A three-component reaction of arynes, sodium sulfinates, and aldehydes toward 2-sulfonyl benzyl alcohol derivatives

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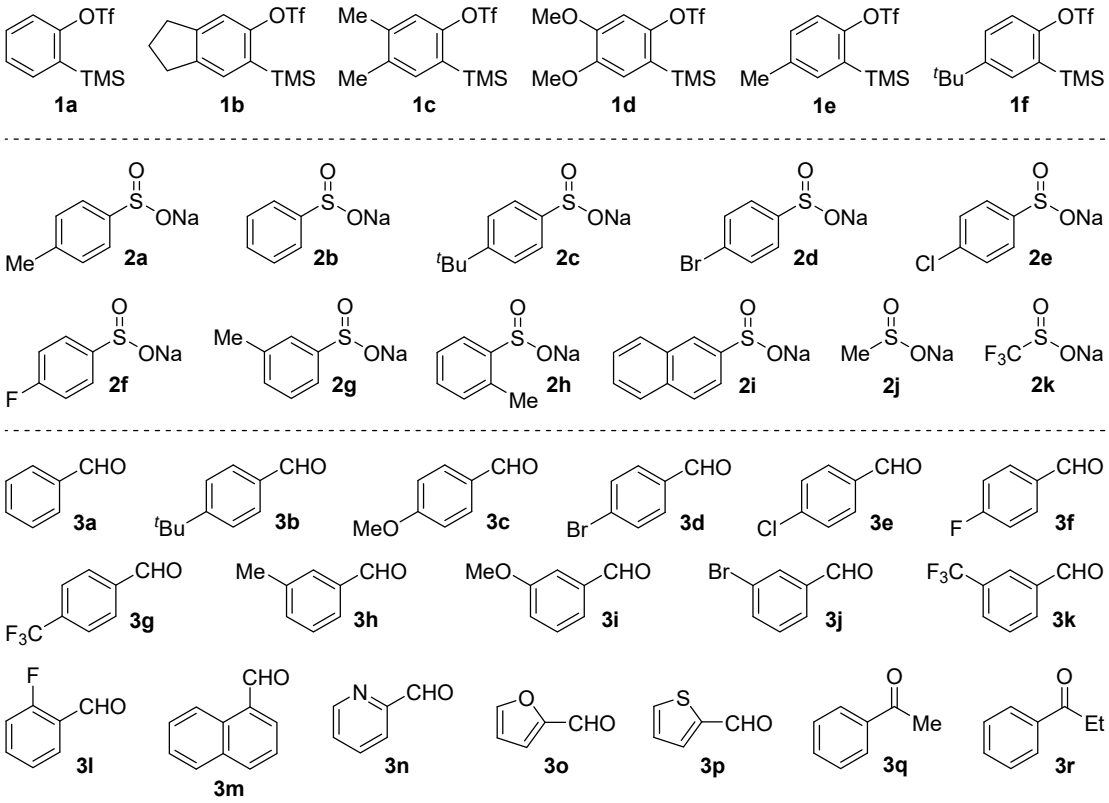
SUPPORTING INFORMATION

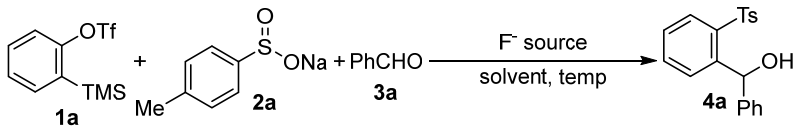
Content:

1. Overview of Substrates Numbering



2. Initial Optimization of Reaction Conditions

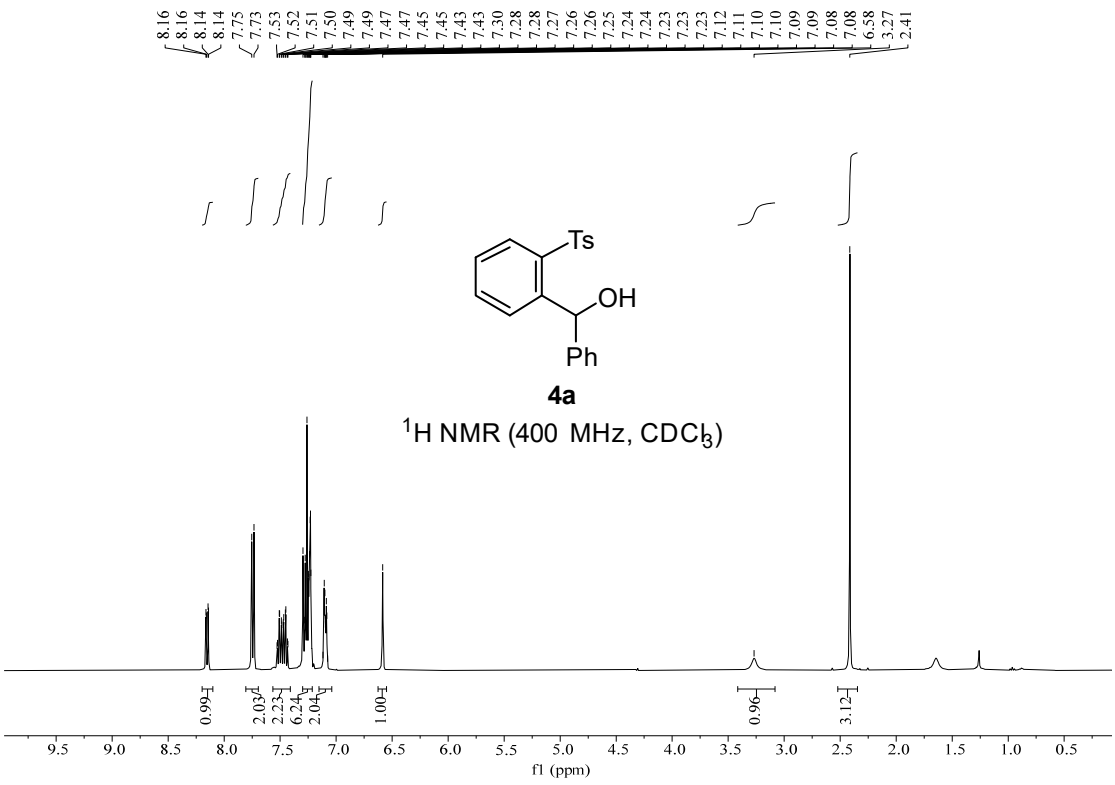
Table S1 Reaction conditions optimization[a,b]

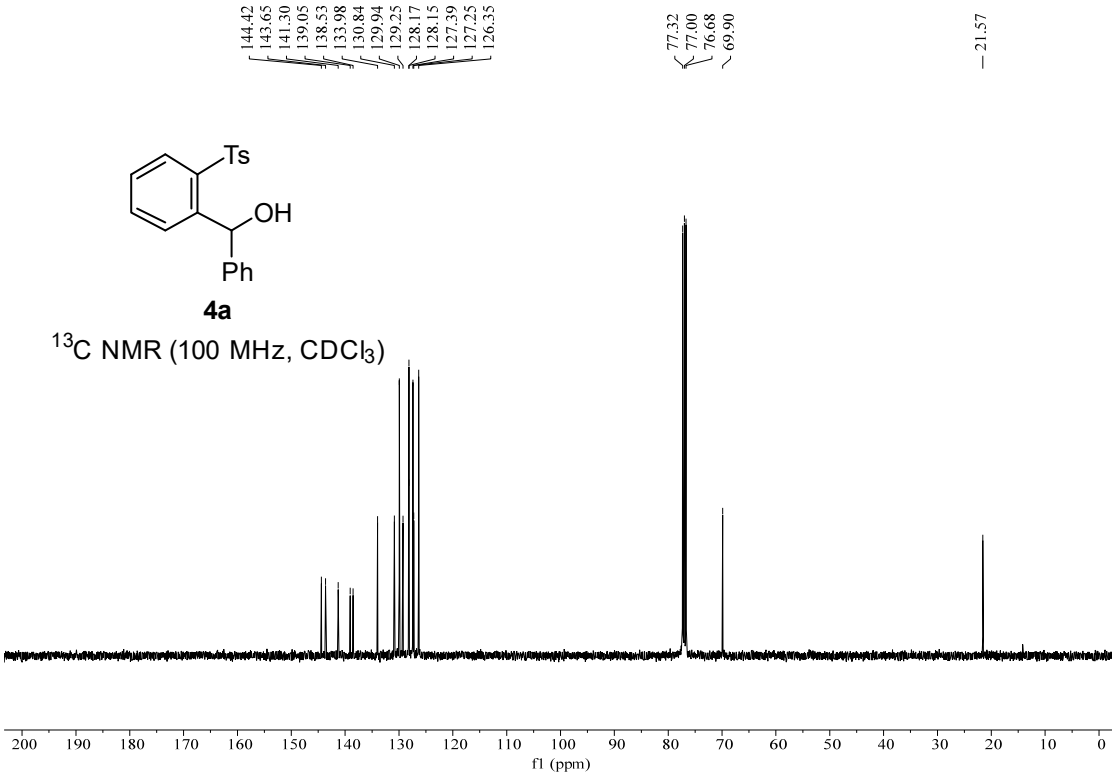


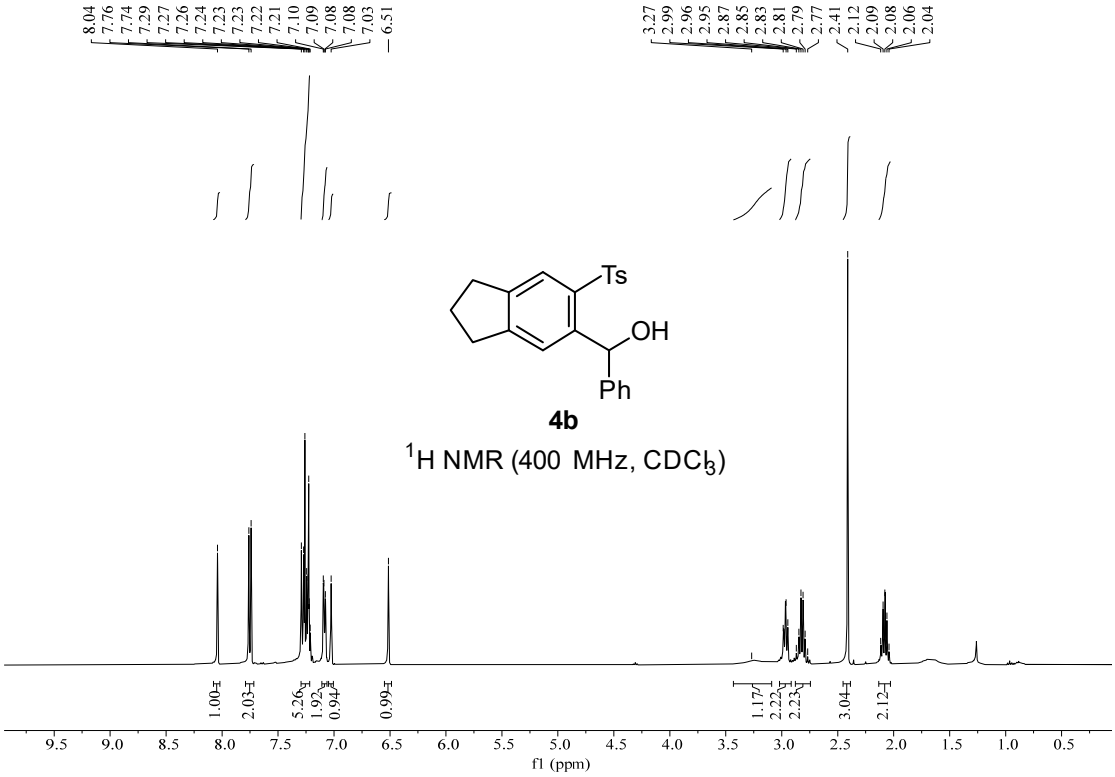
Entry	Equiv of **1a**	Equiv of **2a**	Equiv of **3a**	F^- source (equiv)	Equiv of 18-crown-6	Solvent (mL)	Temp (°C)	Yield (%)
1	2	1	10	TBAF (1.1)	0	CH_3CN (1.0)	rt	trace
2	2	1	10	CsF (4.0)	0	CH_3CN (4.0)	80	10
3	2	2	1	TBAF (5.5)	0	THF (2.0)	45	trace
4	2	2	1	CsF (5.5)	0	CH_3CN (2.0)	45	28
5	2	2	1	CsF (5.5)	5.5	THF (2.0)	45	58
6	2	2	1	KF (5.5)	5.5	THF (2.0)	45	trace
7	2	1	2	CsF (5.5)	5.5	THF (2.0)	45	45
8	2	1	5	CsF (5.5)	5.5	THF (2.0)	45	59
9	2	1	5	CsF (5.5)	5.5	THF (1.0)	45	60
10	2	1	8	CsF (5.5)	5.5	THF (1.0)	45	63
11	2	1	10	CsF (5.5)	5.5	THF (1.0)	45	82

[a] Reaction conditions: **1a**, **2a** (0.2 mmol), **3a**, fluoride, and 18-crown-6 in a solvent under N_2 for 12 h. [b] Isolated yield based on **2a** or **3a**.

