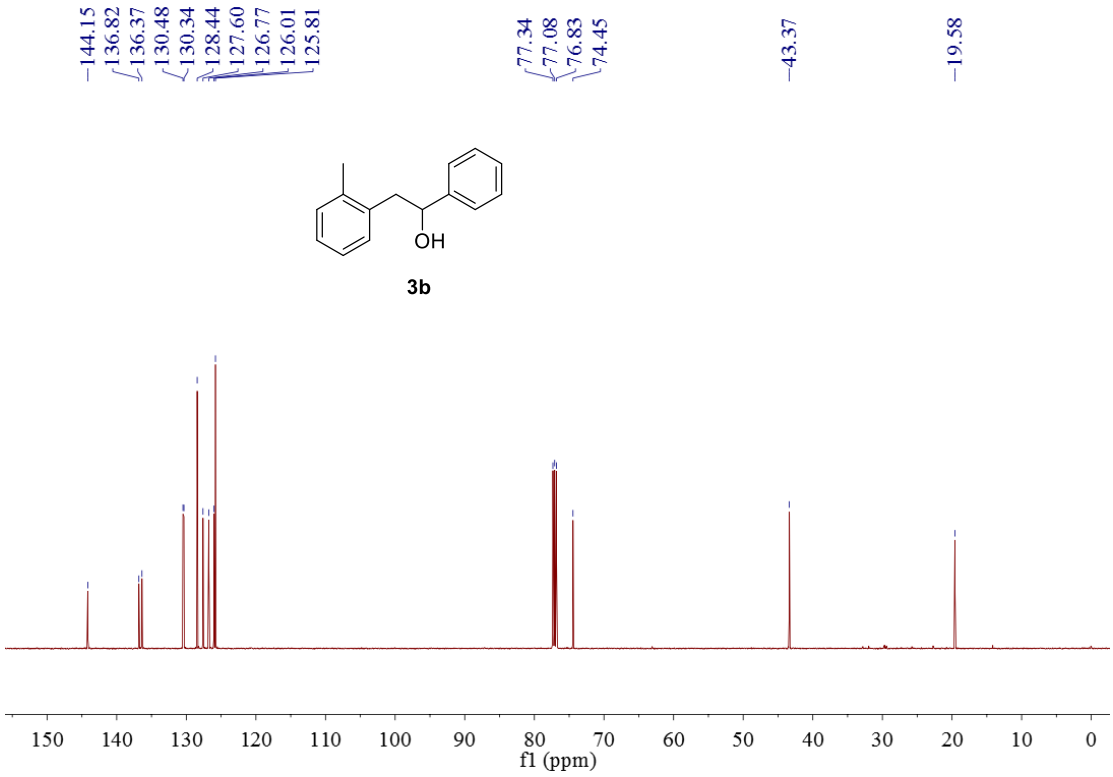
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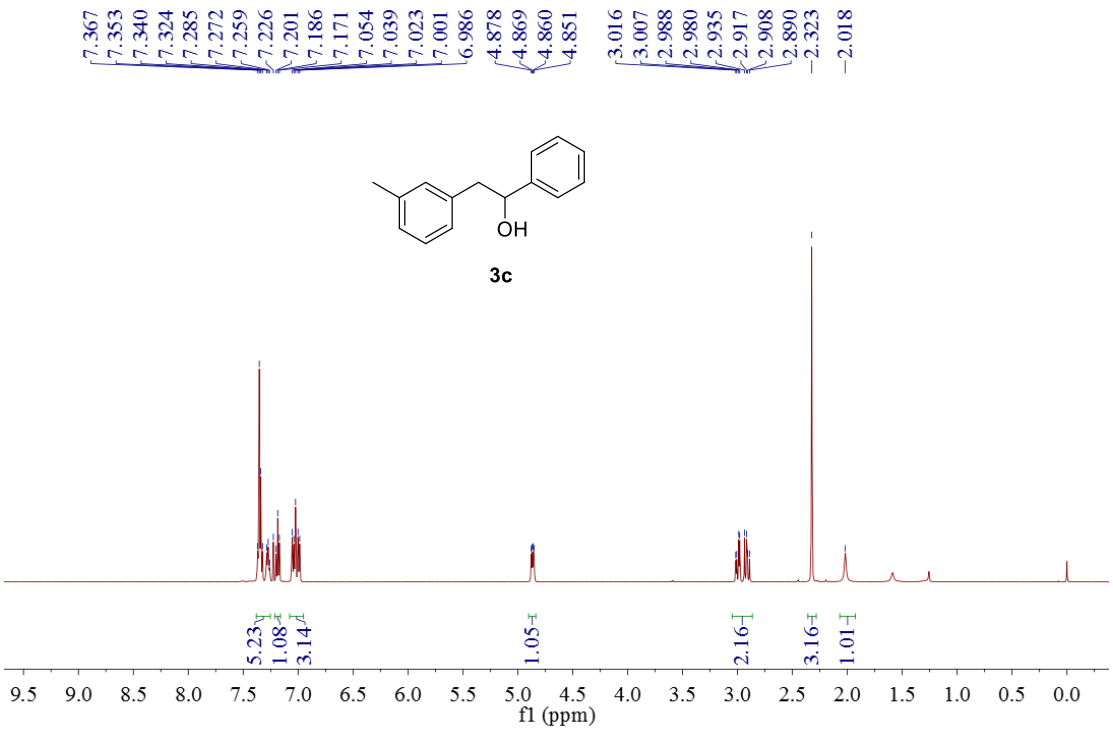
f1 (ppm)

**3b**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2.917
2.908
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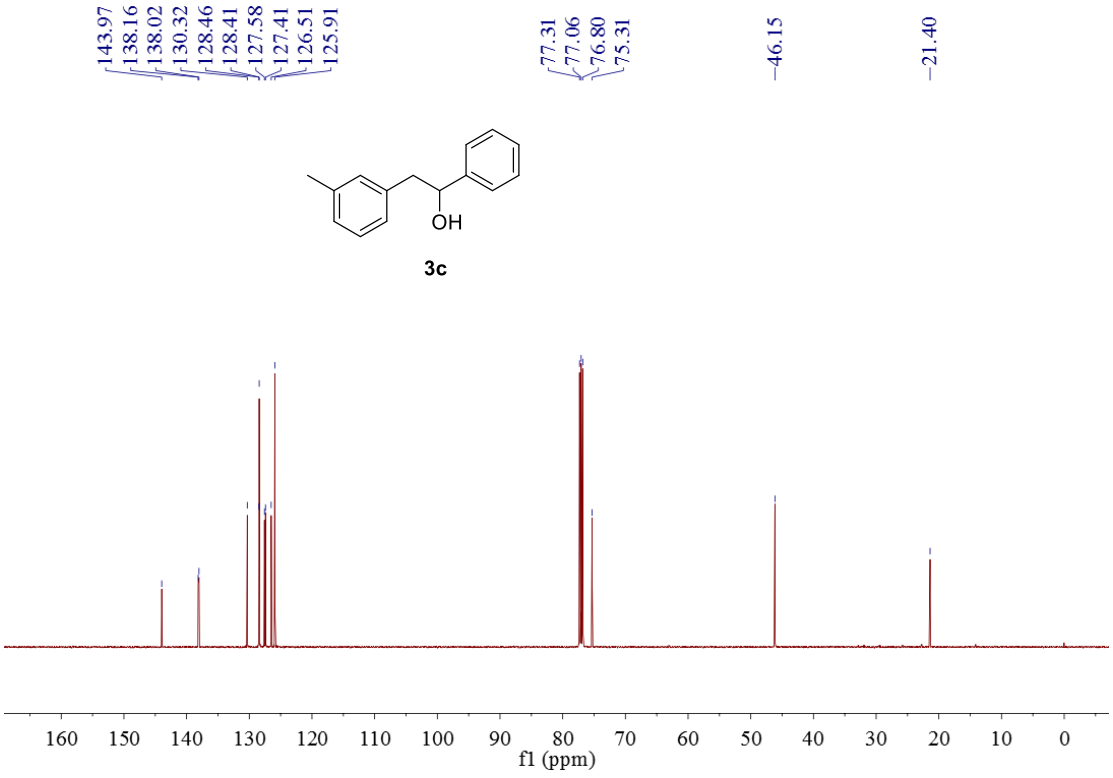
5.23
1.08
3.14
1.05
2.16
3.16
1.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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46.15
21.40
OH
3c
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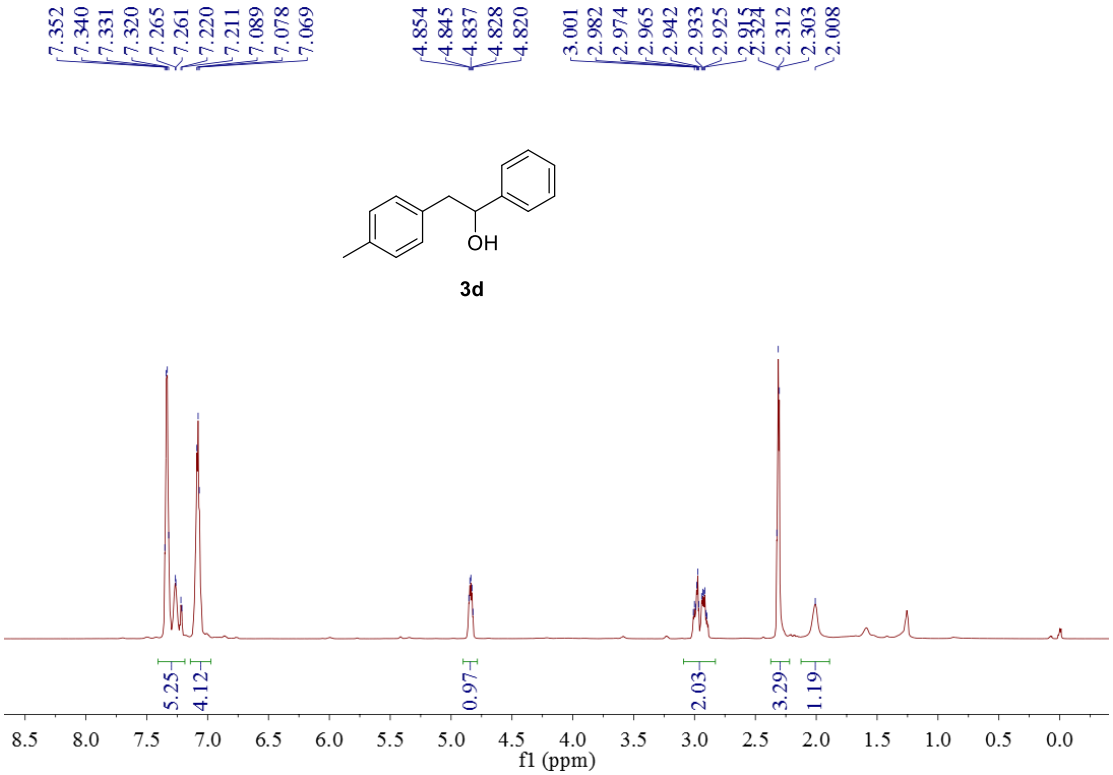
f1 (ppm)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f1 (ppm)