3. Copies of ^{1}H and ^{13}C NMR Spectra for Compounds 4 and 5



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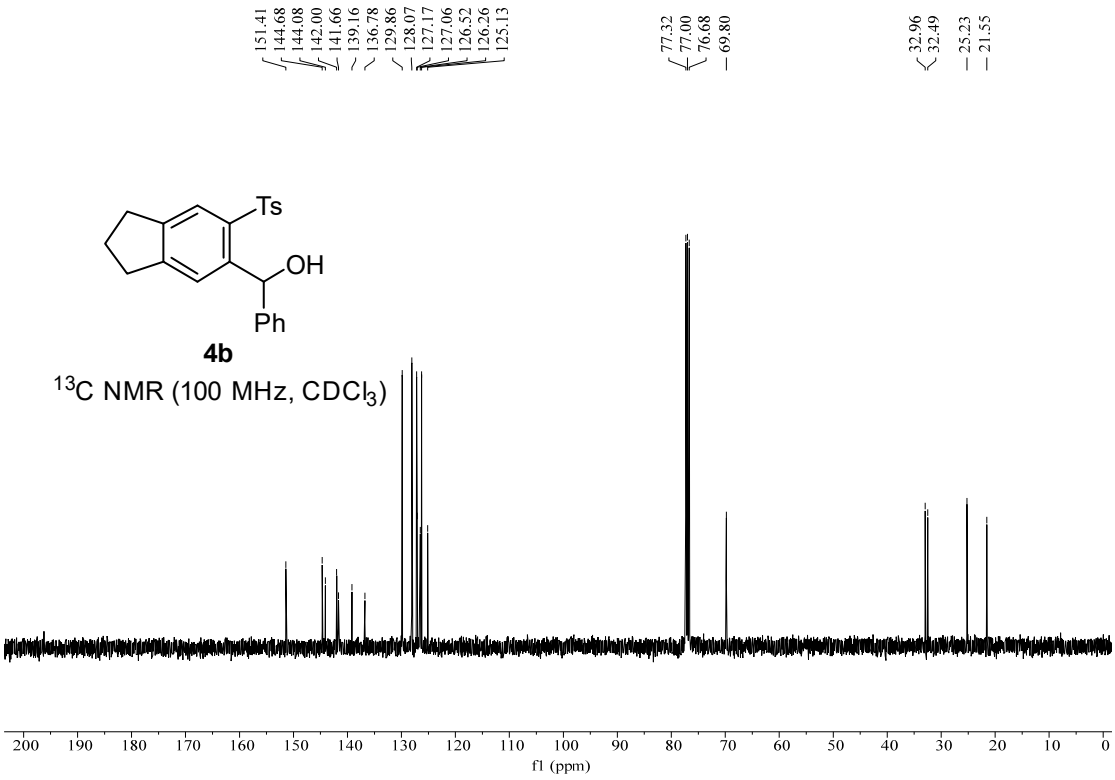
7.76
7.74
7.29
7.27
7.26
7.24
7.23
7.23
7.22
7.21
7.10
7.09
7.08
7.08
7.03
6.51
3.27
2.99
2.96
2.95
2.87
2.85
2.83
2.81
2.79
2.77
2.41
2.12
2.09
2.08
2.06
2.04
Ts
OH
Ph
4b
1H NMR (400 MHz, CDCl3)
1.00
2.03
5.26
1.92
0.94
0.99
1.17
2.22
2.23
3.04
2.12
f1 (ppm)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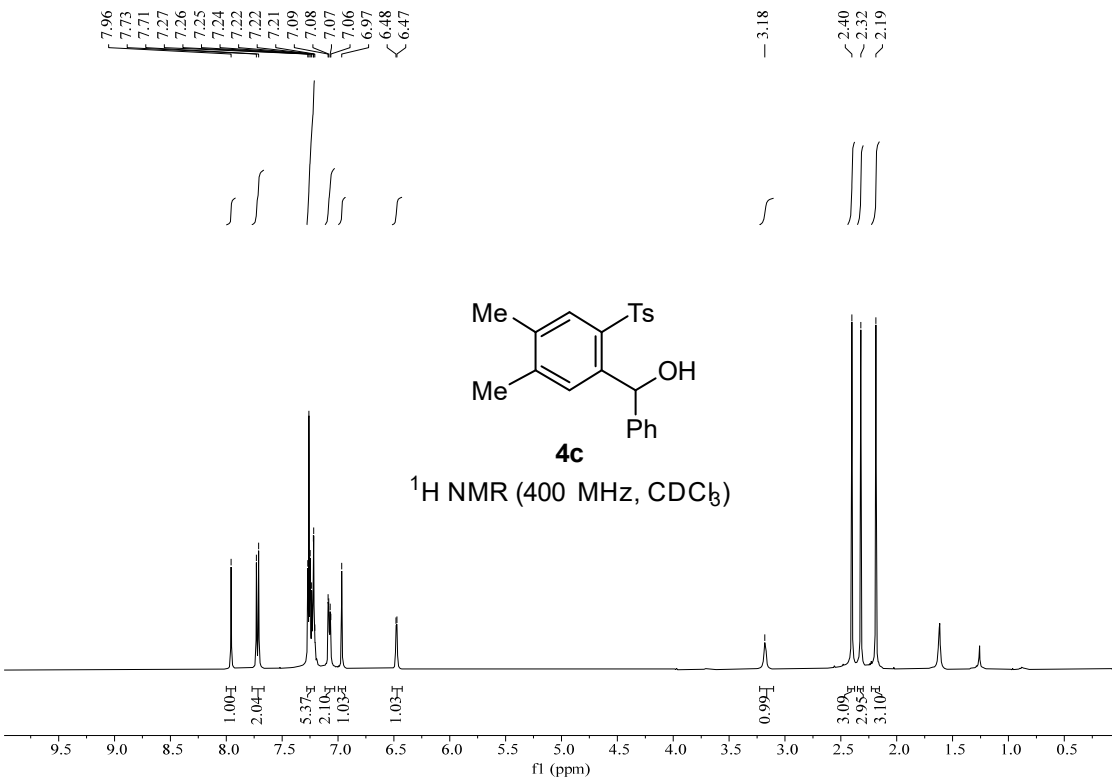
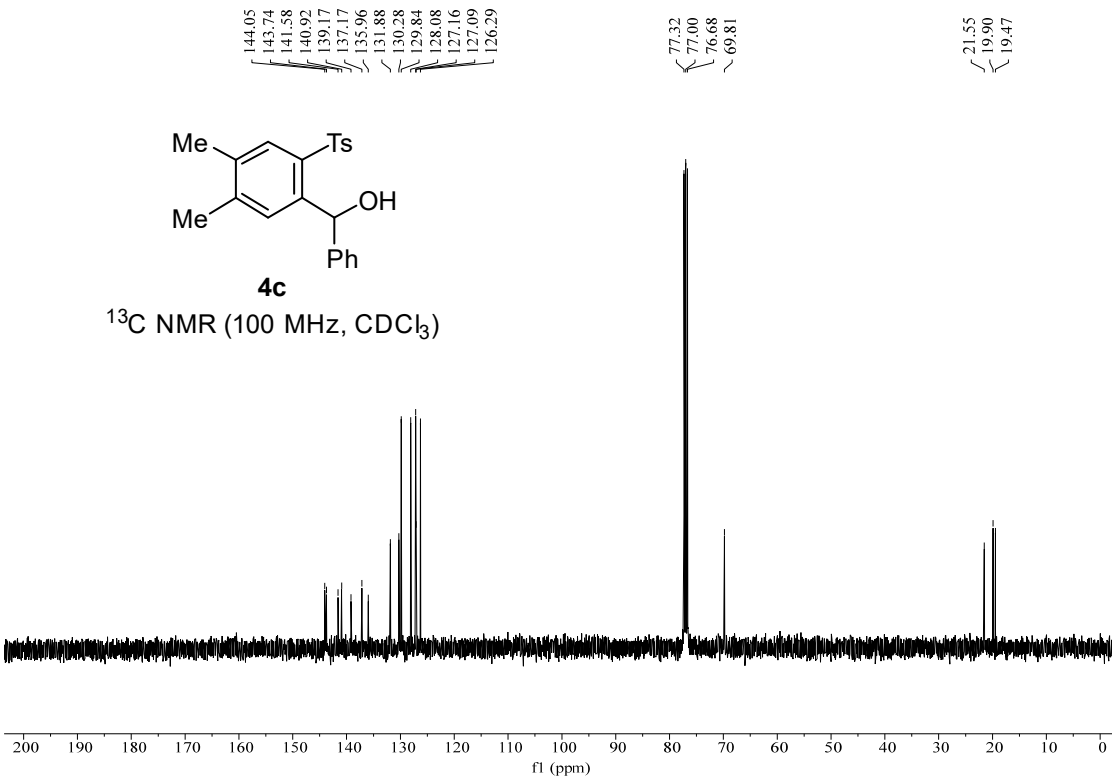
144.68
144.08
142.00
141.66
139.16
136.78
129.86
128.07
127.17
127.06
126.52
126.26
125.13
77.32
77.00
76.68
69.80
32.96
32.49
25.23
21.55
Ts
OH
Ph
4b
13C NMR (100 MHz, CDCl3)
f1 (ppm)



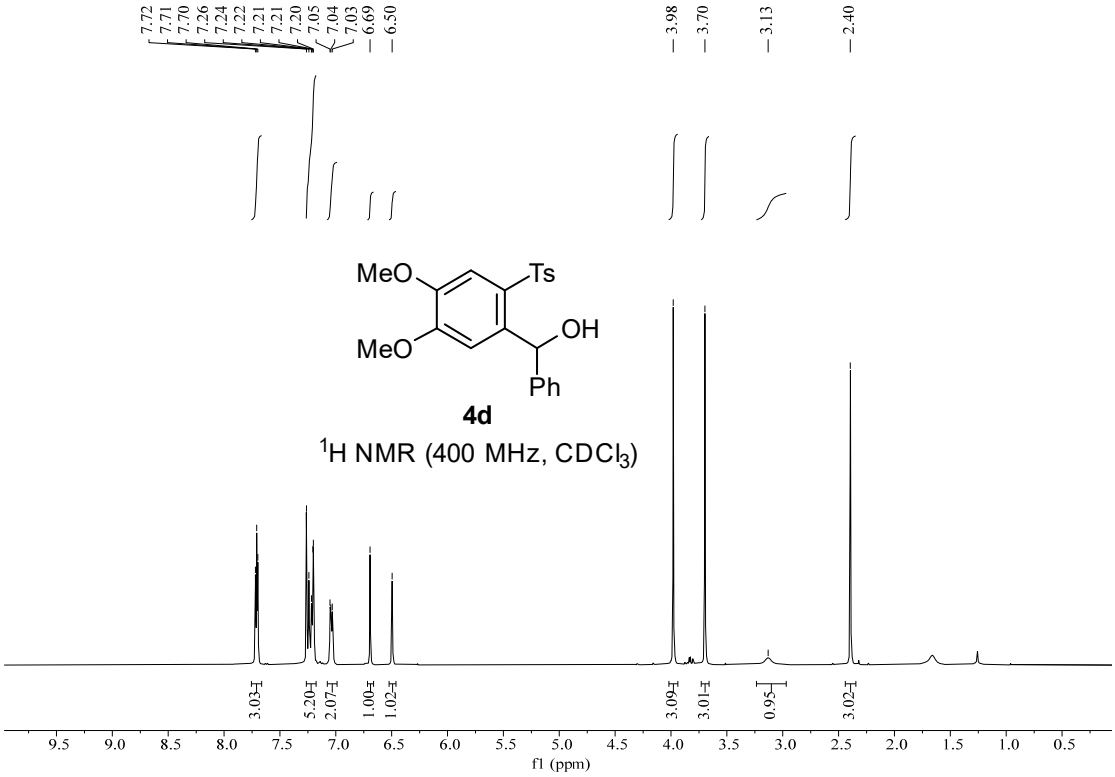
4c

^{1}H NMR (400 MHz, $CDCl_3$)



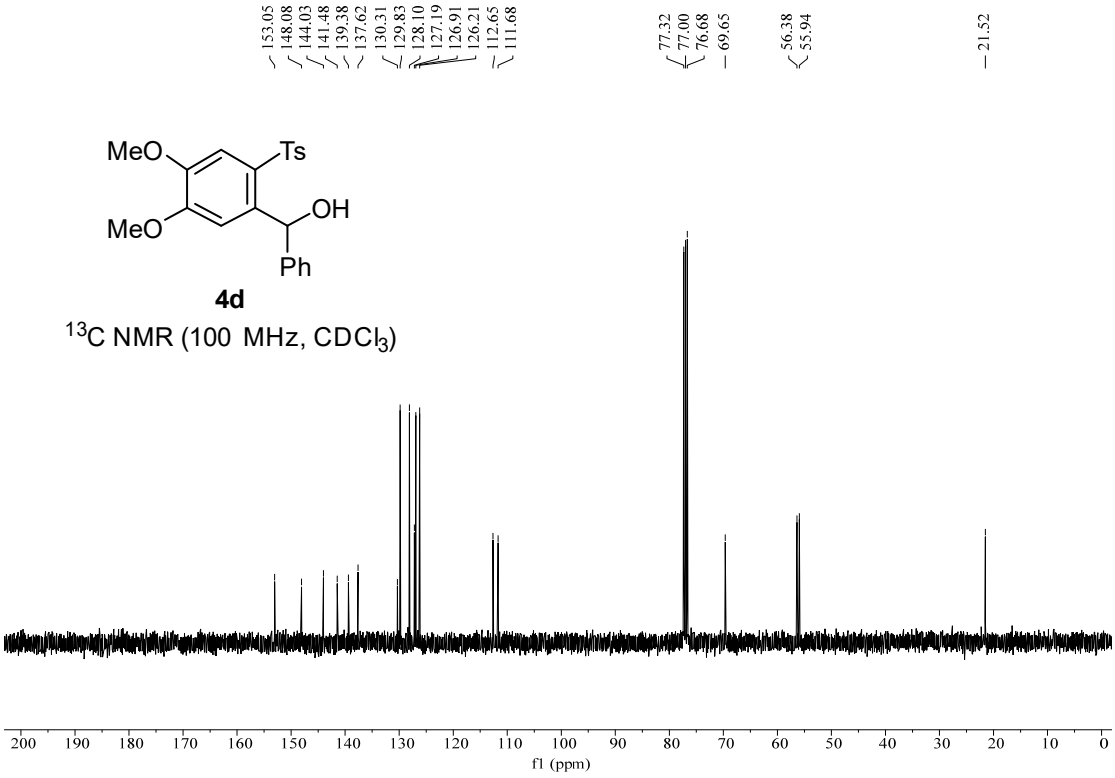
4c

^{13}C NMR (100 MHz, $CDCl_3$)



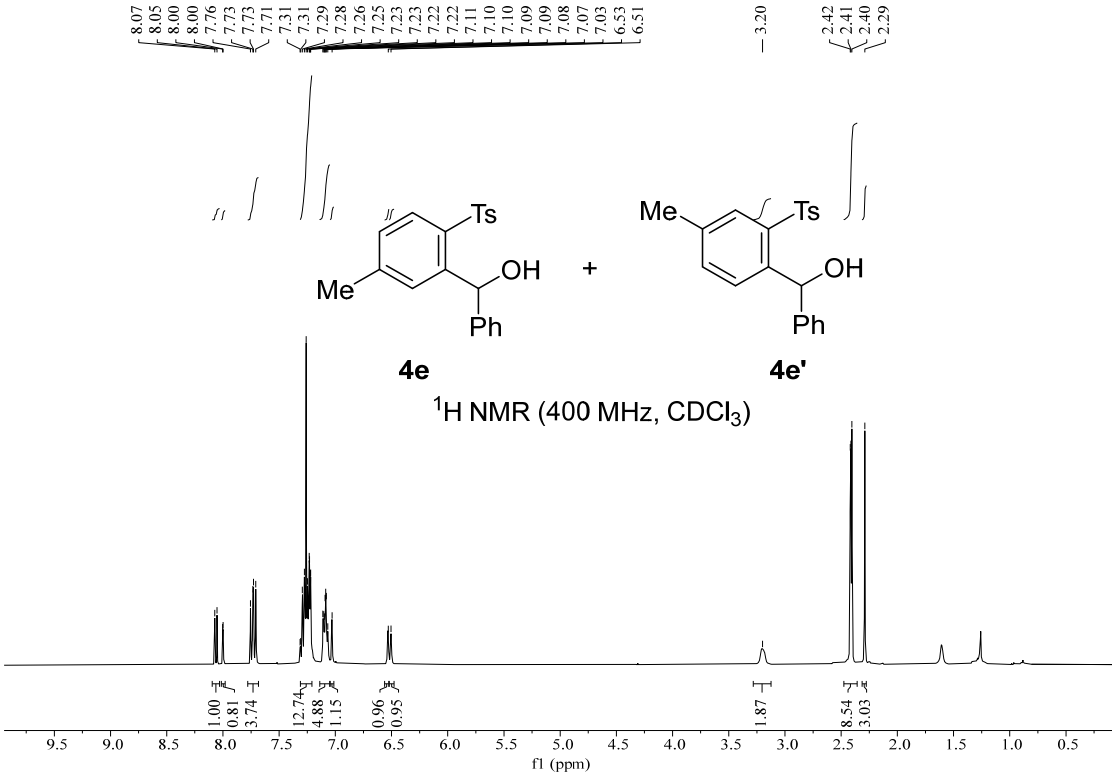
4d

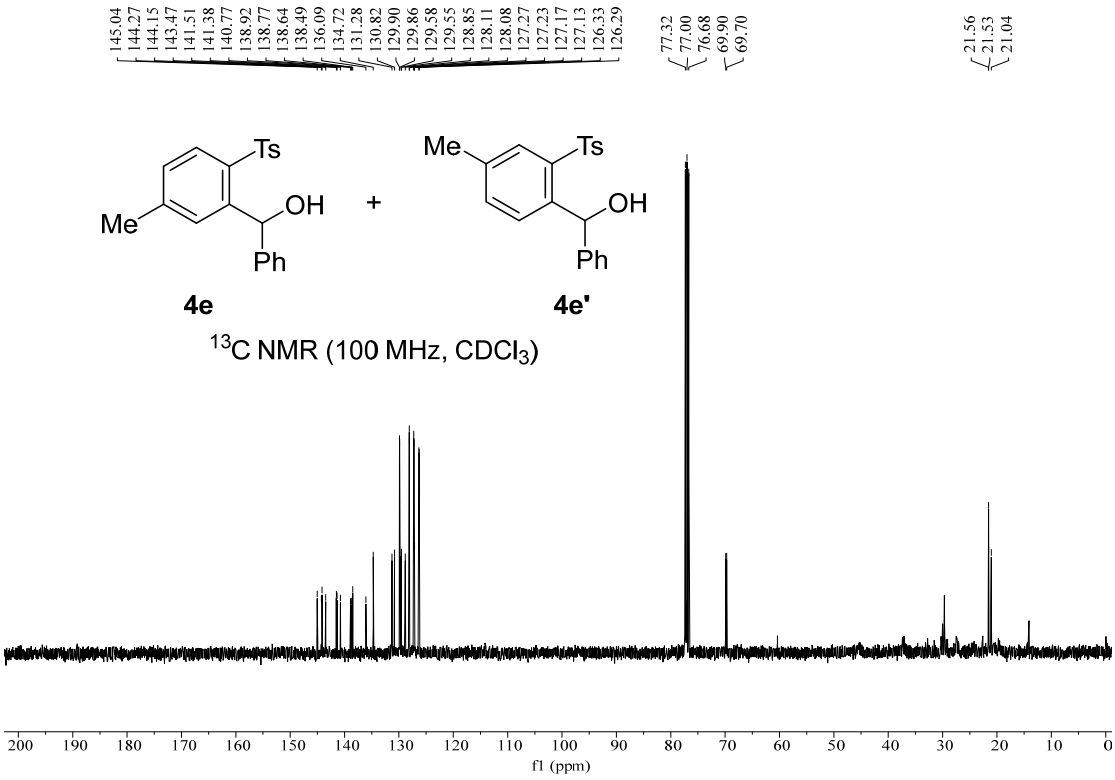
^{1}H NMR (400 MHz, $CDCl_3$)



4d

^{13}C NMR (100 MHz, $CDCl_3$)


8.07
8.05
8.00
8.00
7.76
7.73
7.73
7.71
7.31
7.31
7.29
7.28
7.26
7.25
7.23
7.23
7.22
7.22
7.11
7.10
7.10
7.09
7.09
7.08
7.07
7.03
6.53
6.51
3.20
2.42
2.41
2.40
2.29
Ts
OH
Me
Ph
+
Me
Ts
OH
Ph
4e
4e'
1H NMR (400 MHz, CDCl3)
1.00
0.81
3.74
12.74
4.88
1.15
0.96
0.95
1.87
8.54
3.03
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
f1 (ppm)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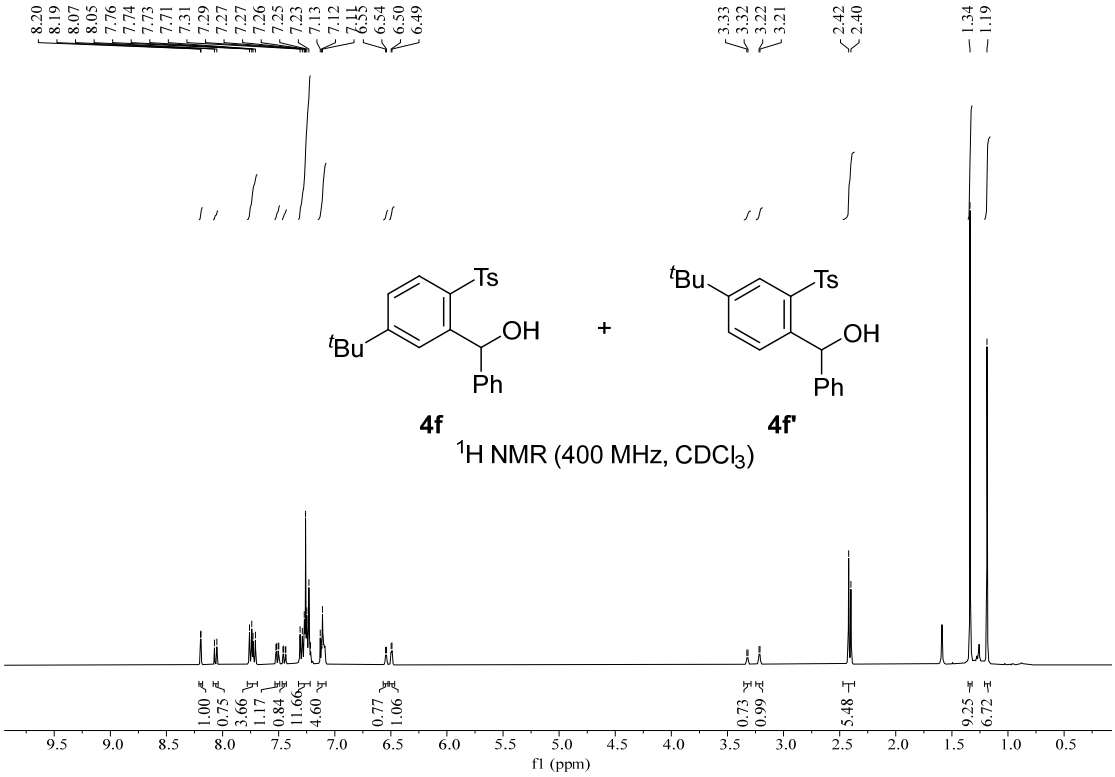
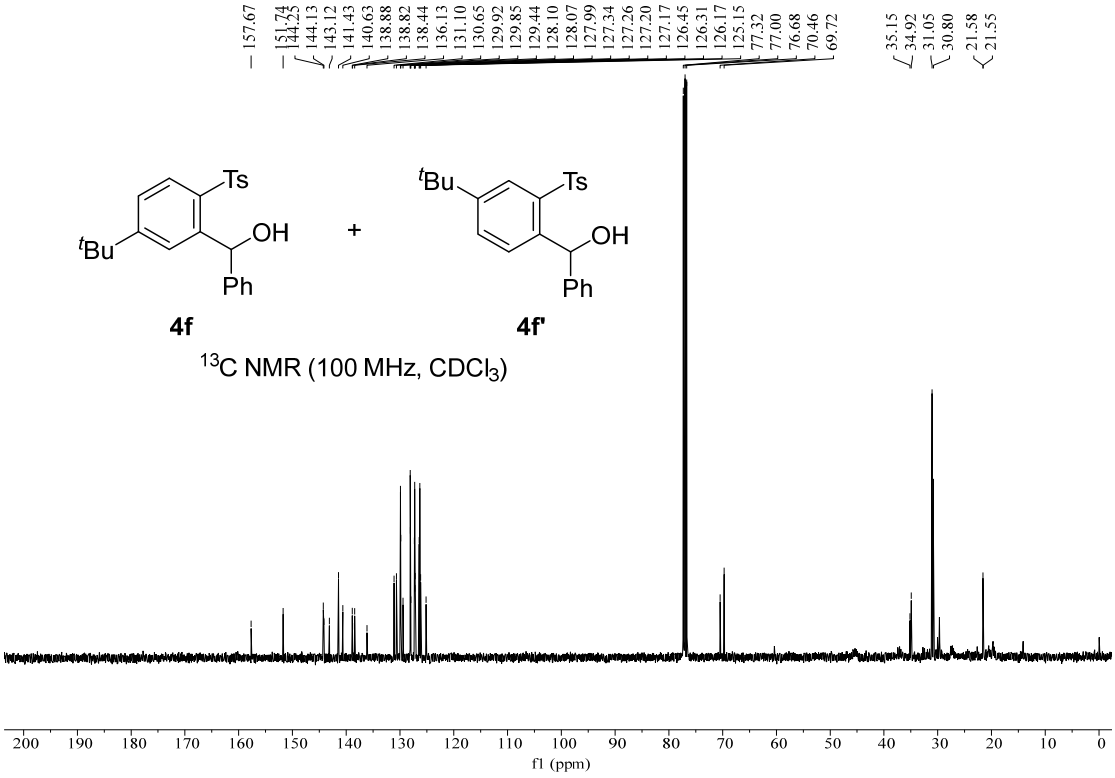
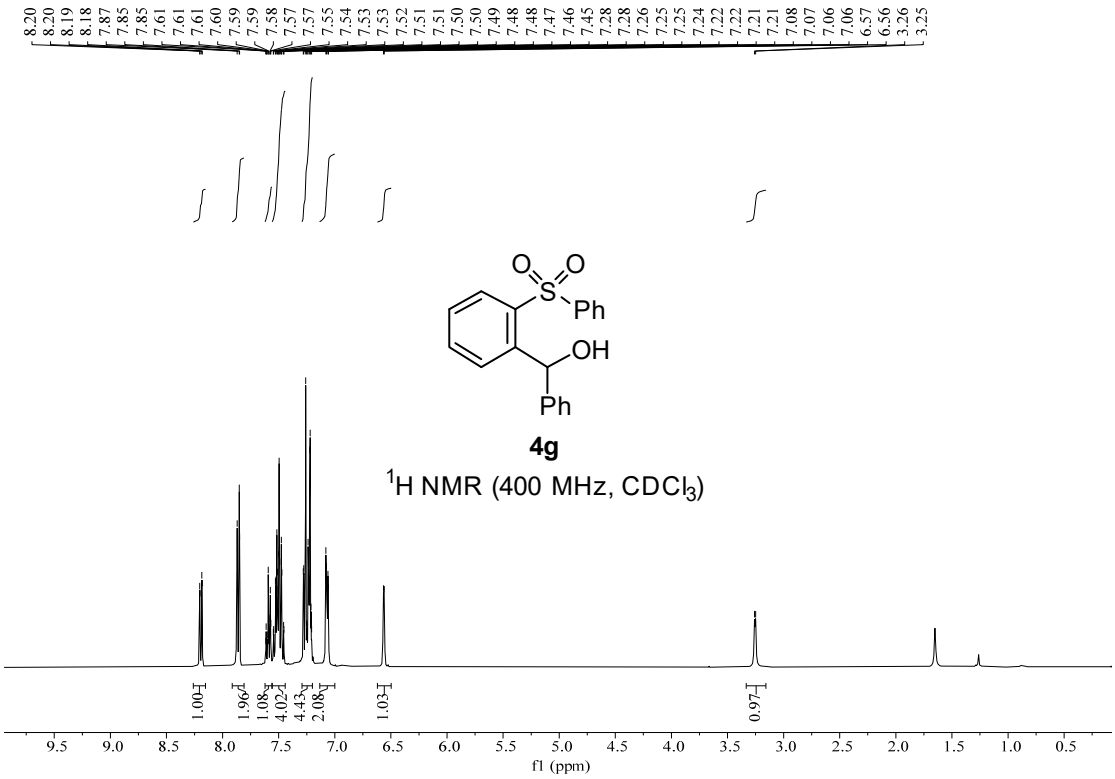
144.27
144.15
143.47
141.51
141.38
140.77
138.92
138.77
138.64
138.49
136.09
134.72
131.28
130.82
129.90
129.86
129.58
129.55
128.85
128.11
128.08
127.27
127.23
127.17
127.13
126.33
126.29
77.32
77.00
76.68
69.90
69.70
21.56
21.53
21.04
Ts
OH
Me
Ph
+
Me
Ts
OH
Ph
4e
4e'
13C NMR (100 MHz, CDCl3)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

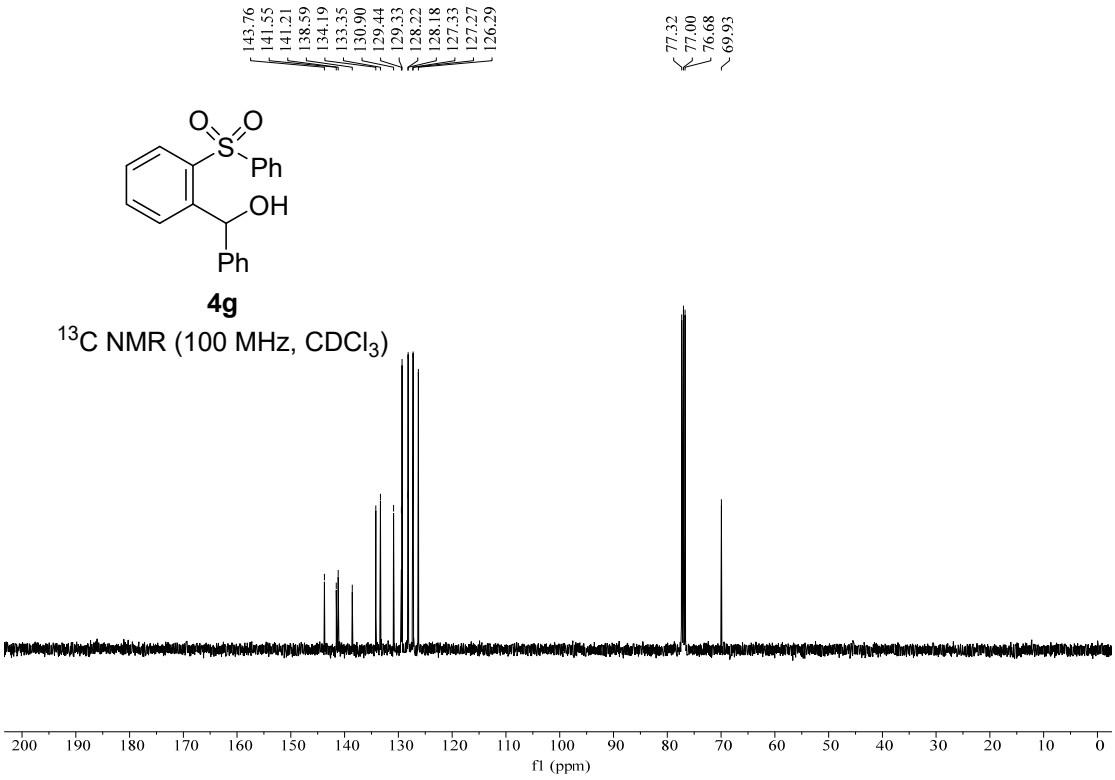

8.20
8.19
8.07
8.05
7.76
7.74
7.73
7.71
7.31
7.29
7.27
7.27
7.26
7.25
7.23
7.13
7.12
7.11
6.55
6.54
6.50
6.49
3.33
3.32
3.22
3.21
2.42
2.40
1.34
1.19
Ts
OH
Ph
tBu
+
4f
4f'
1H NMR (400 MHz, CDCl3)
1.00
0.75
3.66
1.17
0.84
11.66
4.60
0.77
1.06
0.73
0.99
5.48
9.25
6.72
f1 (ppm)


157.67
151.74
144.23
144.13
143.12
141.43
140.63
138.88
138.82
138.44
136.13
131.10
130.65
129.92
129.85
129.44
128.10
128.07
127.99
127.34
127.26
127.20
127.17
126.45
126.31
126.17
125.15
77.32
77.00
76.68
70.46
69.72
35.15
34.92
31.05
30.80
21.58
21.55
Ts
OH
Ph
tBu
+
4f
4f'
13C NMR (100 MHz, CDCl3)
f1 (ppm)



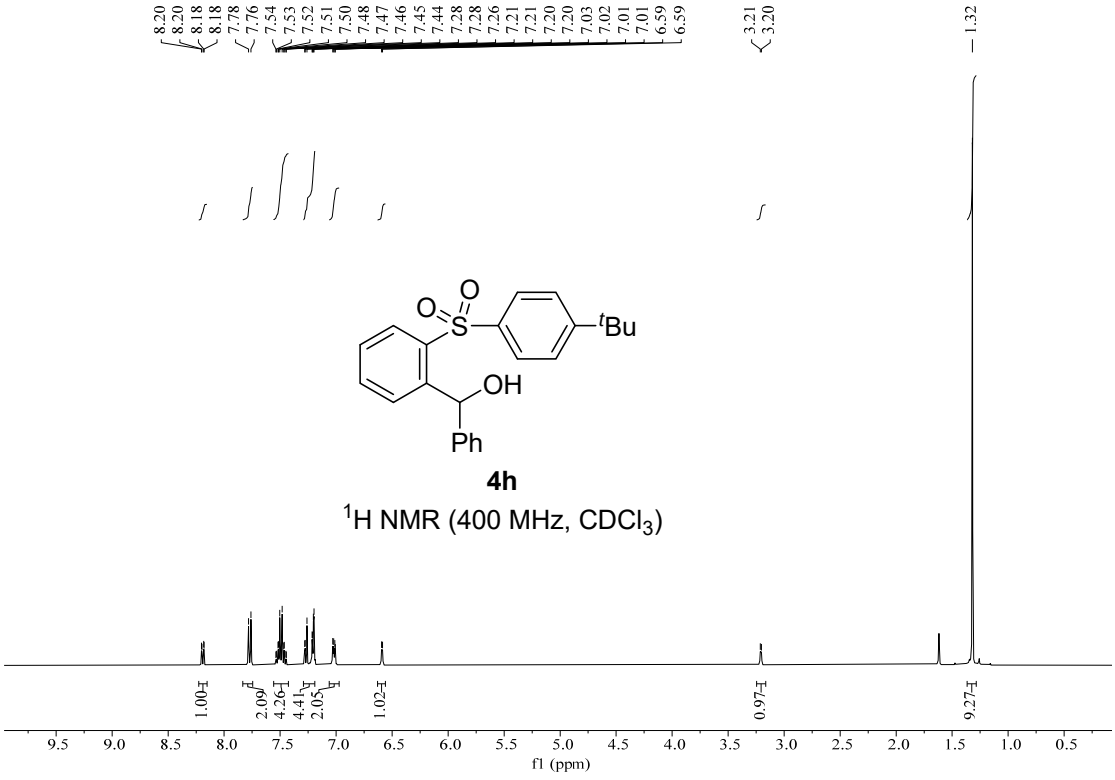
4g

^{1}H NMR (400 MHz, $CDCl_3$)