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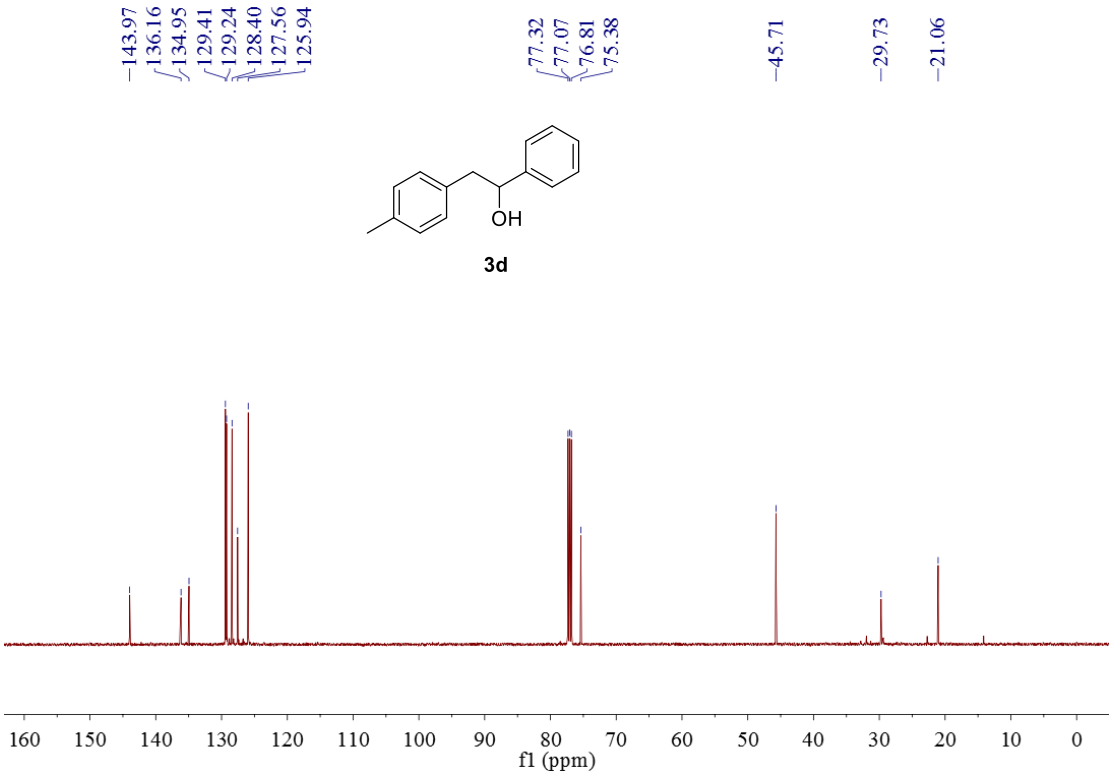
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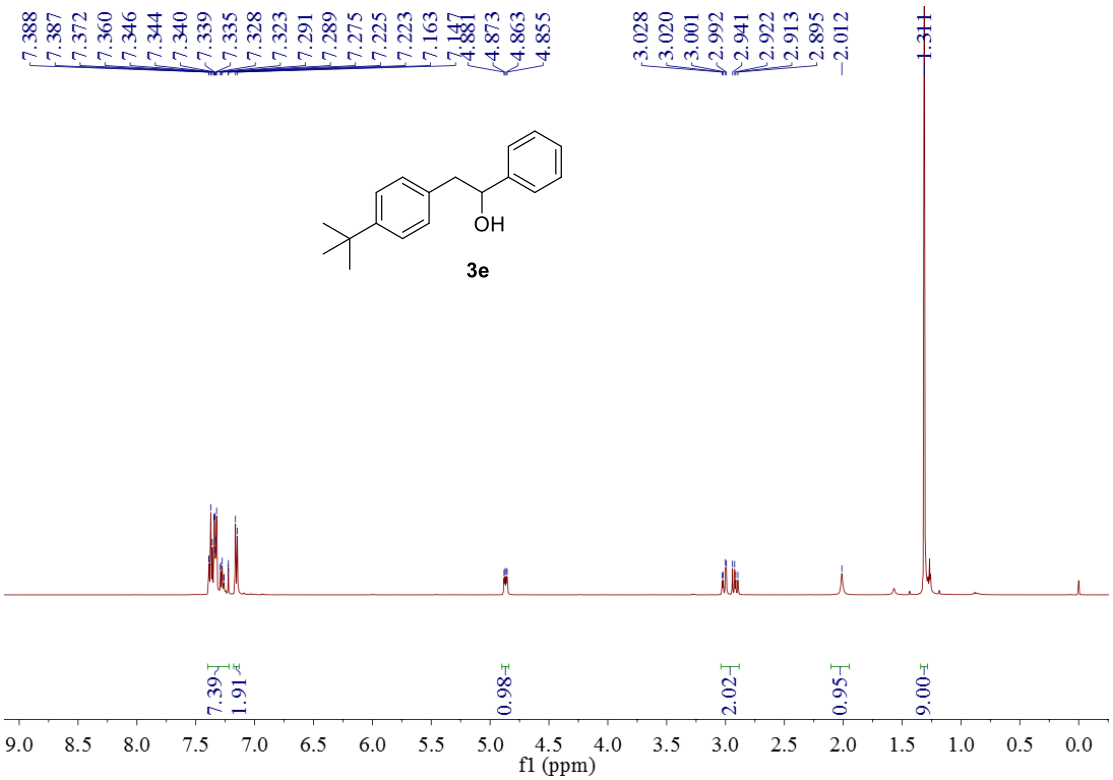
f1 (ppm)

**3d**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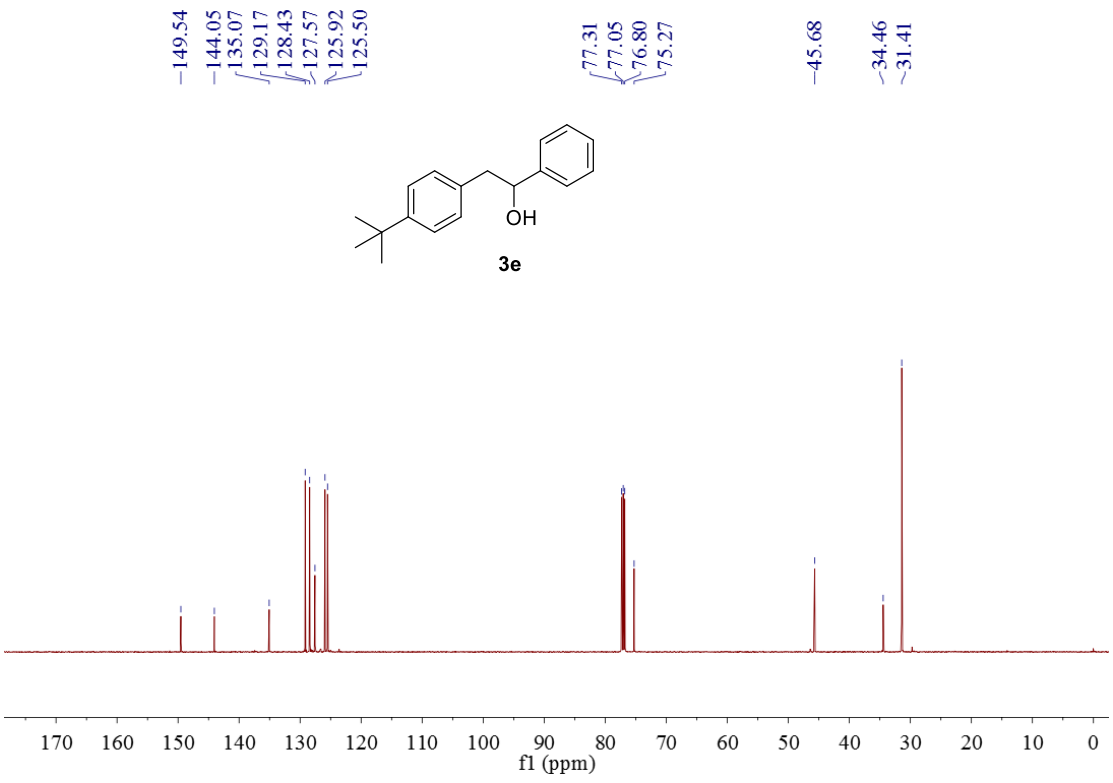
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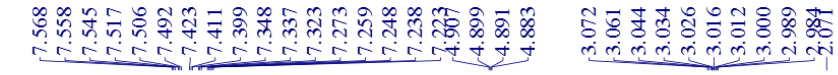
f1 (ppm)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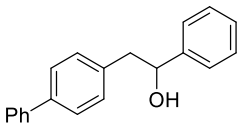
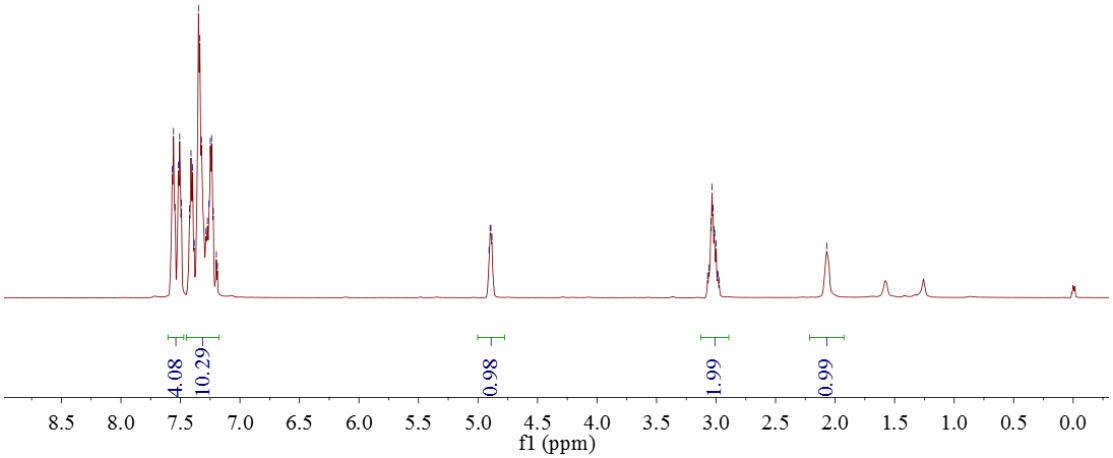