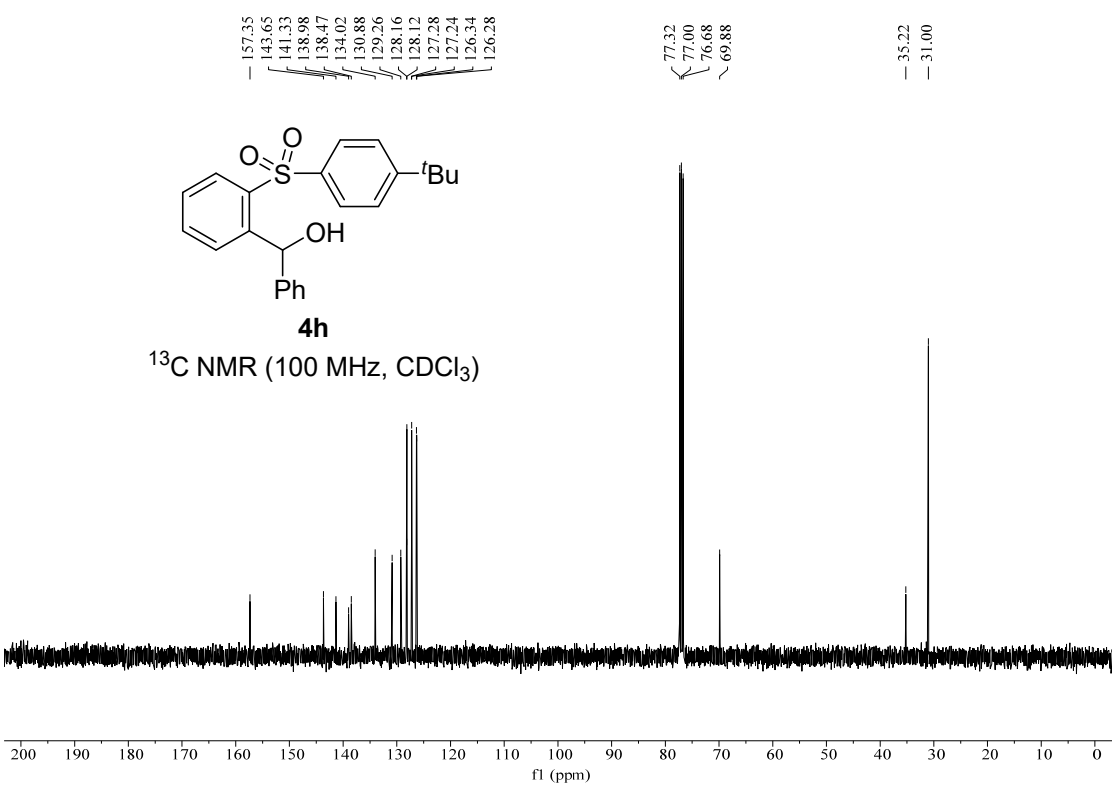
4g

^{13}C NMR (100 MHz, $CDCl_3$)



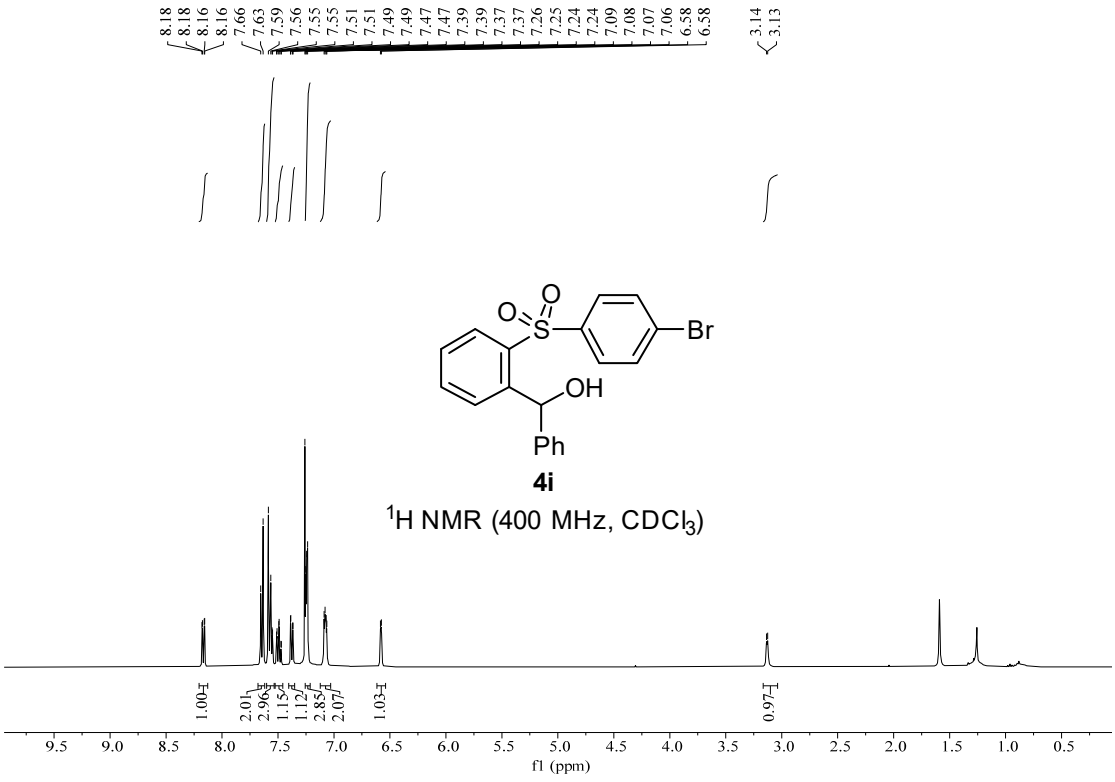
4h

^{1}H NMR (400 MHz, $CDCl_3$)



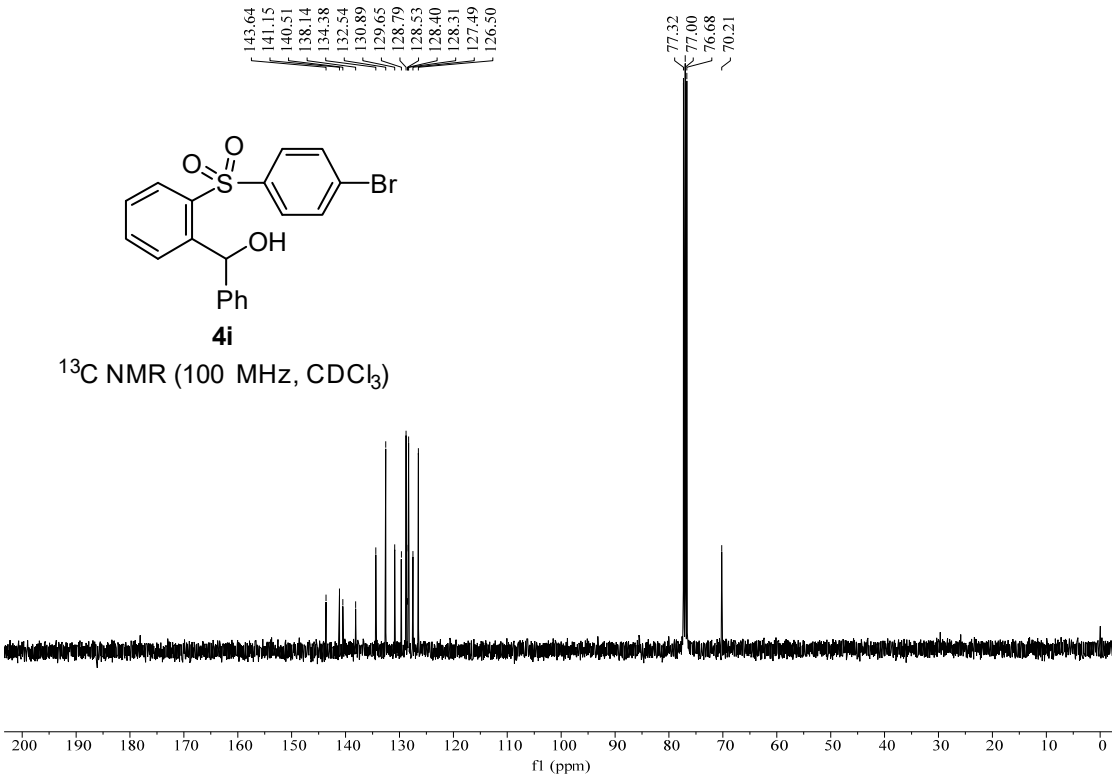
4h

^{13}C NMR (100 MHz, $CDCl_3$)



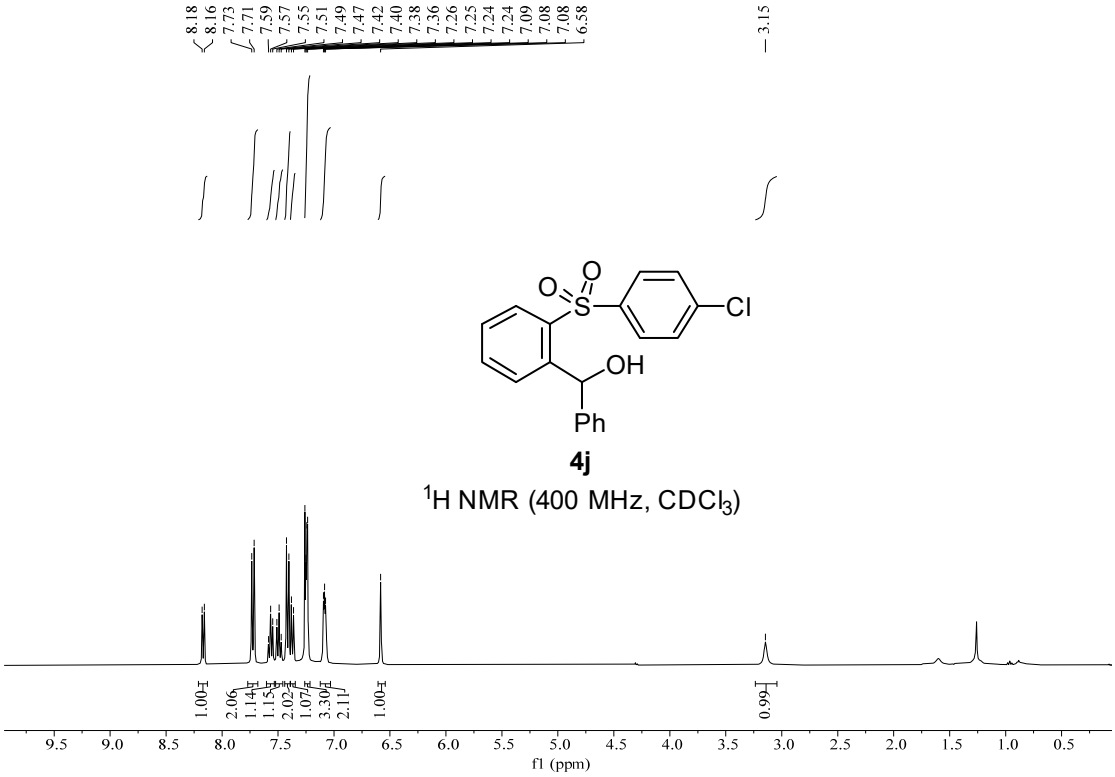
4i

^{1}H NMR (400 MHz, $CDCl_3$)



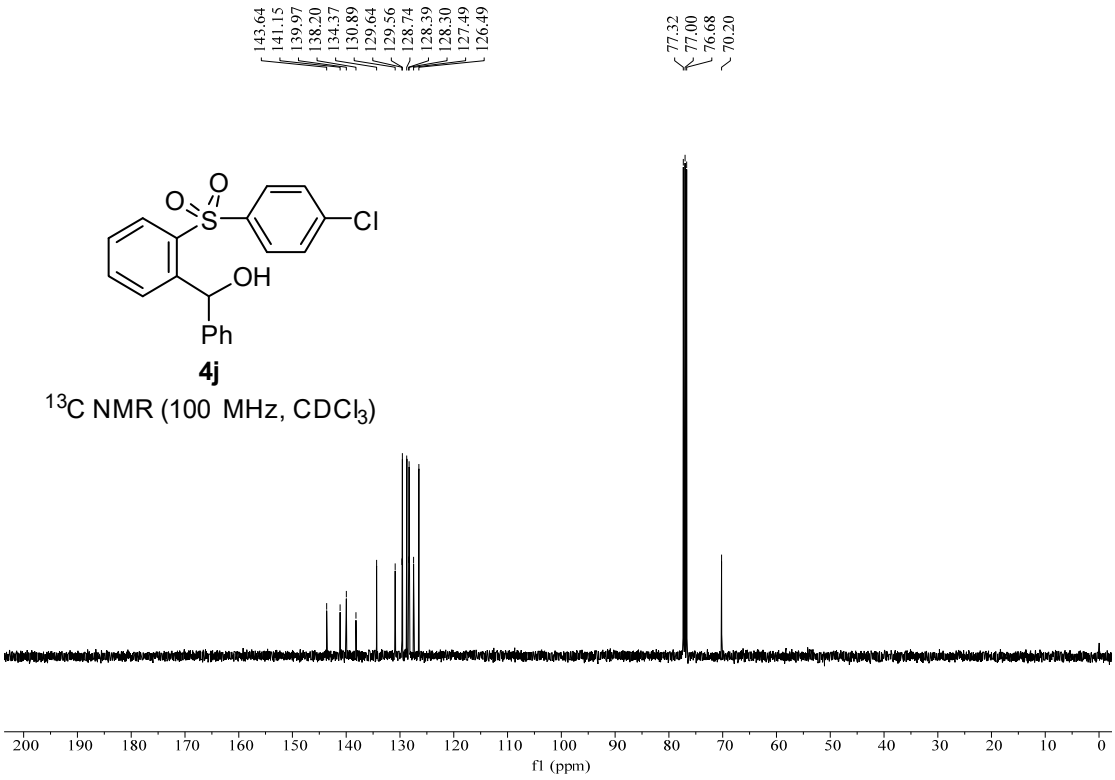
4i

^{13}C NMR (100 MHz, $CDCl_3$)



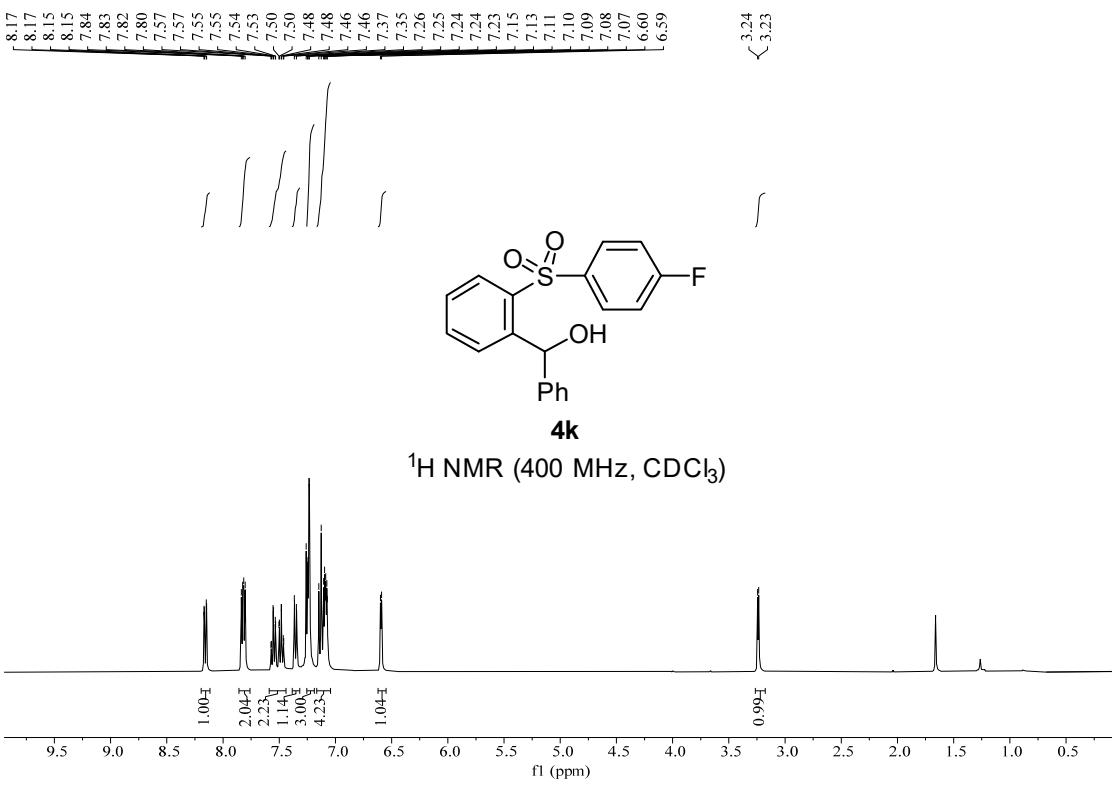
4j

^{1}H NMR (400 MHz, $CDCl_3$)



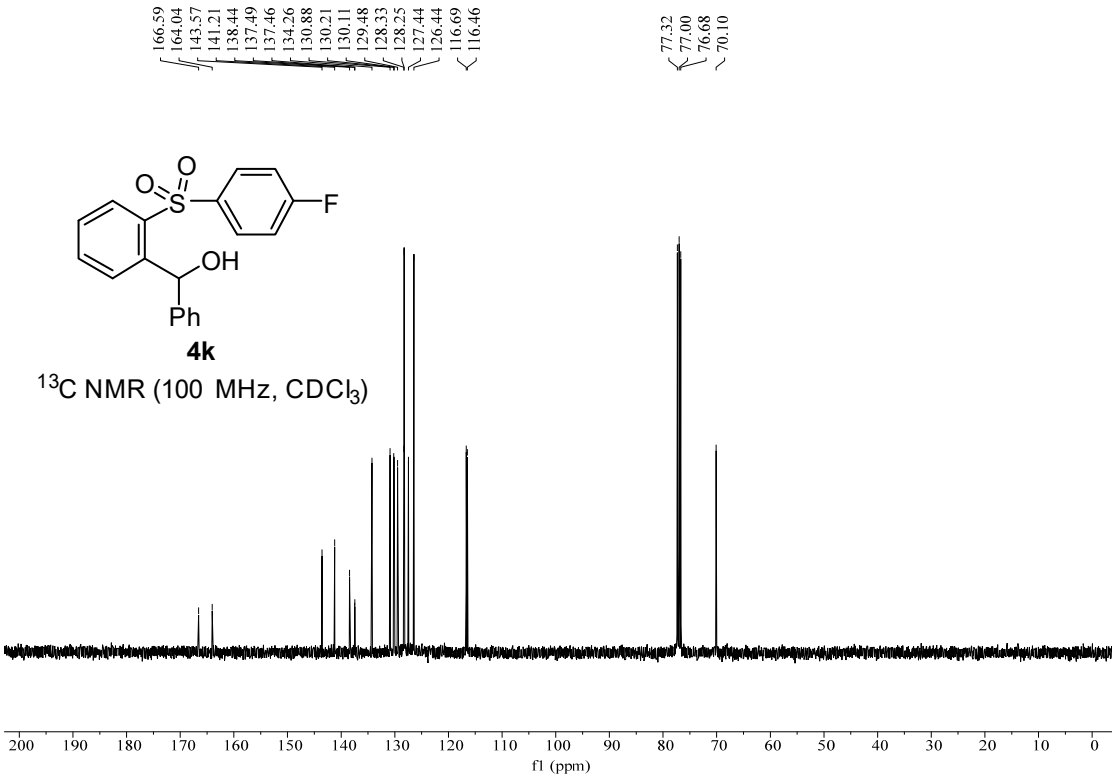
4j

^{13}C NMR (100 MHz, $CDCl_3$)



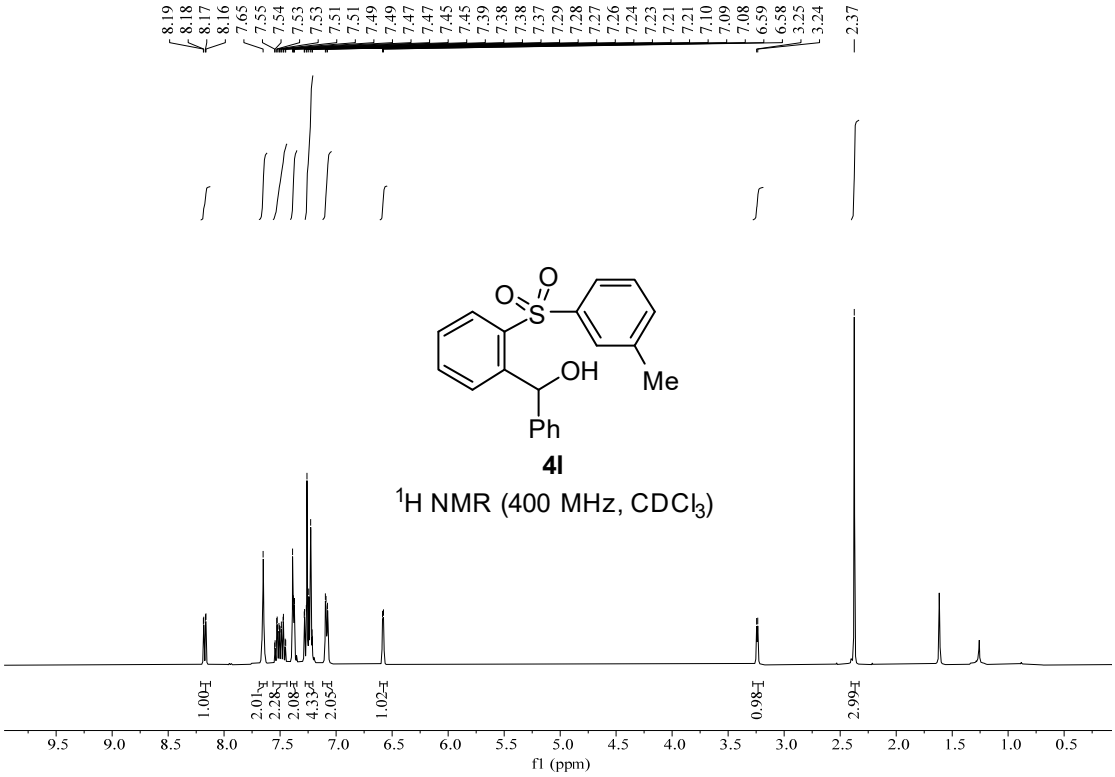
4k

^{1}H NMR (400 MHz, $CDCl_3$)



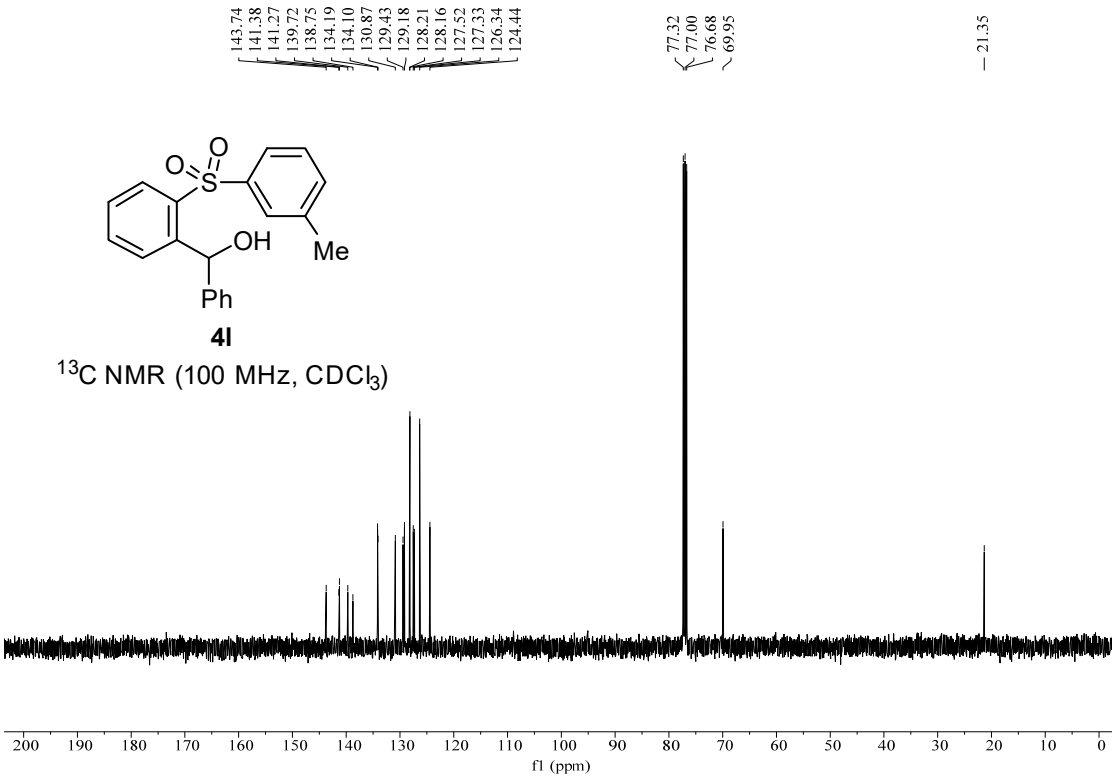
4k

^{13}C NMR (100 MHz, $CDCl_3$)



4l

^{1}H NMR (400 MHz, $CDCl_3$)



4l

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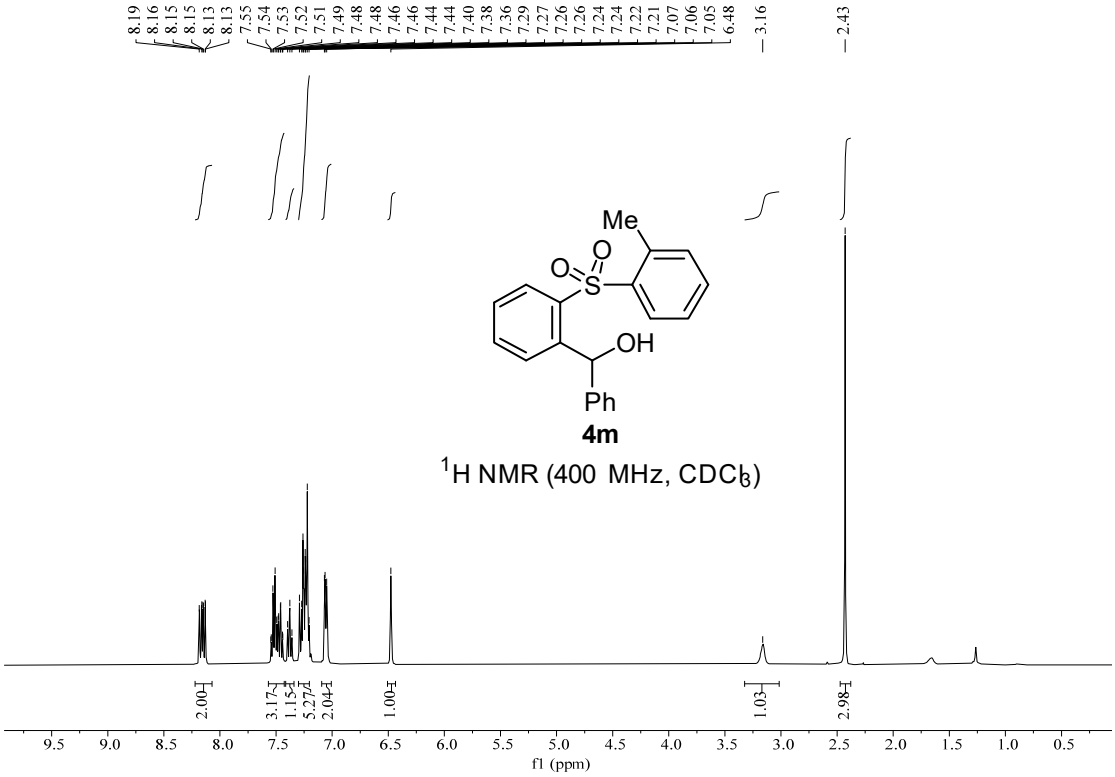
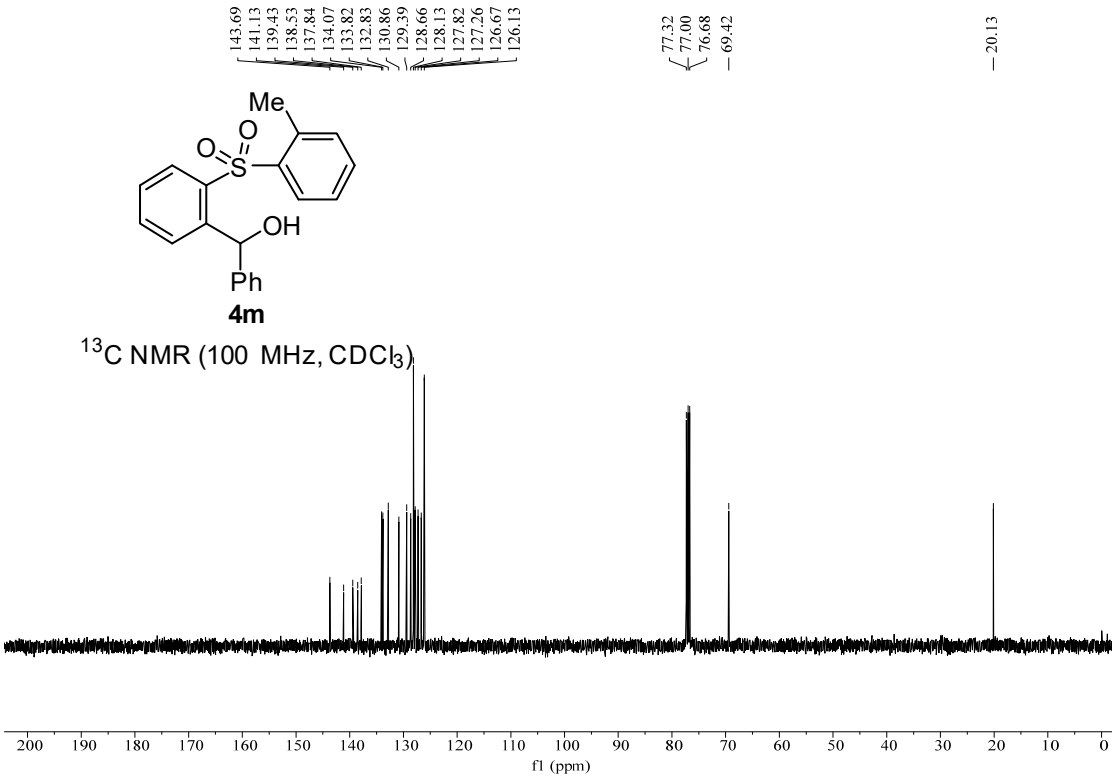


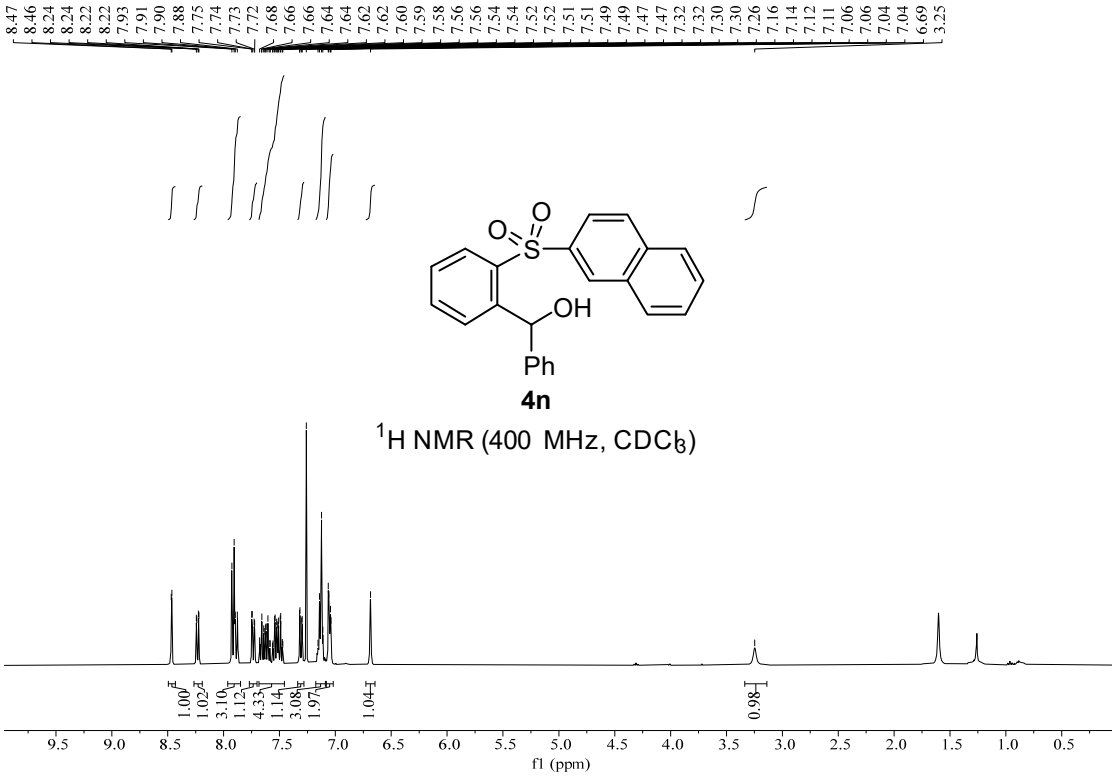
4m

^{1}H NMR (400 MHz, $CDCl_3$)