Ph
OH

**3f**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f1 (ppm)


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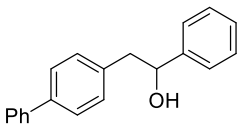
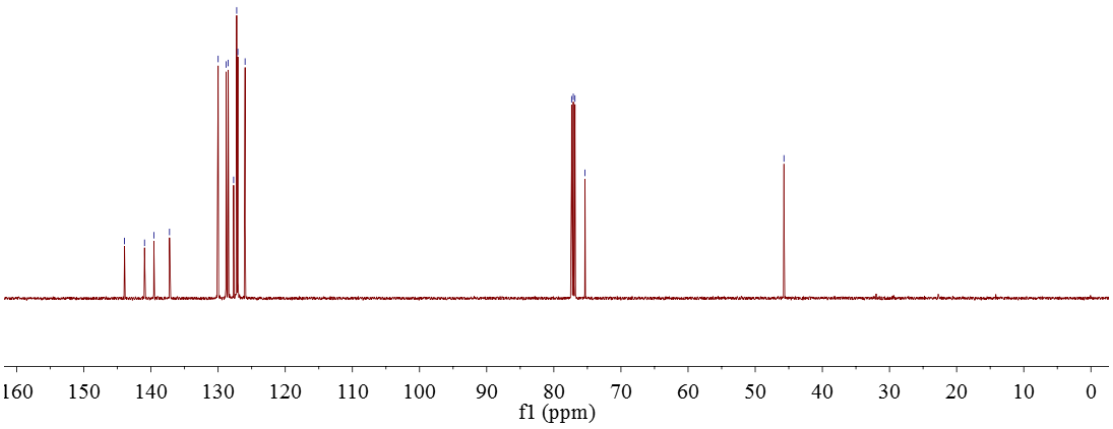

Ph
OH

**3f**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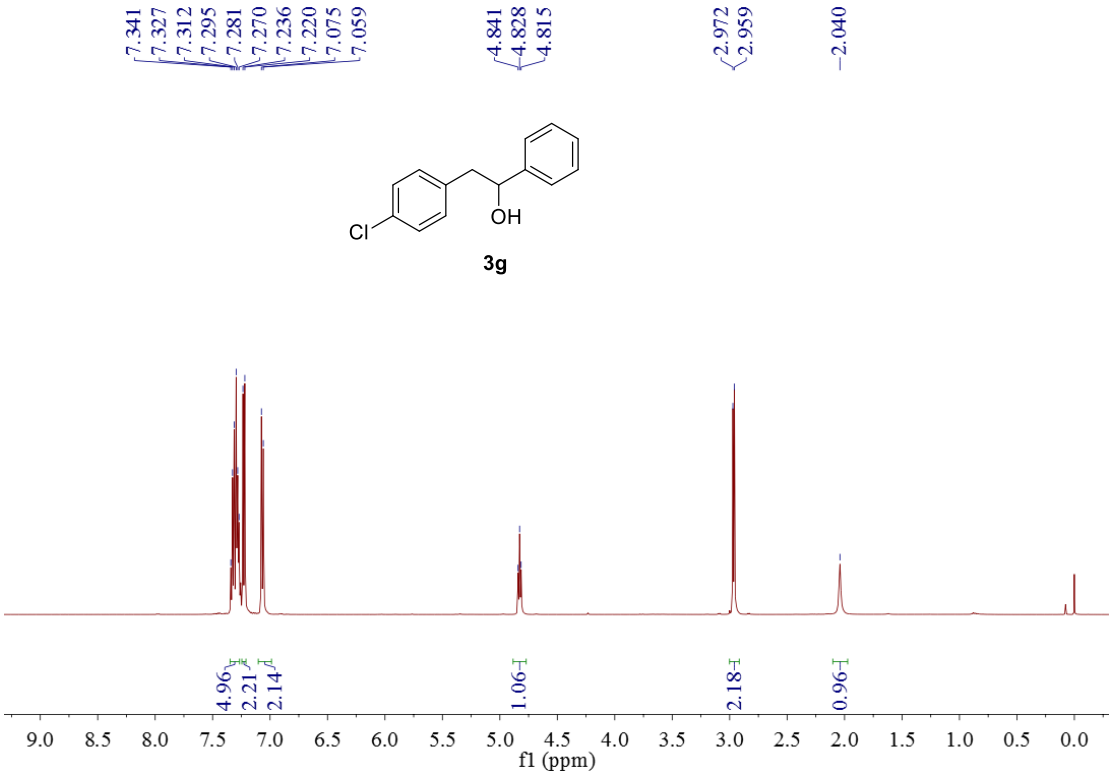
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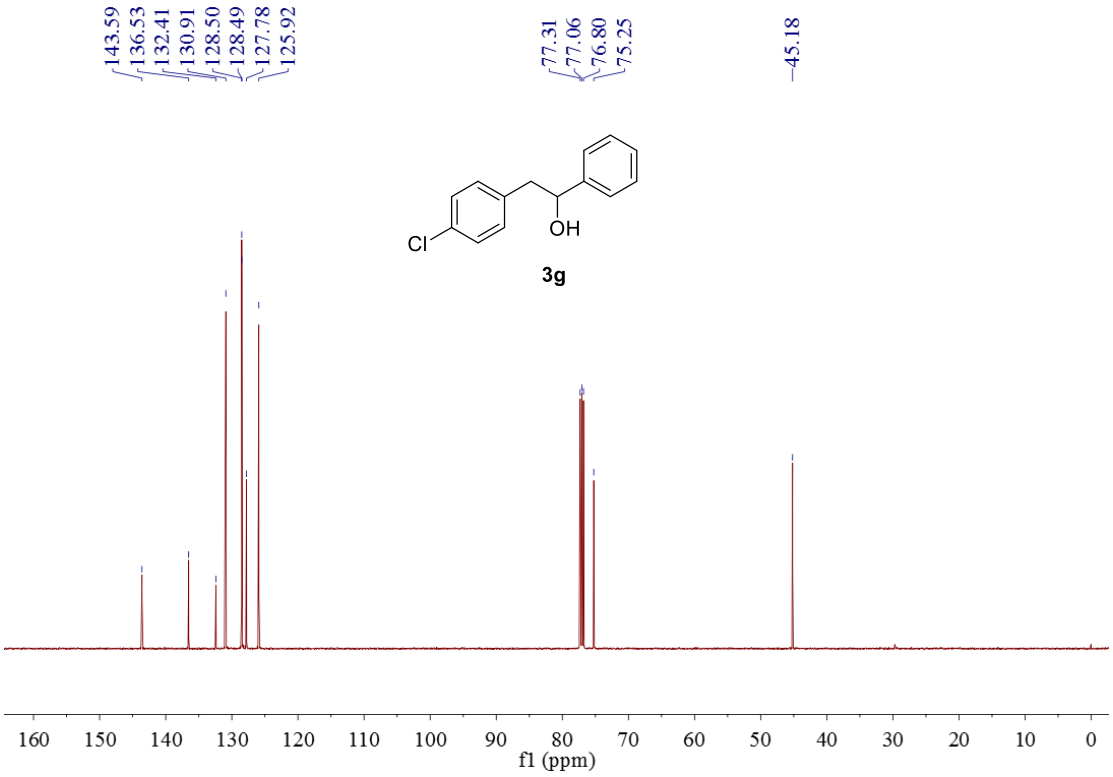
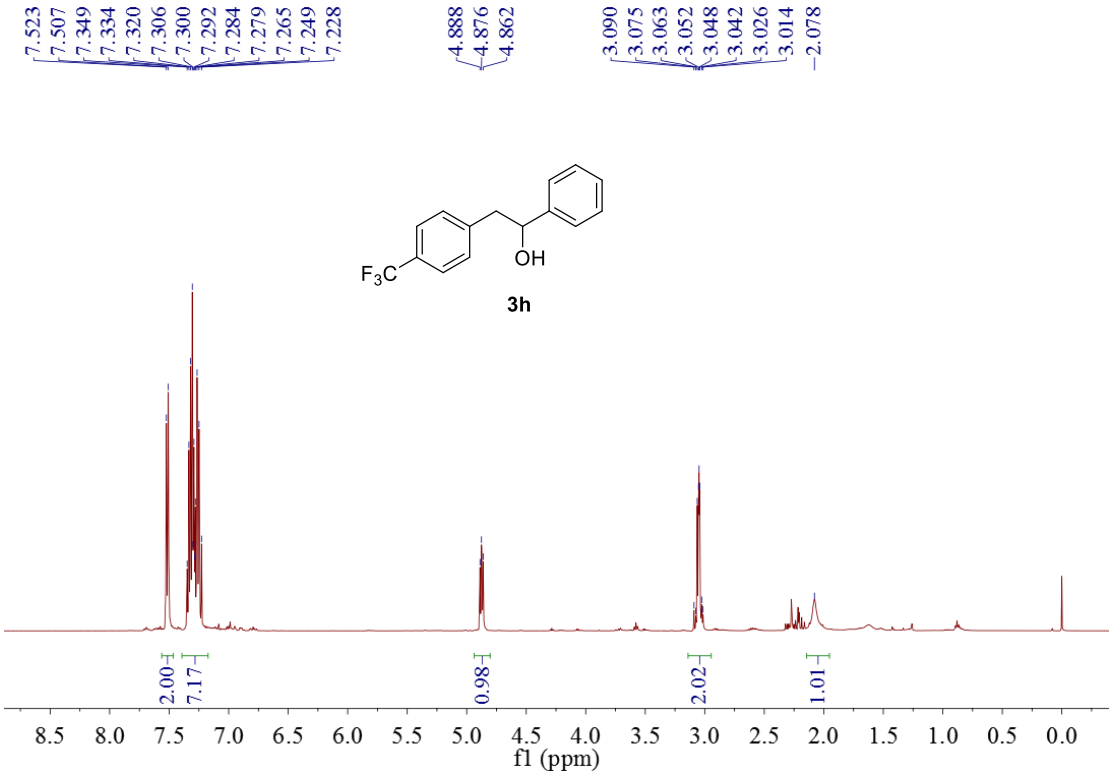
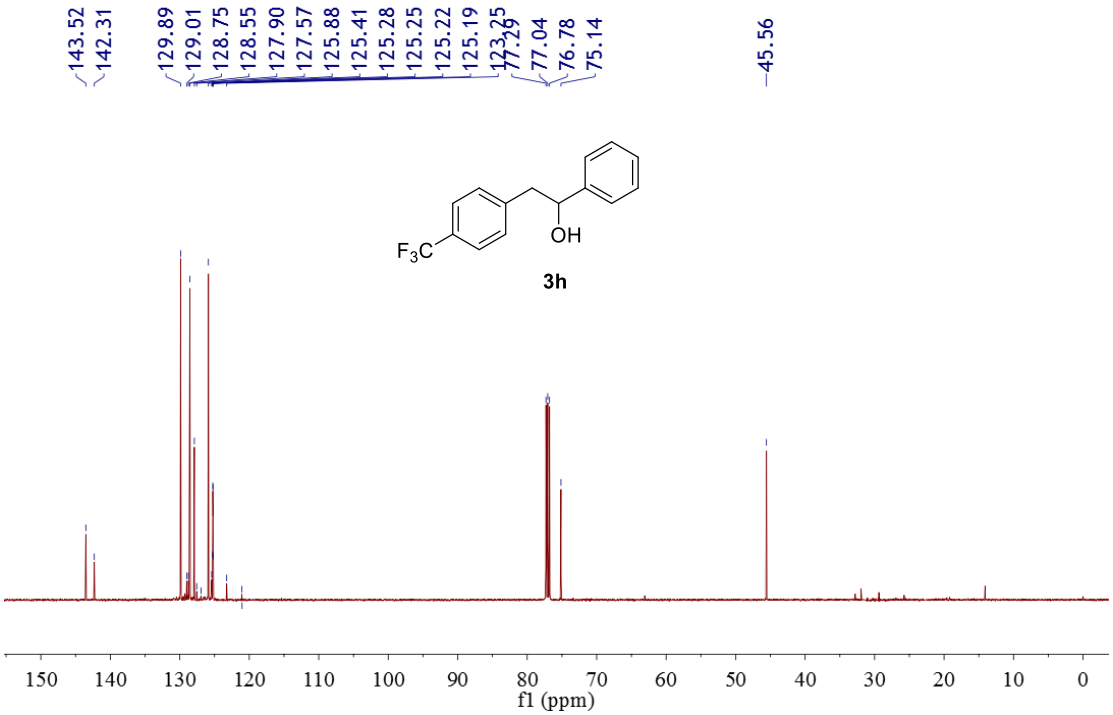
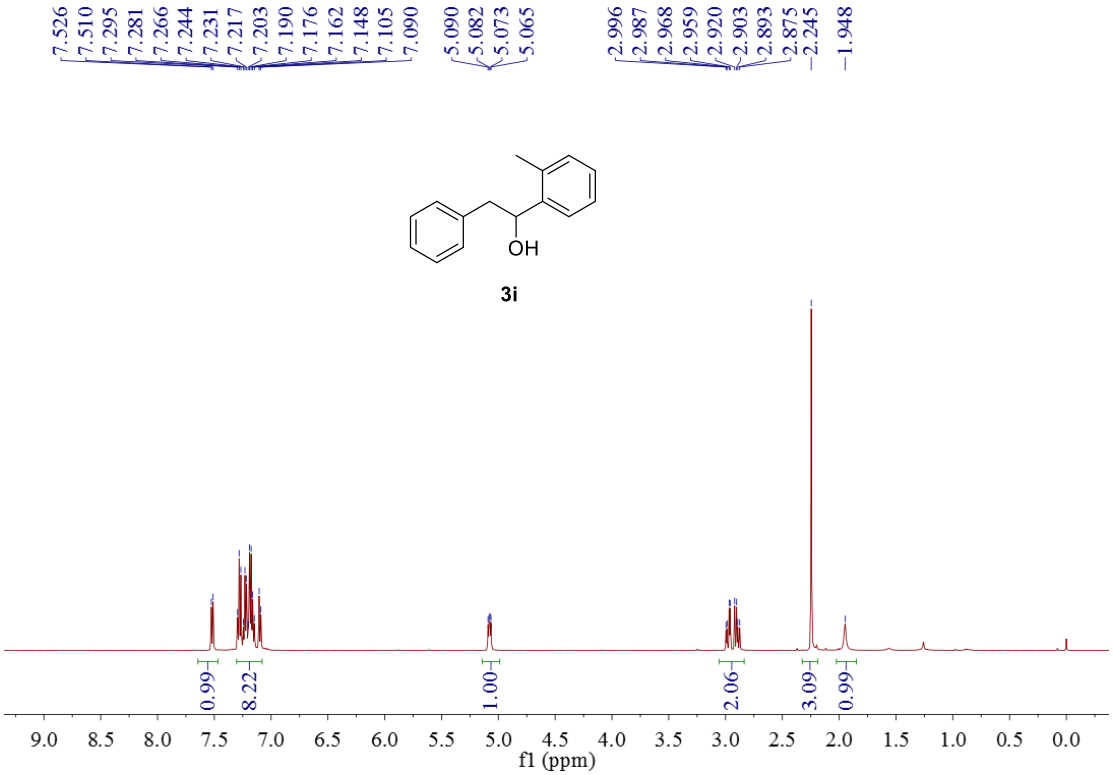
2.972
2.959
2.040
Cl
OH
3g
4.96
2.21
2.14
1.06
2.18
0.96
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

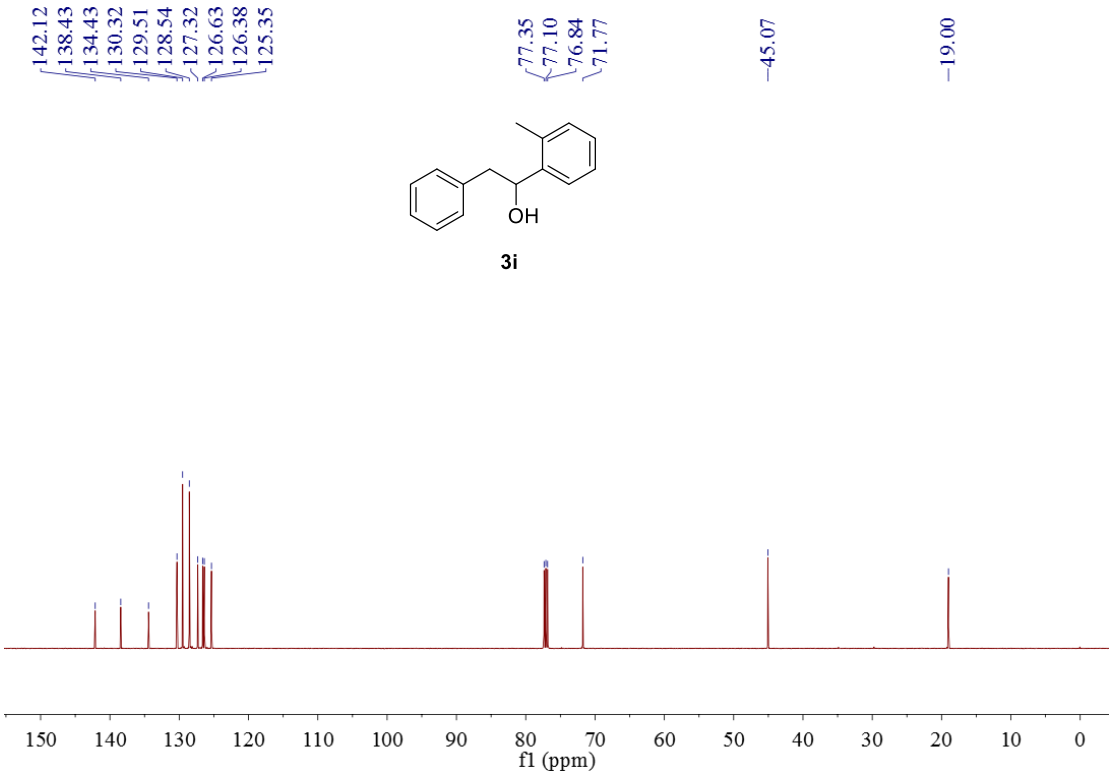

143.59
136.53
132.41
130.91
128.50
128.49
127.78
125.92
77.31
77.06
76.80
75.25
45.18
Cl
OH
3g
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.523
7.507
7.349
7.334
7.320
7.306
7.300
7.292
7.284
7.279
7.265
7.249
7.228
4.888
4.876
4.862
3.090
3.075
3.063
3.052
3.048
3.042
3.026
3.014
2.078
F3C
OH
3h
2.00
7.17
0.98
2.02
1.01
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)