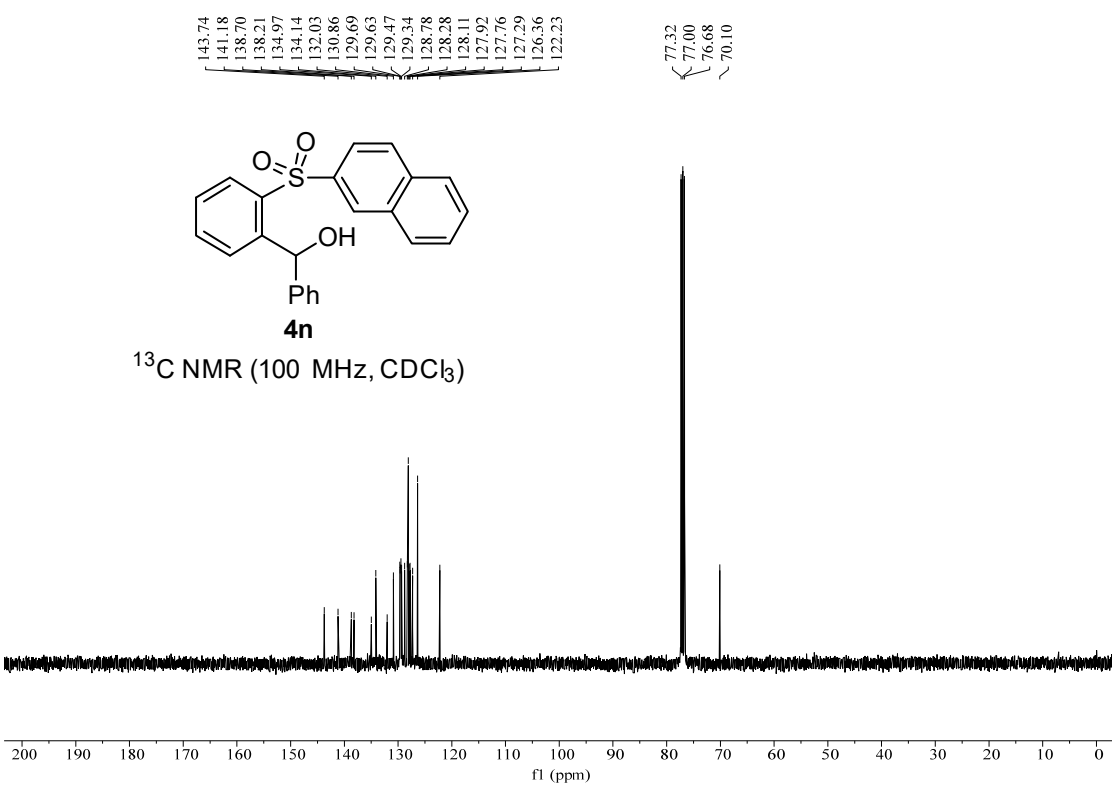
4m

^{13}C NMR (100 MHz, $CDCl_3$)



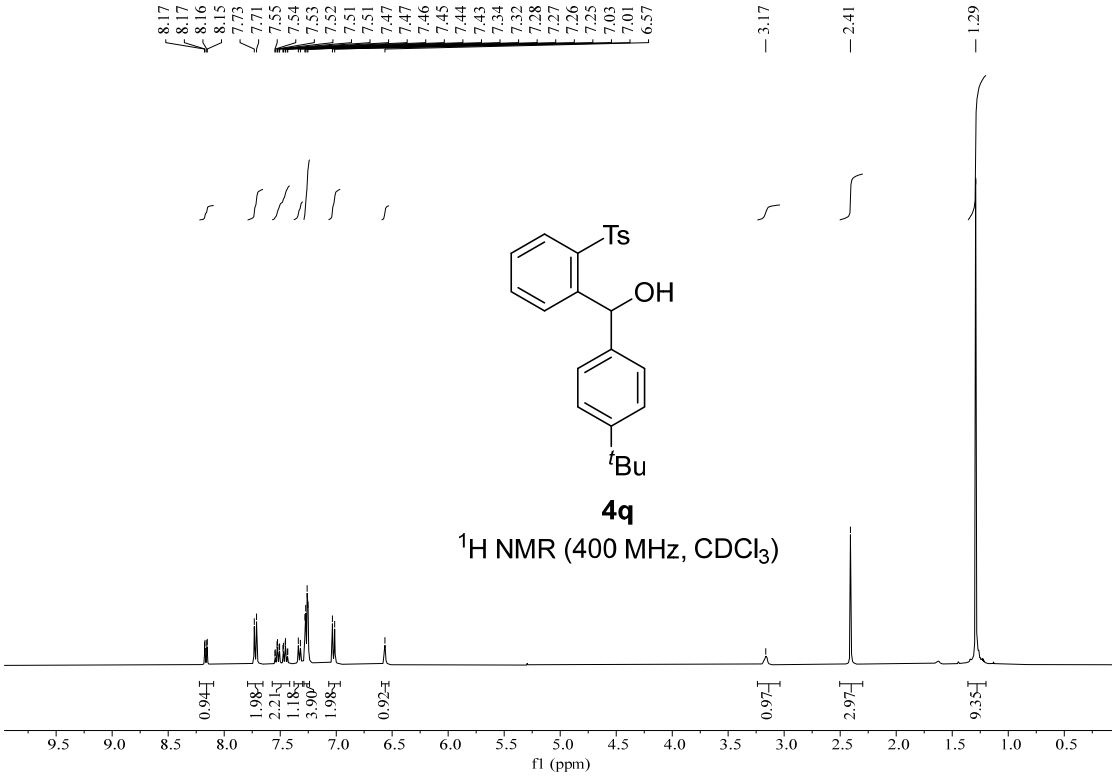
4n

^{1}H NMR (400 MHz, $CDCl_3$)



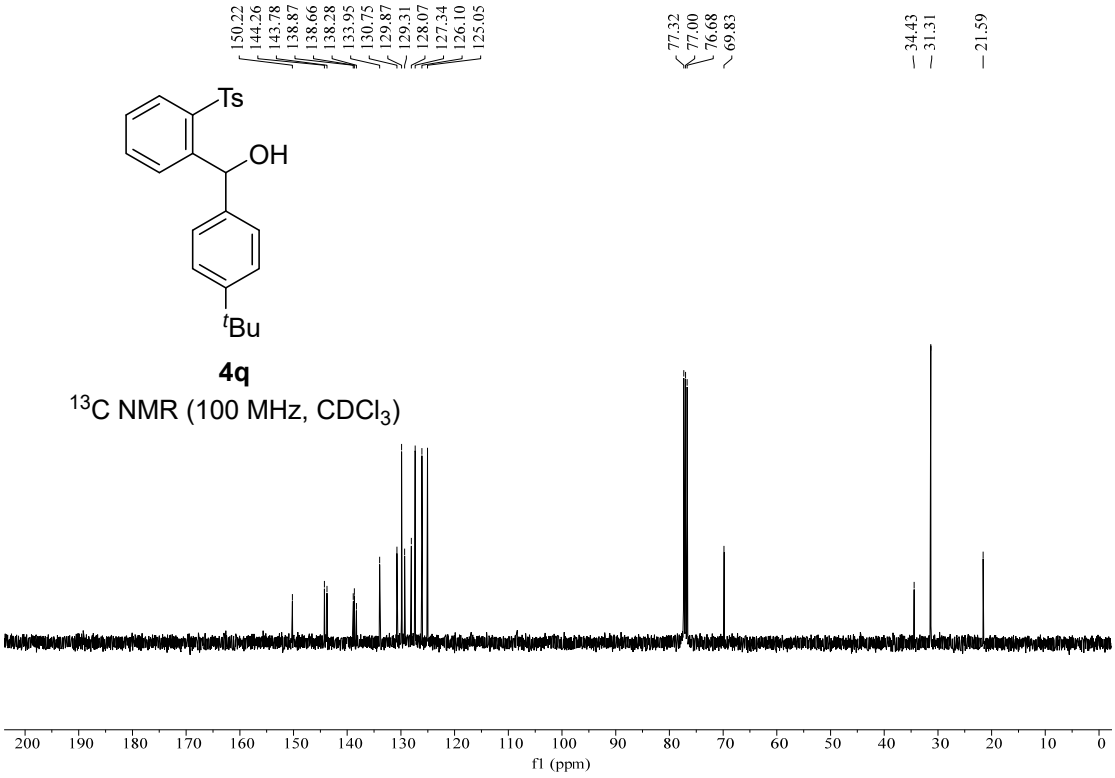
4n

^{13}C NMR (100 MHz, $CDCl_3$)



4q

^{1}H NMR (400 MHz, $CDCl_3$)



4q

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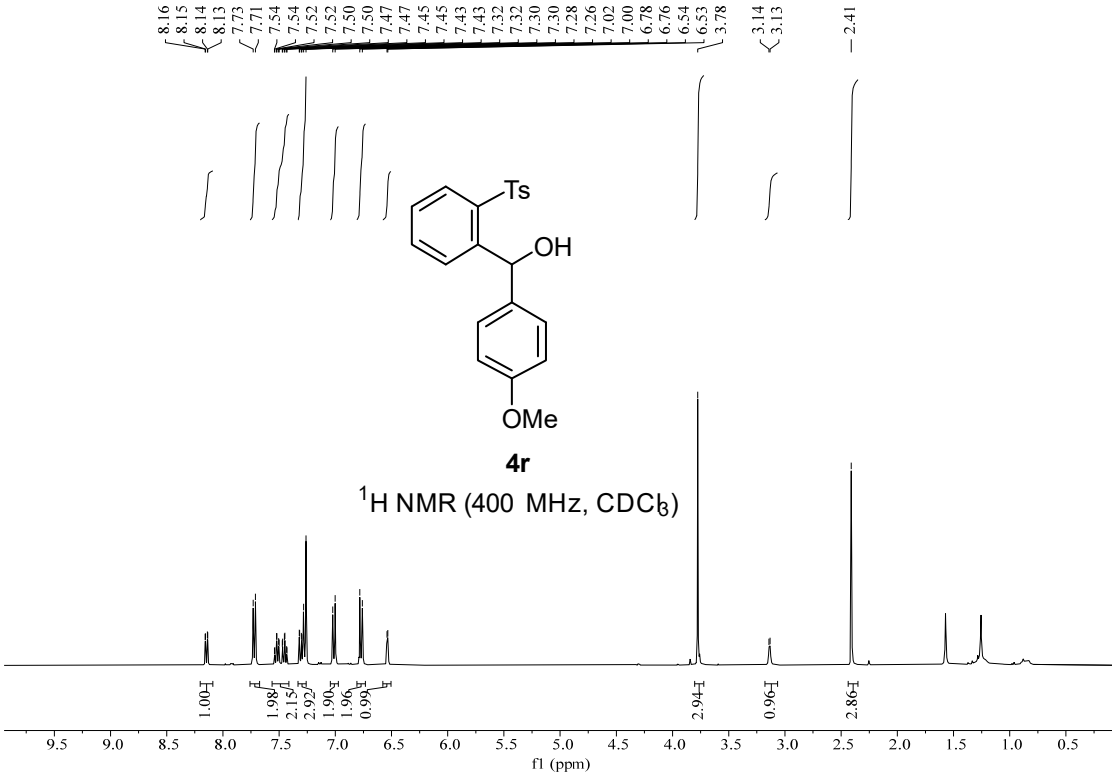
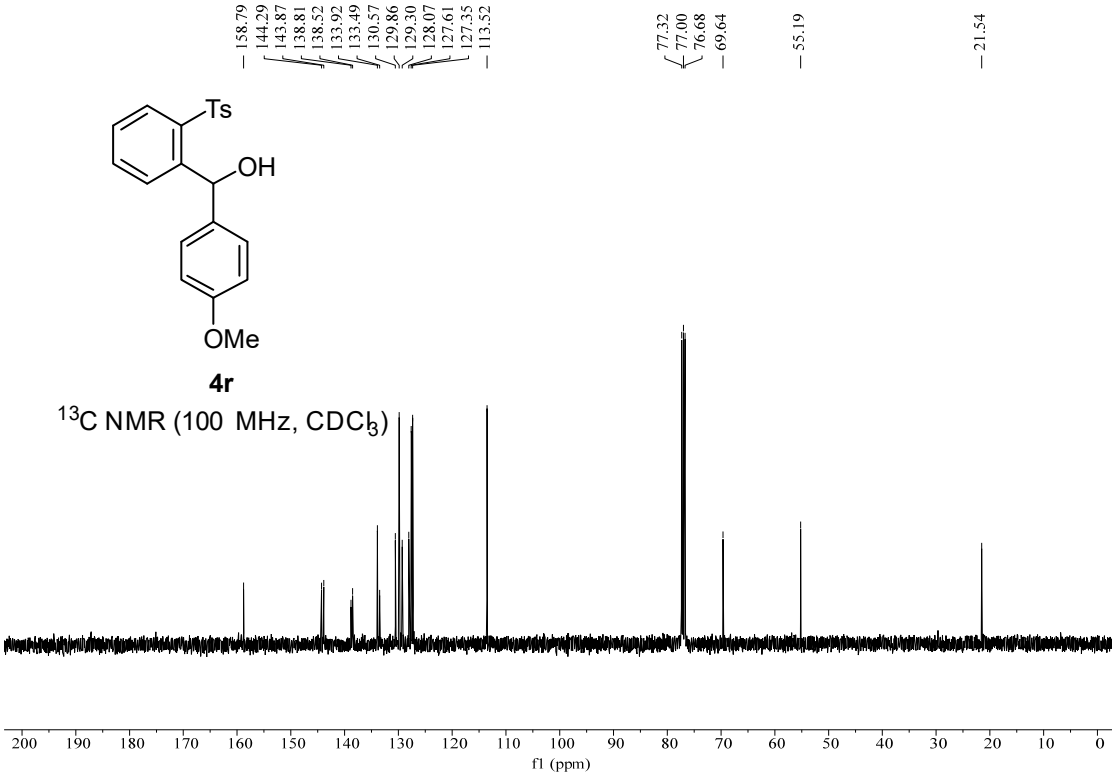
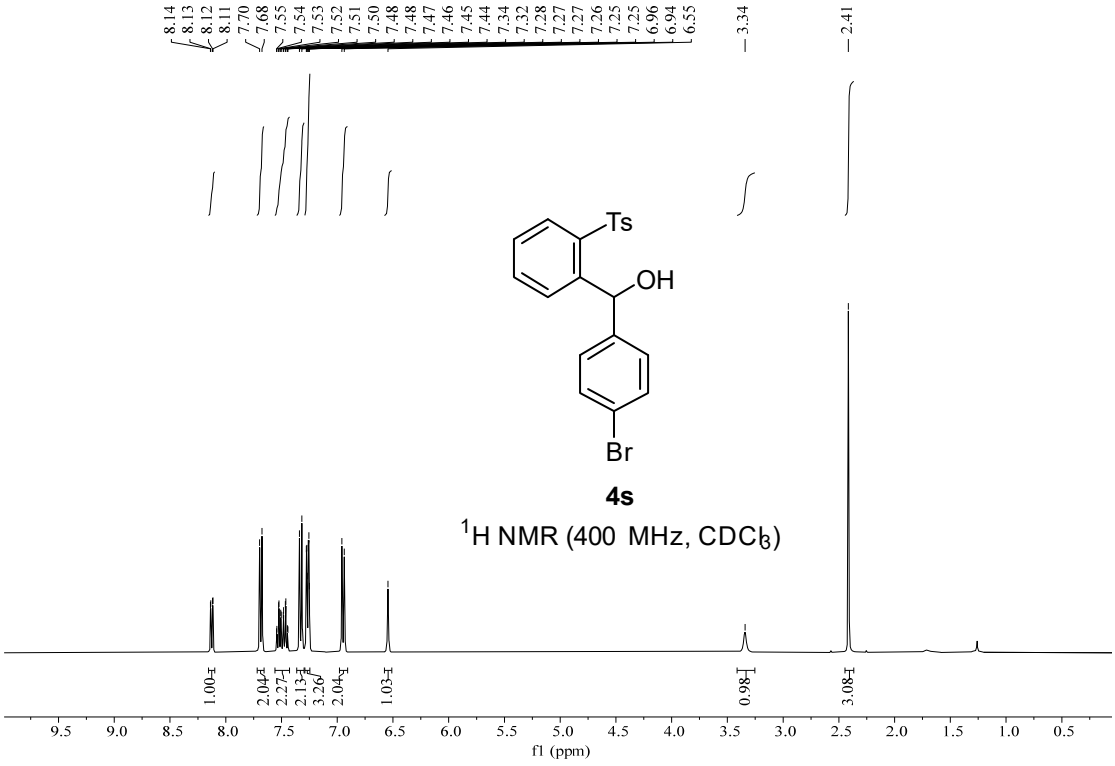
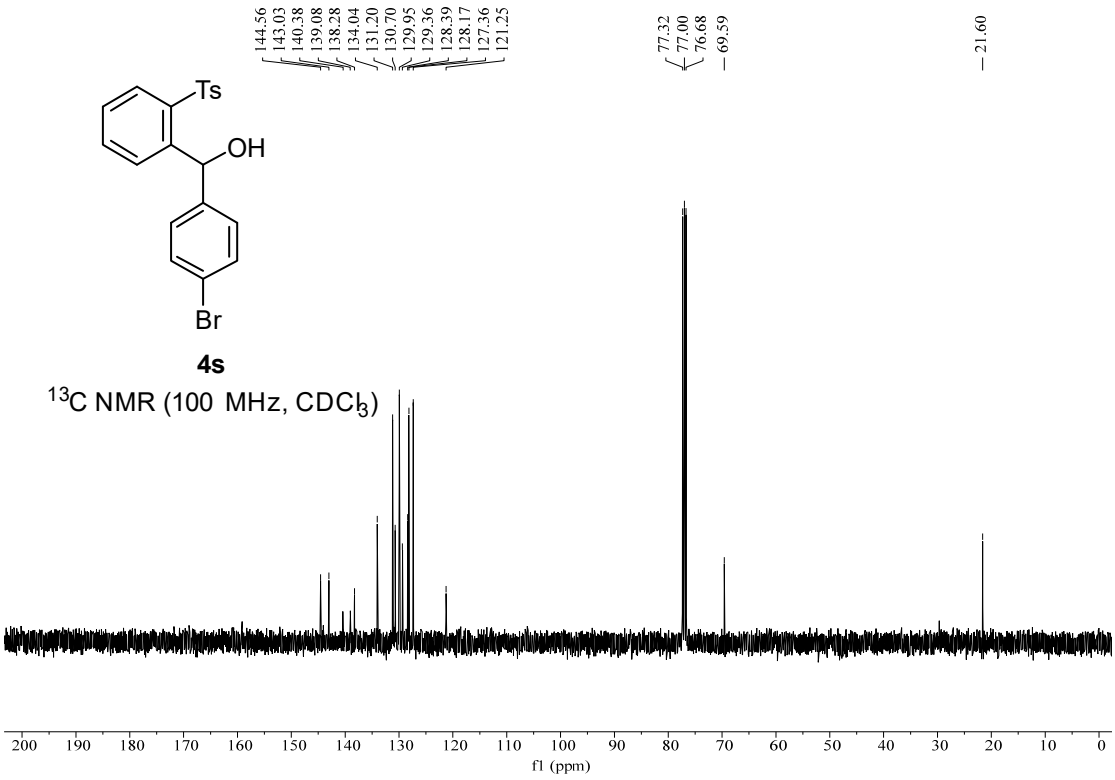
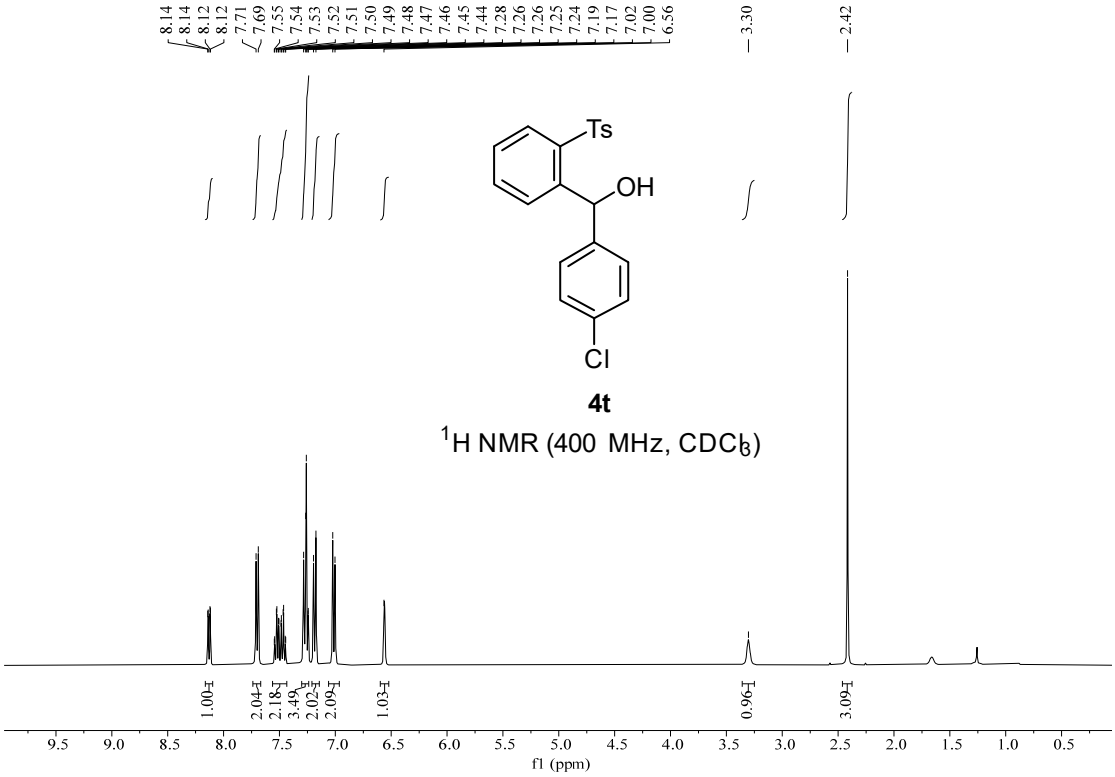