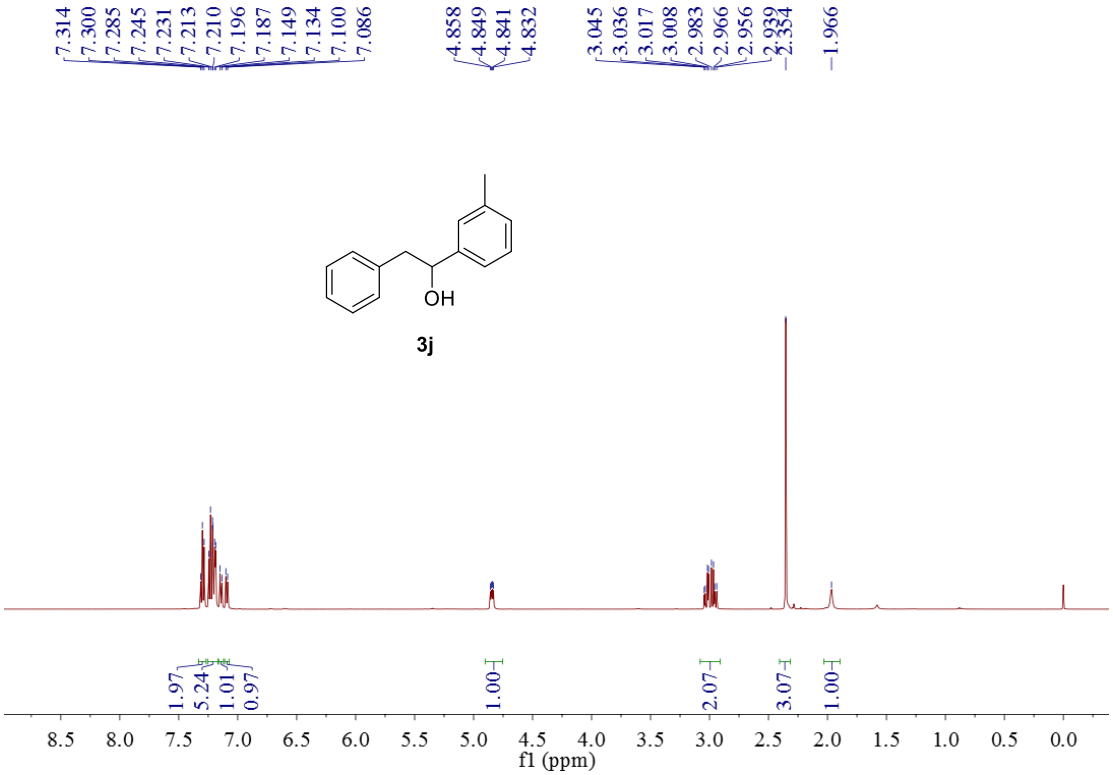

143.52
142.31
129.89
129.01
128.75
128.55
127.90
127.57
125.88
125.41
125.28
125.25
125.22
125.19
123.25
77.29
77.04
76.78
75.14
45.56
F3C
OH
3h
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.526
7.510
7.295
7.281
7.266
7.244
7.231
7.217
7.203
7.190
7.176
7.162
7.148
7.105
7.090
5.090
5.082
5.073
5.065
2.996
2.987
2.968
2.959
2.920
2.903
2.893
2.875
2.245
1.948
OH
0.99
8.22
1.00
2.06
3.09
0.99
f1 (ppm)

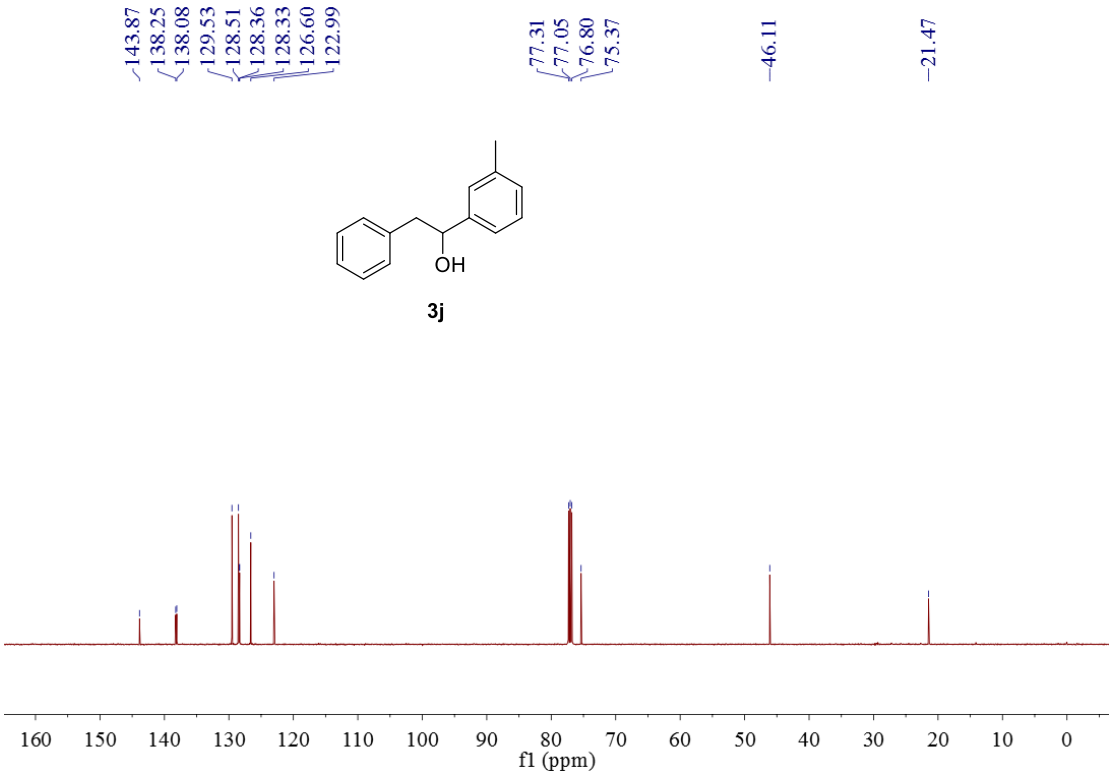
**3i**


142.12
138.43
134.43
130.32
129.51
128.54
127.32
126.63
126.38
125.35
77.35
77.10
76.84
71.77
45.07
19.00
OH
f1 (ppm)

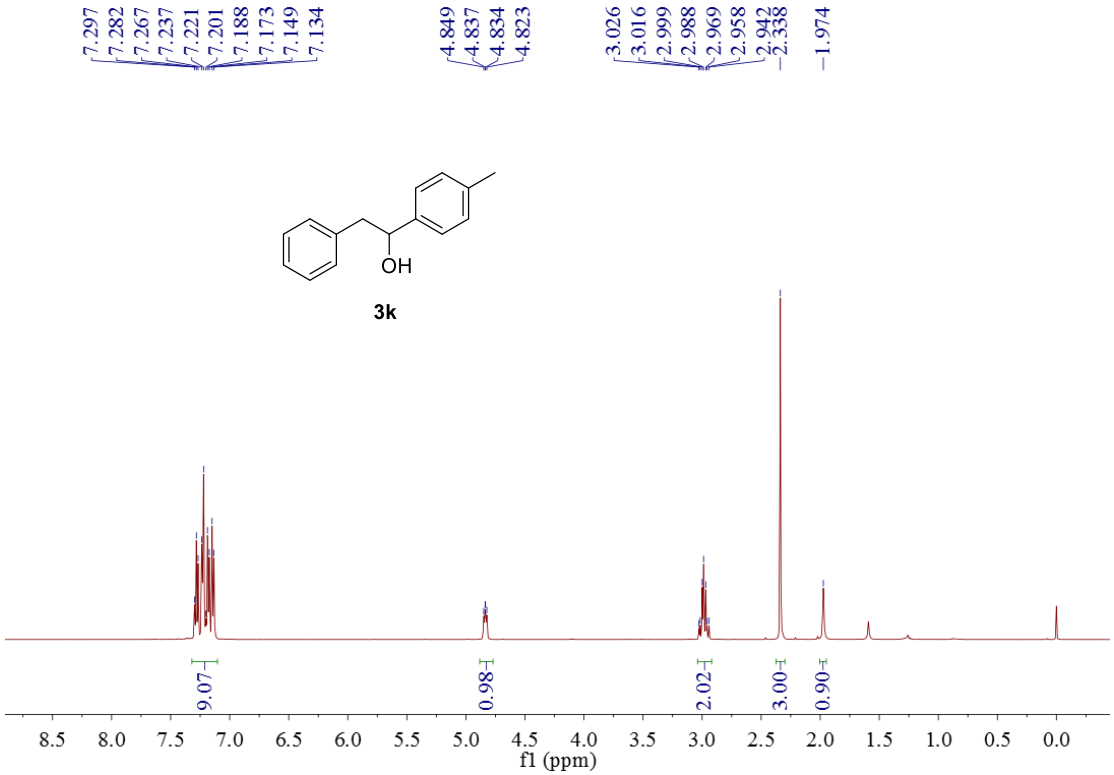
**3i**

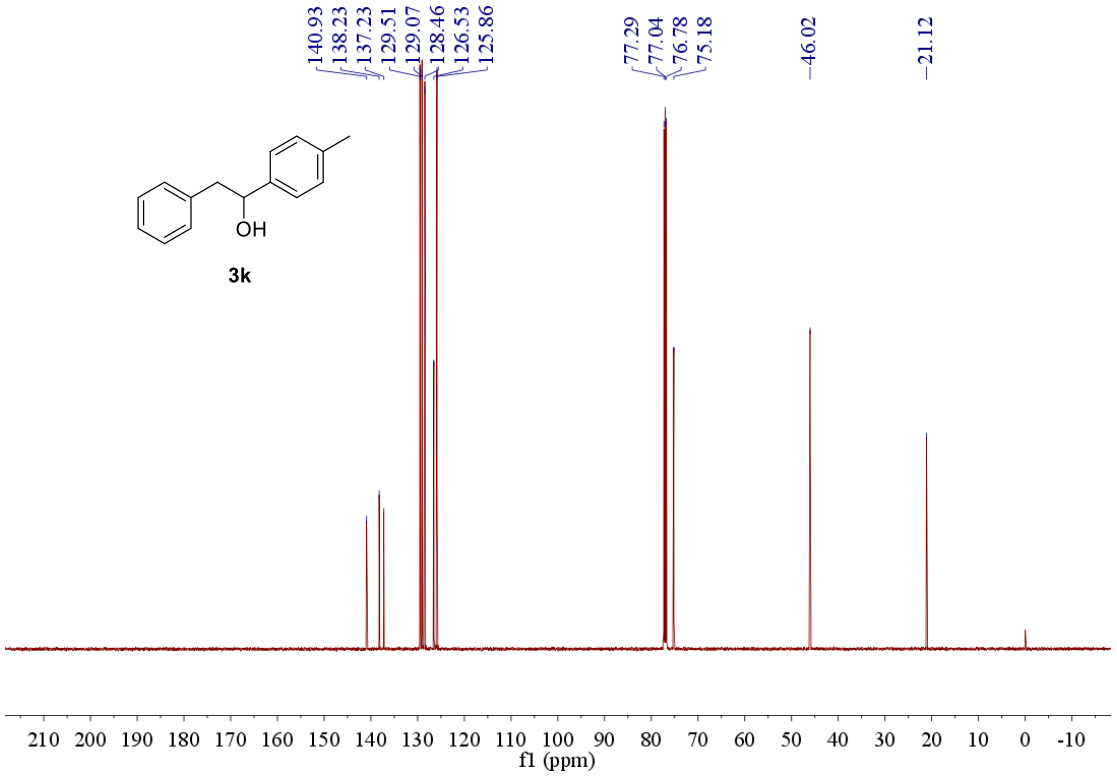

7.314
7.300
7.285
7.245
7.231
7.213
7.210
7.196
7.187
7.149
7.134
7.100
7.086
4.858
4.849
4.841
4.832
3.045
3.036
3.017
3.008
2.983
2.966
2.956
2.939
2.334
1.966
OH
1.97
5.24
1.01
0.97
1.00
2.07
3.07
1.00
f1 (ppm)

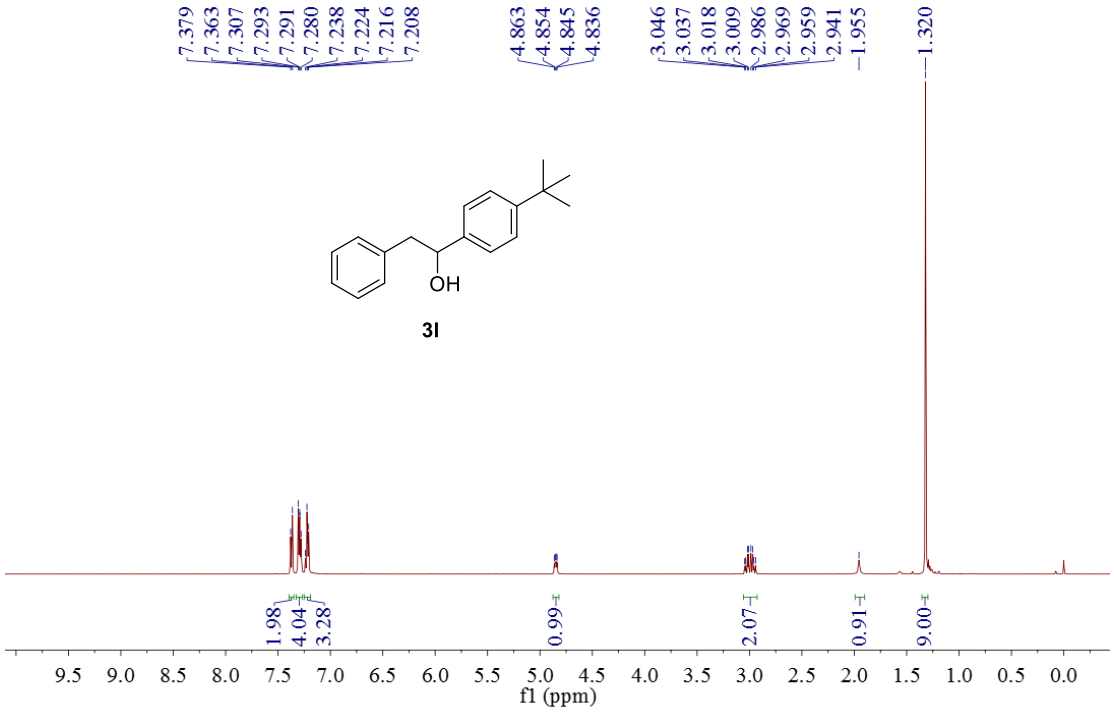
**3j**


143.87
138.25
138.08
129.53
128.51
128.36
128.33
126.60
122.99
77.31
77.05
76.80
75.37
46.11
21.47
OH
f1 (ppm)

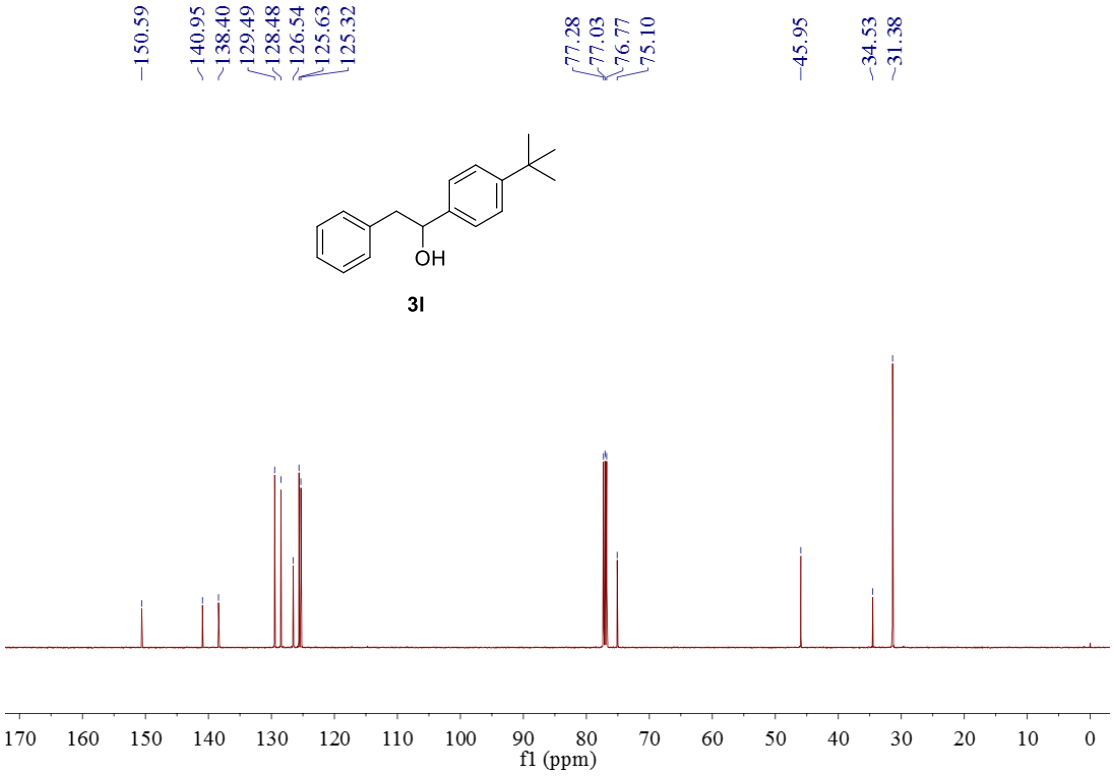
**3j**


7.297
7.282
7.267
7.237
7.221
7.201
7.188
7.173
7.149
7.134
4.849
4.837
4.834
4.823
3.026
3.016
2.999
2.988
2.969
2.958
2.942
2.338
1.974
OH
3k
9.07
0.98
2.02
3.00
0.90
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)


140.93
138.23
137.23
129.51
129.07
128.46
126.53
125.86
77.29
77.04
76.78
75.18
46.02
21.12
OH
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

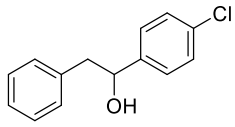

7.379
7.363
7.307
7.293
7.291
7.280
7.238
7.224
7.216
7.208
4.863
4.854
4.845
4.836
3.046
3.037
3.018
3.009
2.986
2.969
2.959
2.941
1.955
1.320
OH
1.98
4.04
3.28
0.99
2.07
0.91
9.00
f1 (ppm)

**3l**

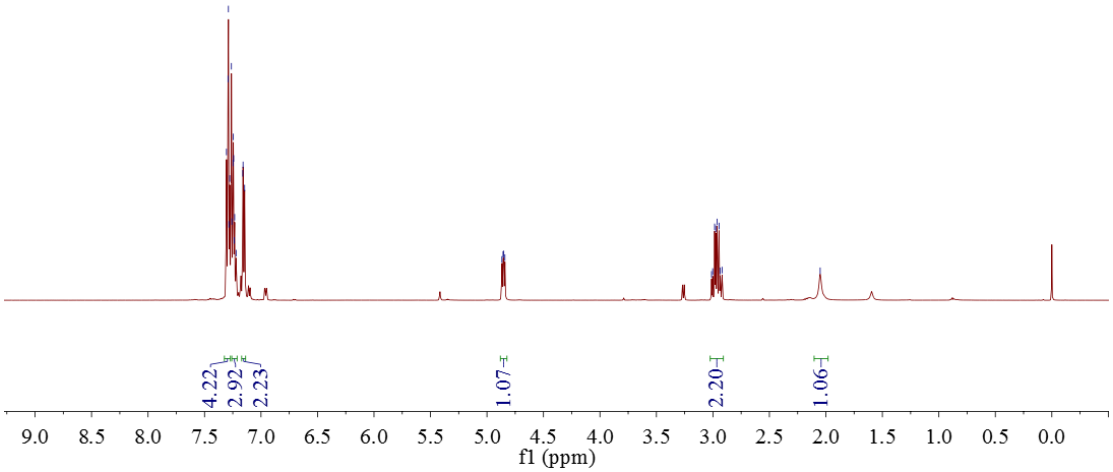

150.59
140.95
138.40
129.49
128.48
126.54
125.63
125.32
77.28
77.03
76.77
75.10
45.95
34.53
31.38
OH
f1 (ppm)