8.16
8.15
8.14
8.13
7.73
7.71
7.54
7.54
7.52
7.52
7.50
7.50
7.47
7.47
7.45
7.45
7.43
7.43
7.32
7.30
7.28
7.26
7.02
7.00
6.78
6.76
6.54
6.53
3.78
3.14
3.13
2.41
Ts
OH
OMe
4r
1H NMR (400 MHz, CDCl3)
1.00
1.98
2.15
2.92
1.90
1.96
0.99
2.94
0.96
2.86
f1 (ppm)


158.79
144.29
143.87
138.81
138.52
133.92
133.49
130.57
129.86
129.30
128.07
127.61
127.35
113.52
77.32
77.00
76.68
69.64
55.19
21.54
Ts
OH
OMe
4r
13C NMR (100 MHz, CDCl3)
f1 (ppm)

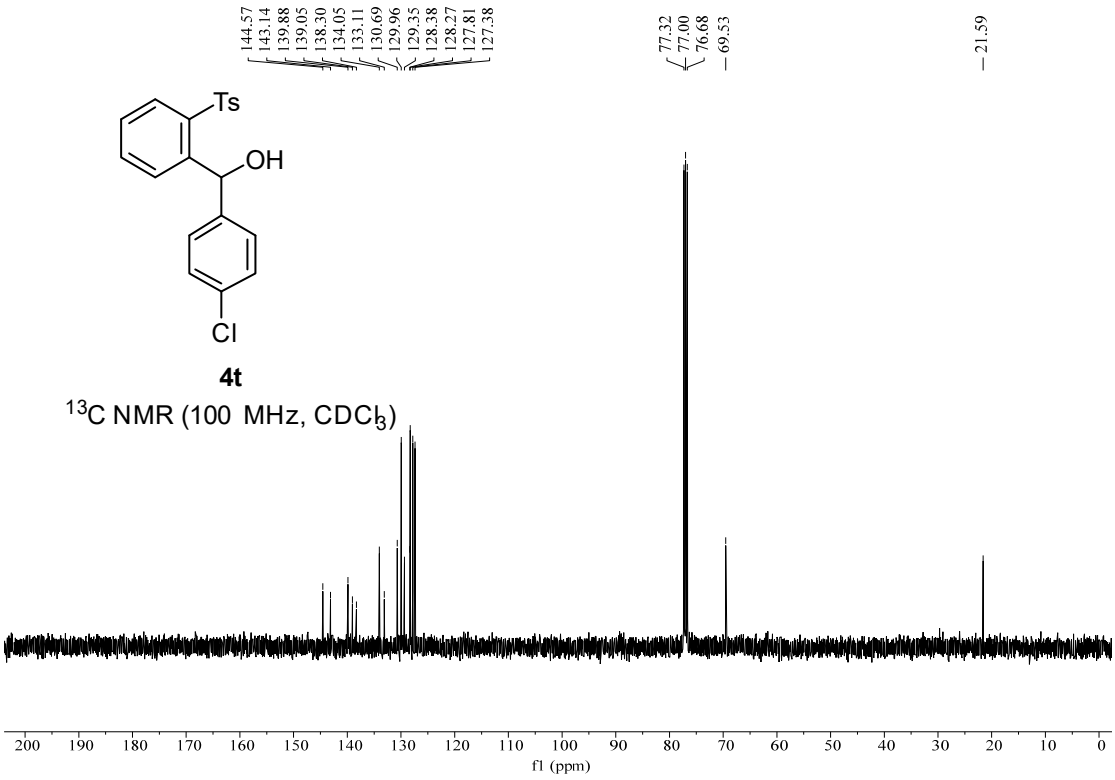

8.14
8.13
8.12
8.11
7.70
7.68
7.55
7.54
7.53
7.52
7.51
7.50
7.48
7.47
7.46
7.45
7.44
7.34
7.32
7.28
7.27
7.27
7.26
7.25
7.25
6.96
6.94
6.55
3.34
2.41
Ts
OH
Br
4s
1H NMR (400 MHz, CDCl3)
1.00
2.04
2.27
2.13
3.26
2.04
1.03
0.98
3.08
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


144.56
143.03
140.38
139.08
138.28
134.04
131.20
130.70
129.95
129.36
128.39
128.17
127.36
121.25
77.32
77.00
76.68
69.59
21.60
Ts
OH
Br
4s
13C NMR (100 MHz, CDCl3)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



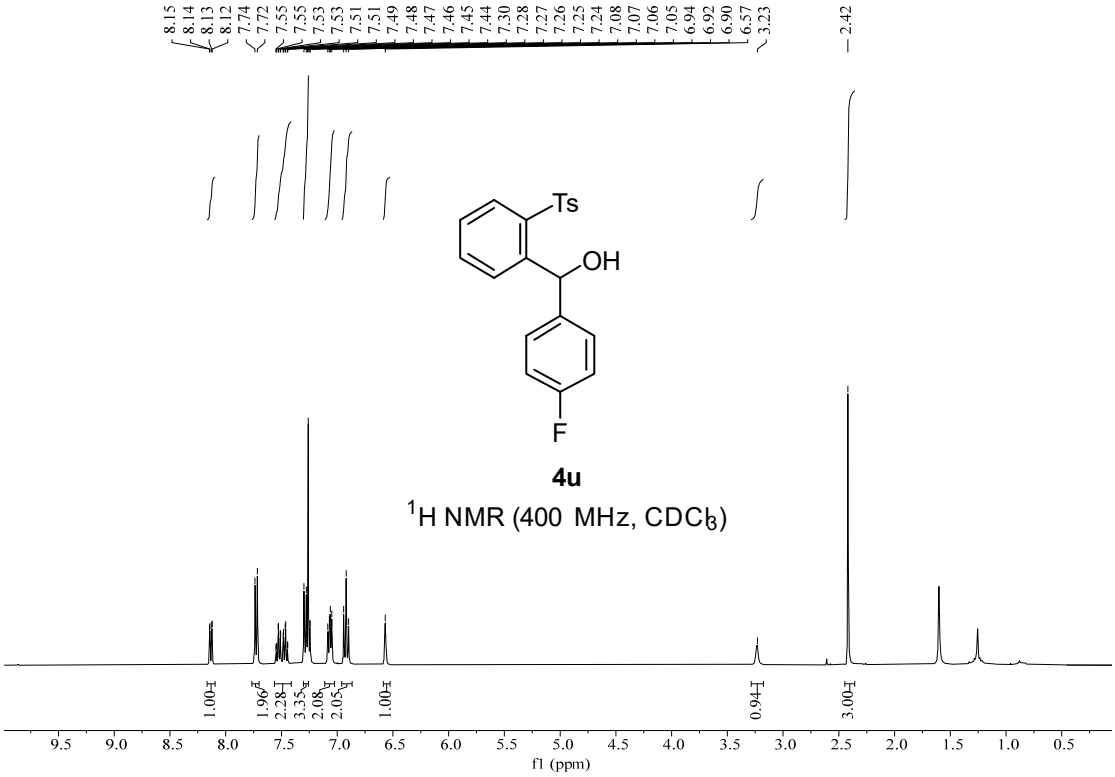
4t

^{1}H NMR (400 MHz, $CDCl_3$)



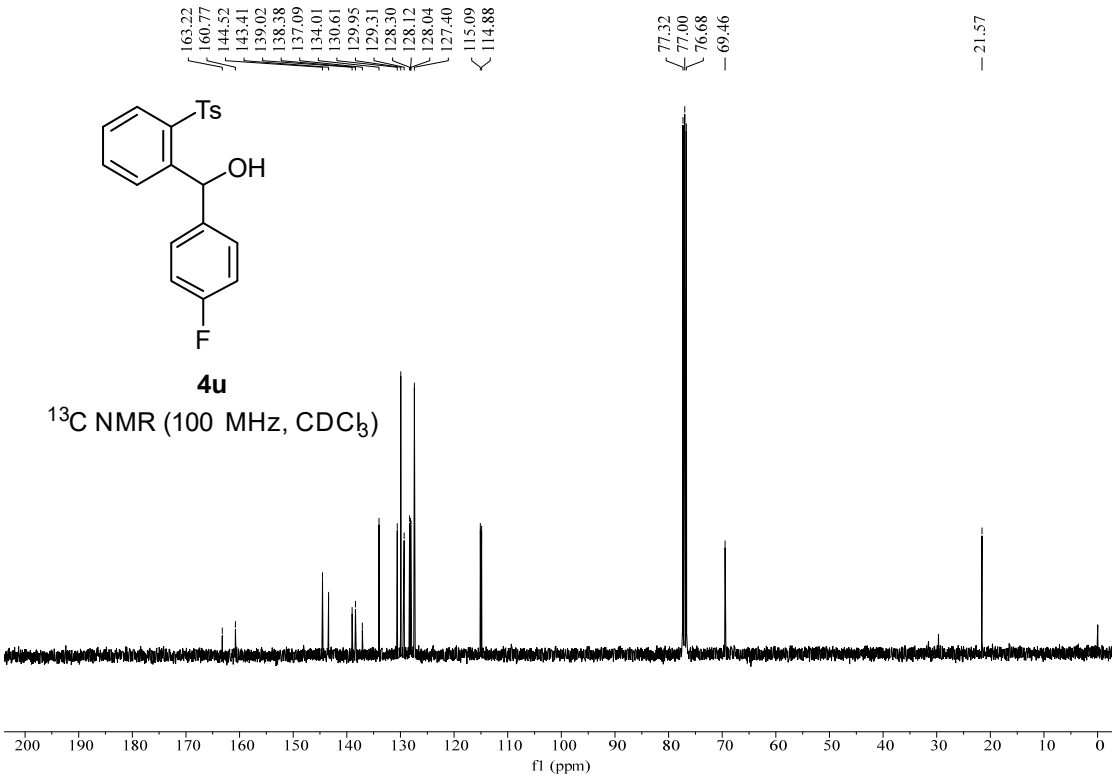
4t

^{13}C NMR (100 MHz, $CDCl_3$)