**3l**

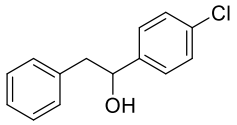

7.306
7.292
7.289
7.284
7.277
7.262
7.258
7.248
7.245
7.242
7.237
7.233
7.221
7.161
7.159
7.145
4.868
4.857
4.851
4.841
3.012
3.002
2.985
2.975
2.962
2.946
2.935
2.918
2.050


Cl
OH

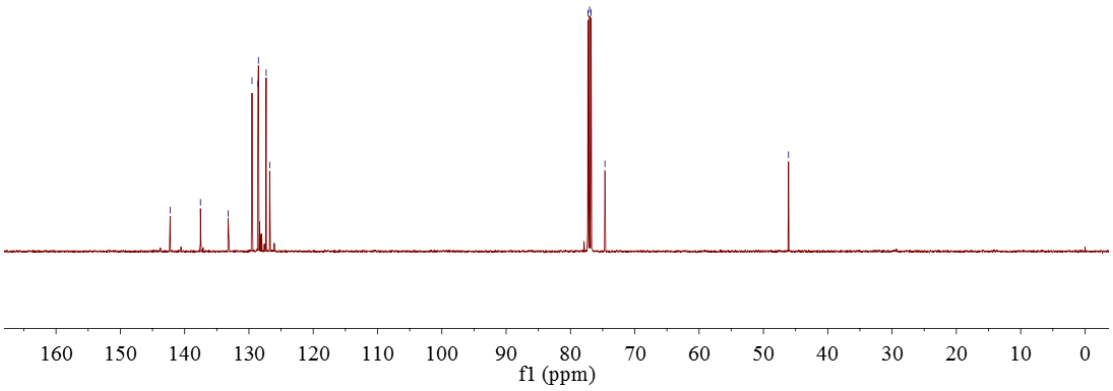
**3m**

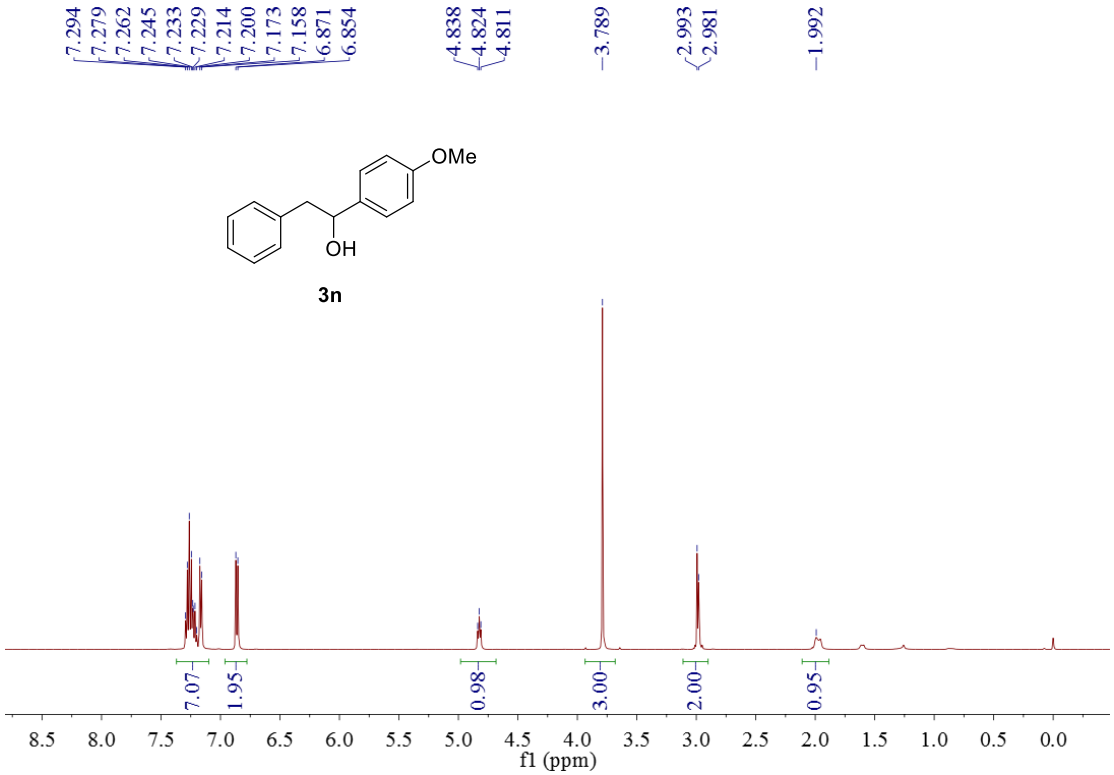

4.22
2.92
2.23
1.07
2.20
1.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

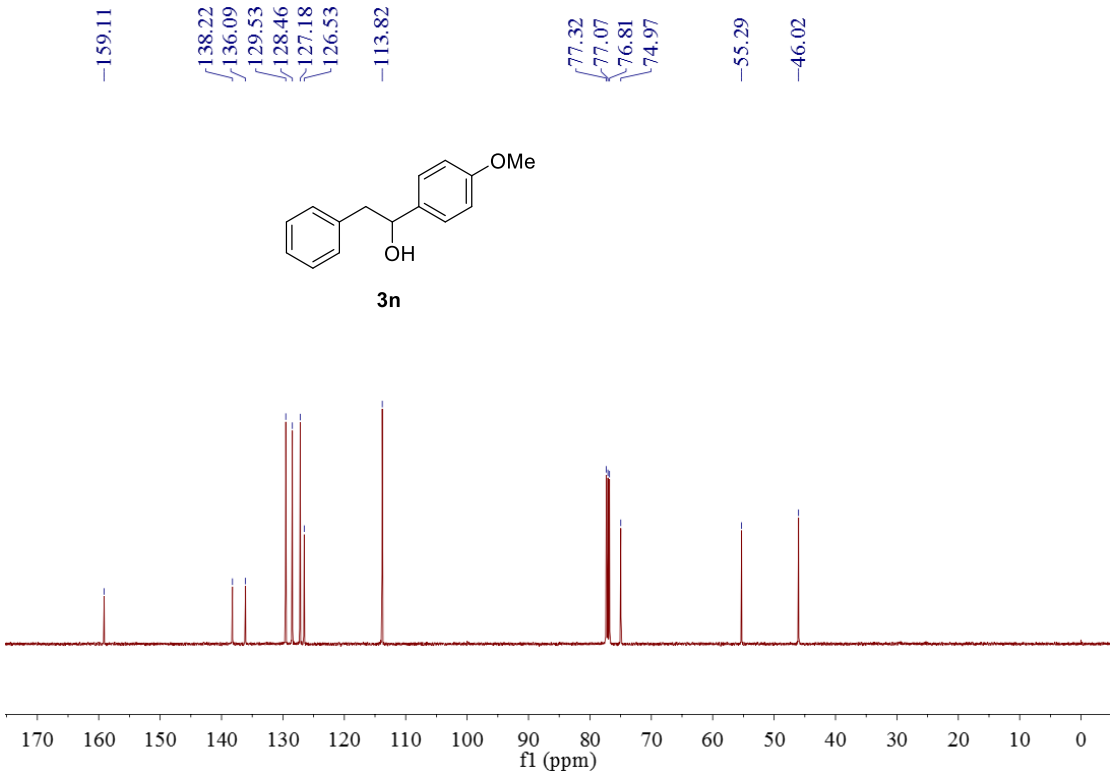

142.25
137.54
133.24
129.52
128.59
128.52
127.31
126.78
77.29
77.04
76.78
74.64
46.10

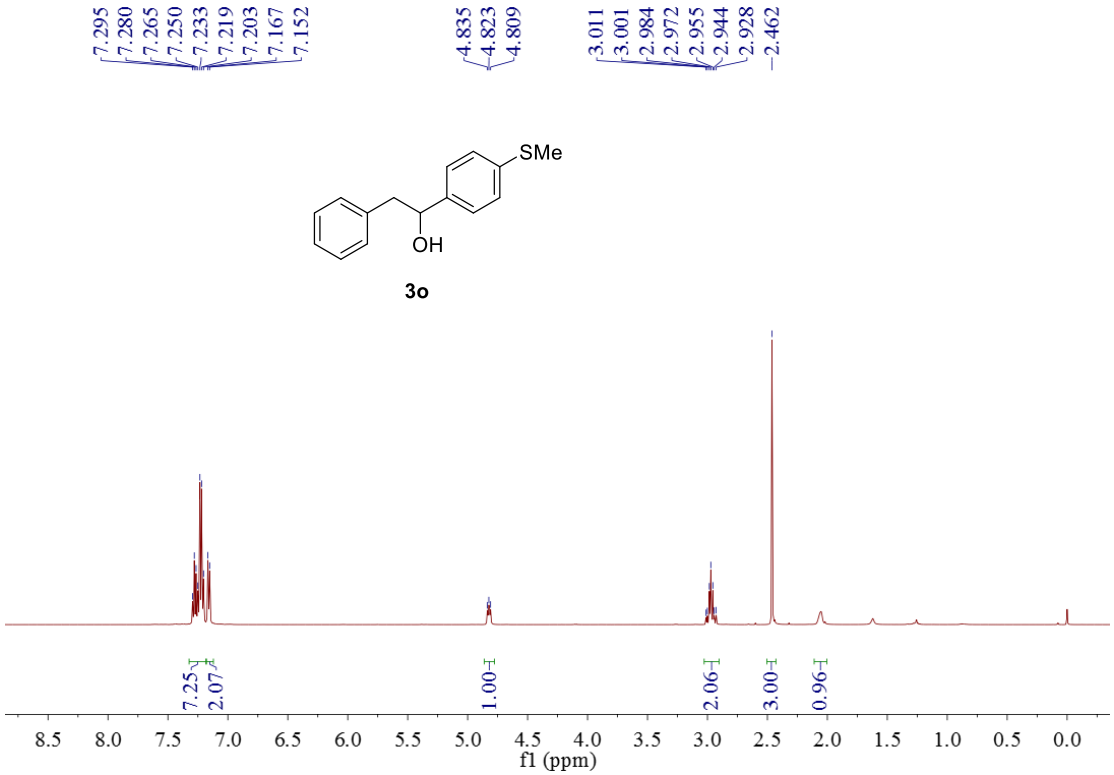

Cl
OH

**3m**

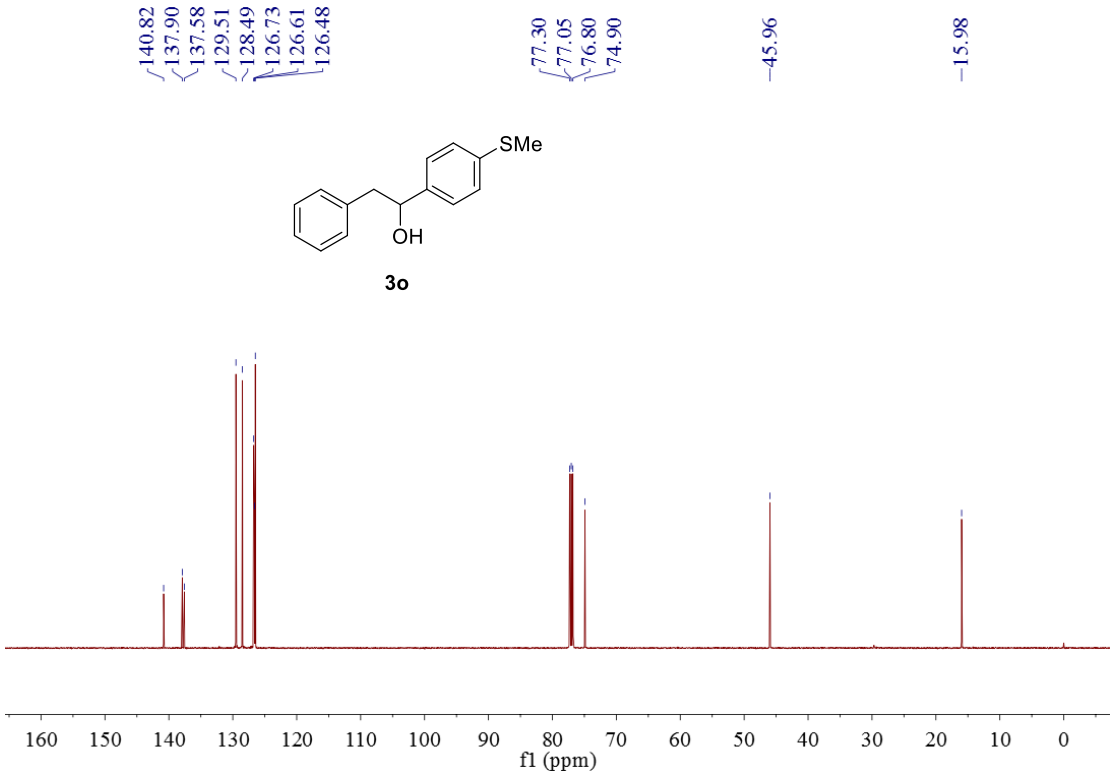

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.294
7.279
7.262
7.245
7.233
7.229
7.214
7.200
7.173
7.158
6.871
6.854
4.838
4.824
4.811
3.789
2.993
2.981
1.992
OMe
OH
3n
7.07
1.95
0.98
3.00
2.00
0.95
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)

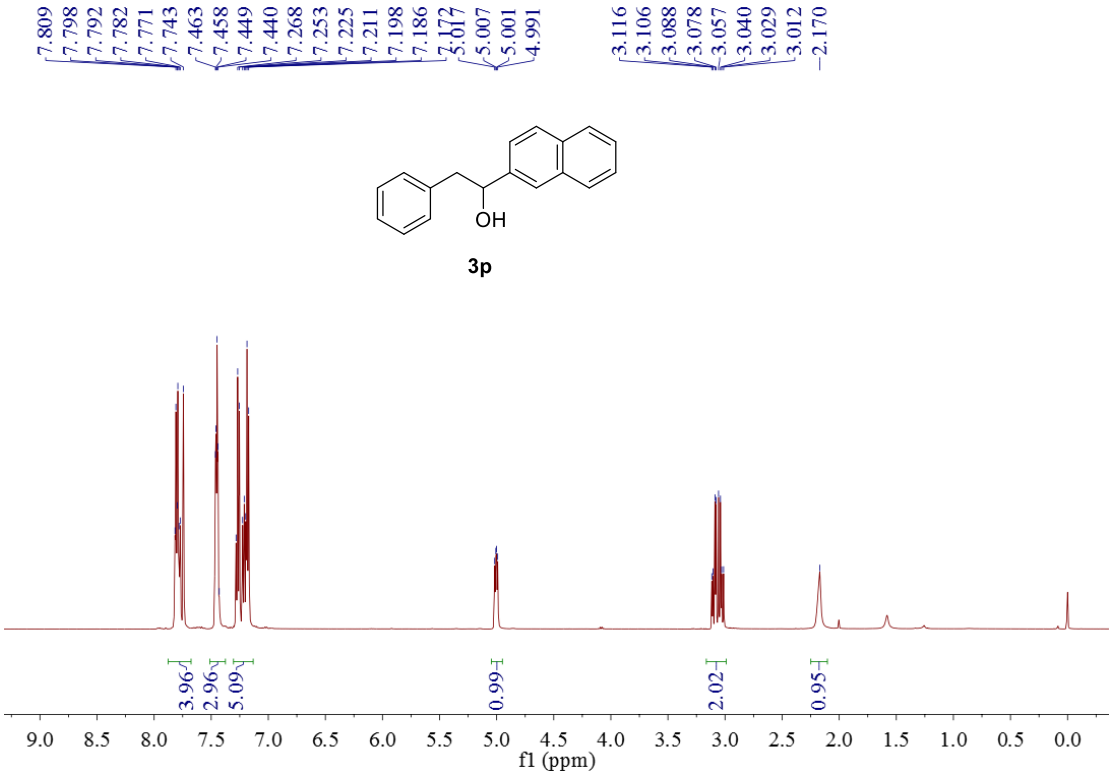

159.11
138.22
136.09
129.53
128.46
127.18
126.53
113.82
77.32
77.07
76.81
74.97
55.29
46.02
OMe
OH
3n
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

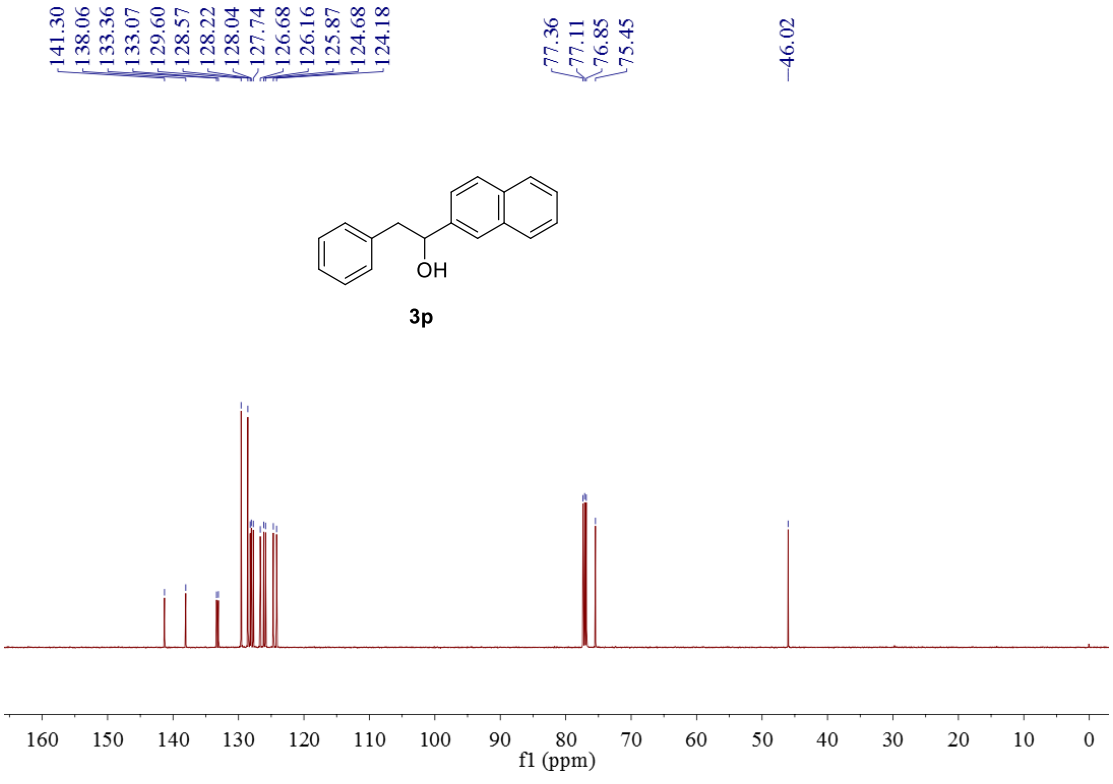

7.295
7.280
7.265
7.250
7.233
7.219
7.203
7.167
7.152
4.835
4.823
4.809
3.011
3.001
2.984
2.972
2.955
2.944
2.928
2.462
SMe
OH
7.25
2.07
1.00
2.06
3.00
0.96
f1 (ppm)

**3o**


140.82
137.90
137.58
129.51
128.49
126.73
126.61
126.48
77.30
77.05
76.80
74.90
45.96
15.98
SMe
OH
f1 (ppm)

**3o**


7.809
7.798
7.792
7.782
7.771
7.743
7.463
7.458
7.449
7.440
7.268
7.253
7.225
7.211
7.198
7.186
5.017
5.007
5.001
4.991
3.116
3.106
3.088
3.078
3.057
3.040
3.029
3.012
2.170
OH
3p
3.96
2.96
5.09
0.99
2.02
0.95
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)


141.30
138.06
133.36
133.07
129.60
128.57
128.22
128.04
127.74
126.68
126.16
125.87
124.68
124.18
77.36
77.11
76.85
75.45
46.02
OH
3p
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)