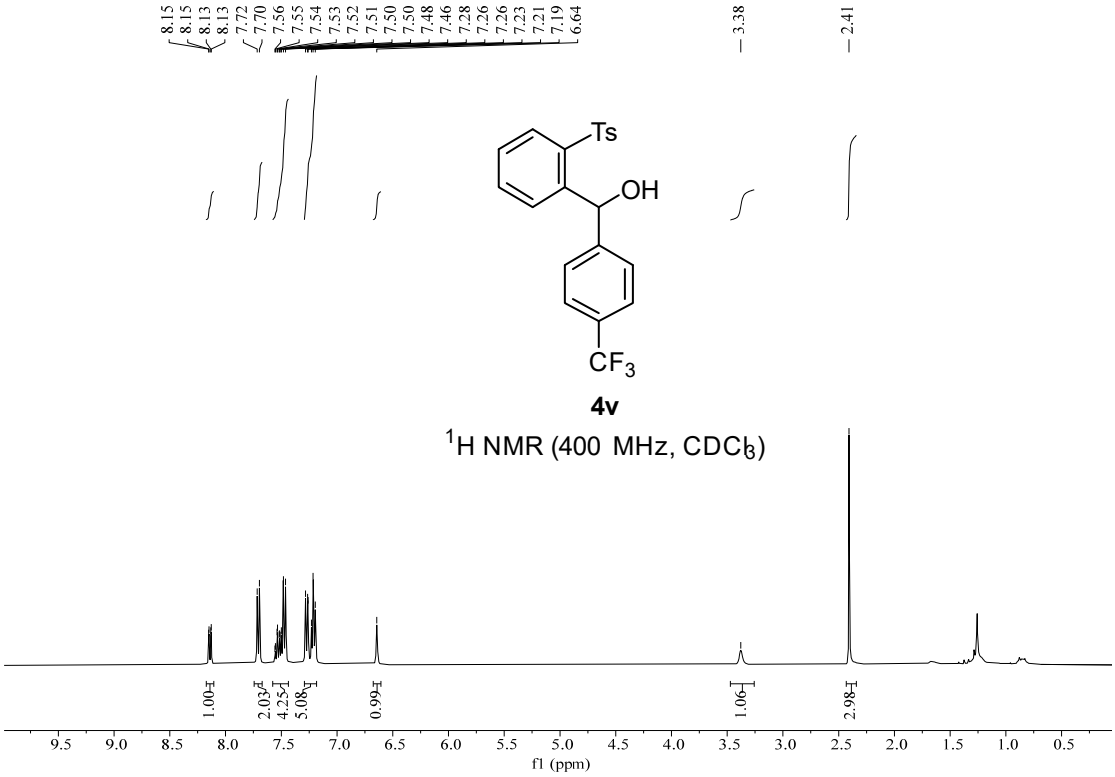
4u

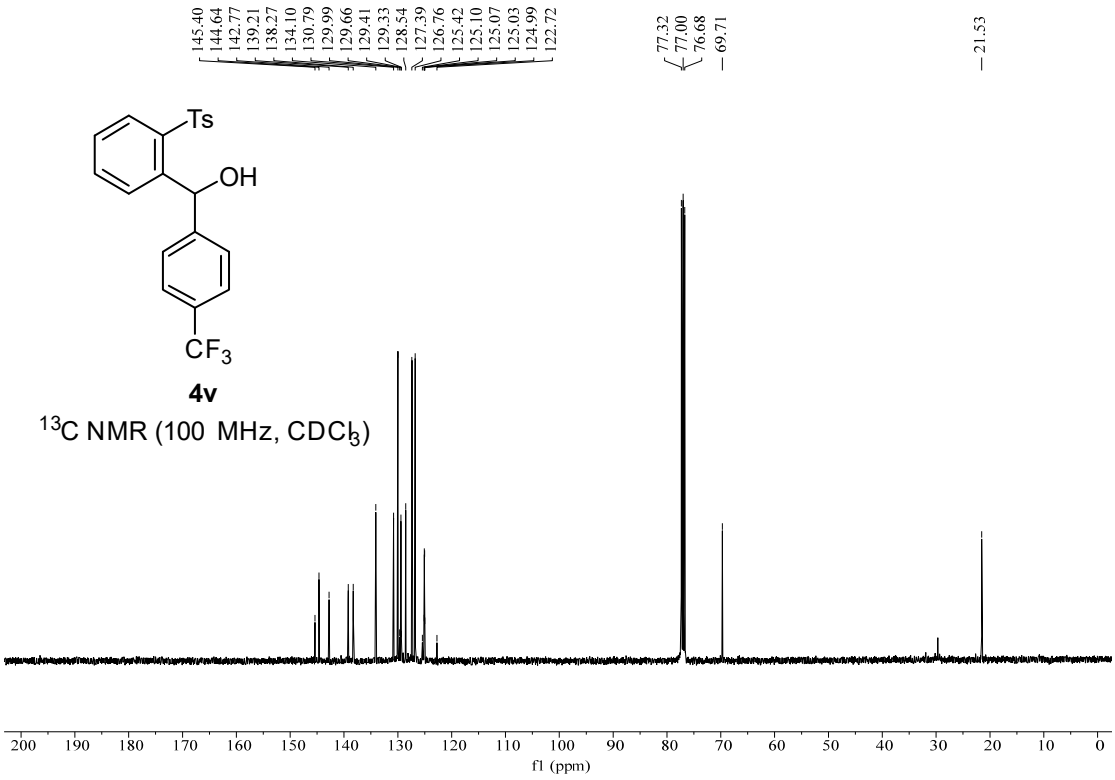
^{1}H NMR (400 MHz, $CDCl_3$)

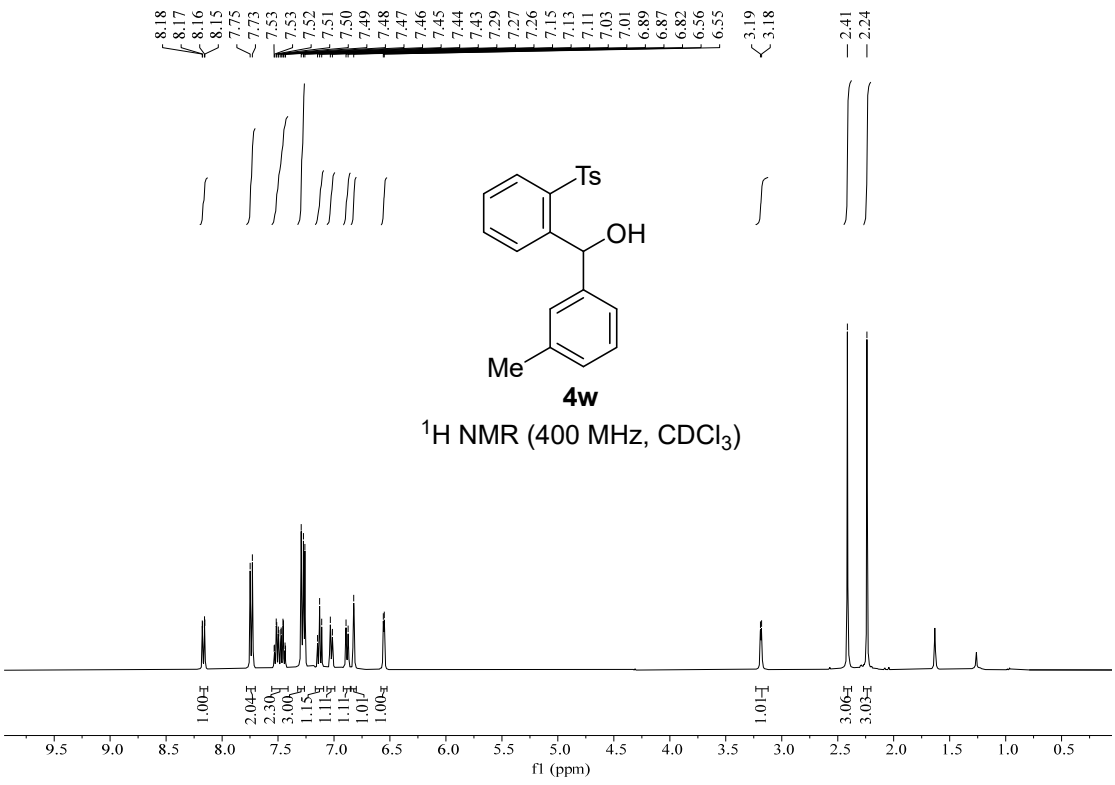


4u

^{13}C NMR (100 MHz, $CDCl_3$)

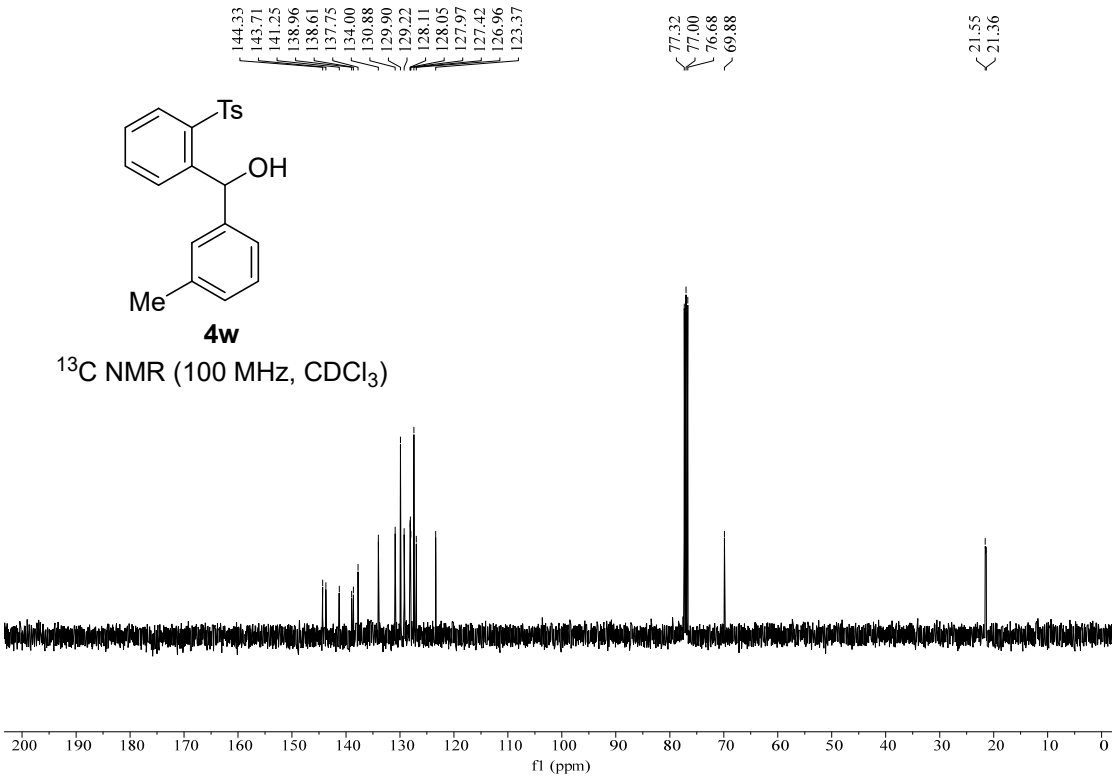

8.15
8.15
8.13
8.13
7.72
7.70
7.56
7.55
7.54
7.53
7.52
7.51
7.50
7.50
7.48
7.46
7.28
7.26
7.26
7.23
7.21
7.19
6.64
3.38
2.41
Ts
OH
CF3
4v
1H NMR (400 MHz, CDCl3)
1.00
2.03
4.25
5.08
0.99
1.06
2.98
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


145.40
144.64
142.77
139.21
138.27
134.10
130.79
129.99
129.66
129.41
129.33
128.54
127.39
126.76
125.42
125.10
125.07
125.03
124.99
122.72
77.32
77.00
76.68
69.71
21.53
Ts
OH
CF3
4v
13C NMR (100 MHz, CDCl3)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



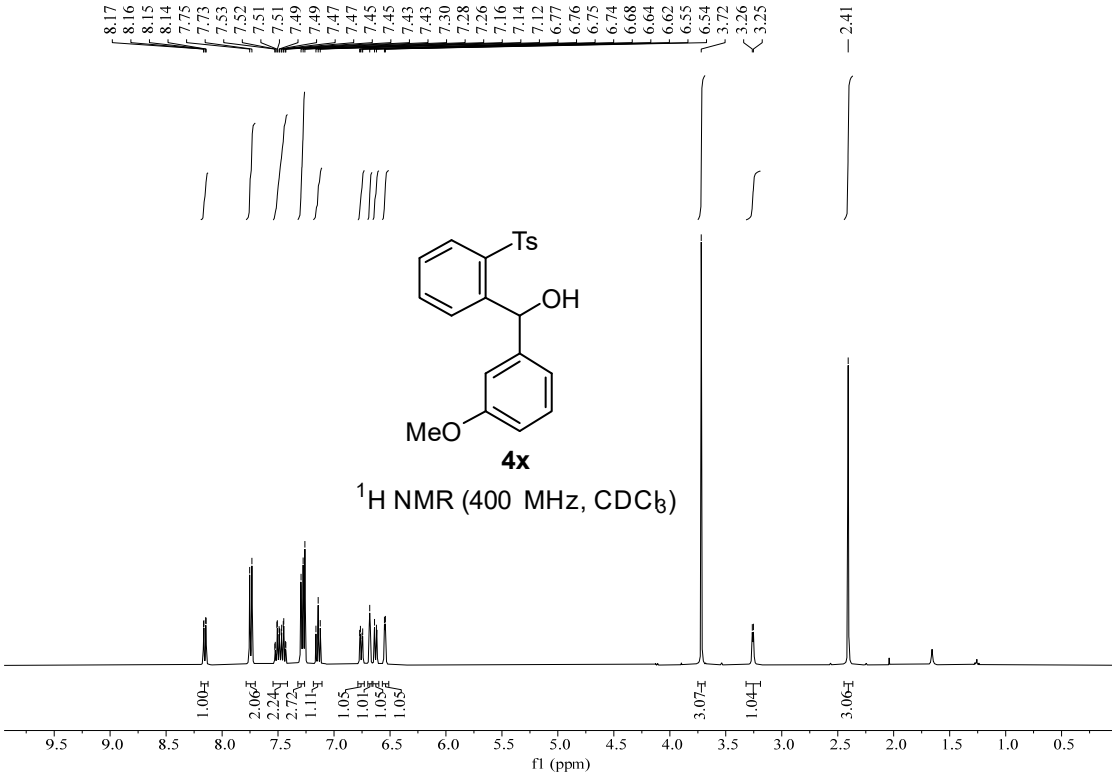
4w

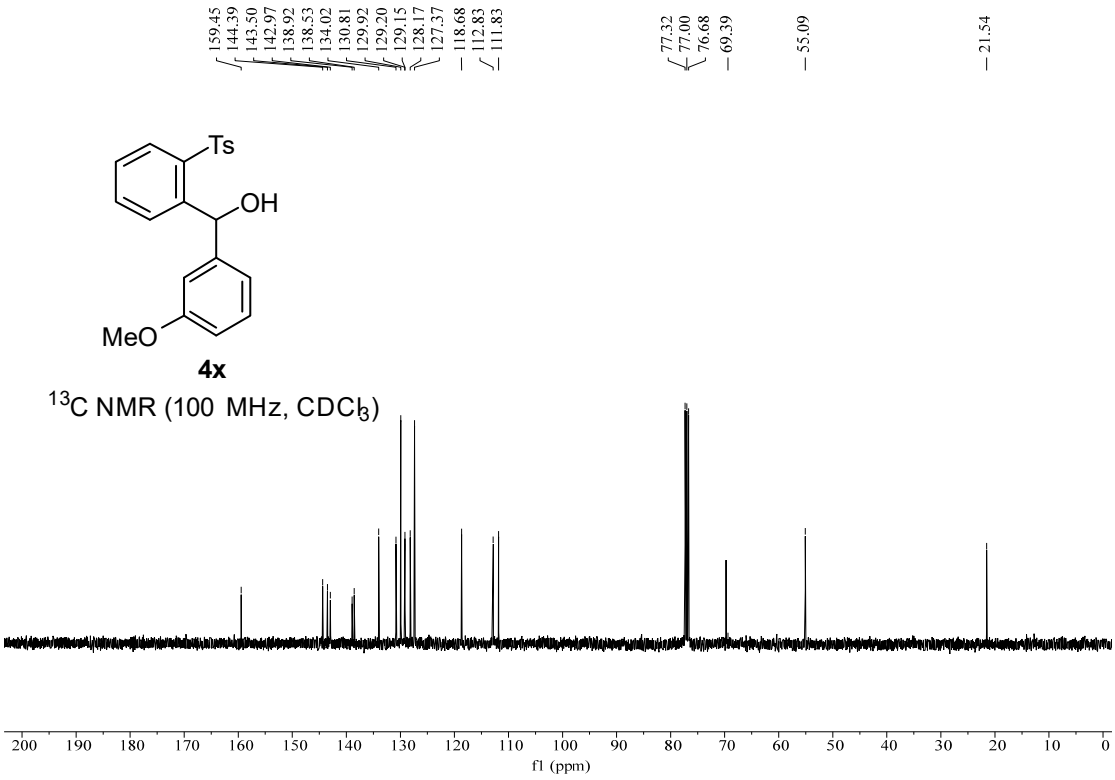
^{1}H NMR (400 MHz, $CDCl_3$)

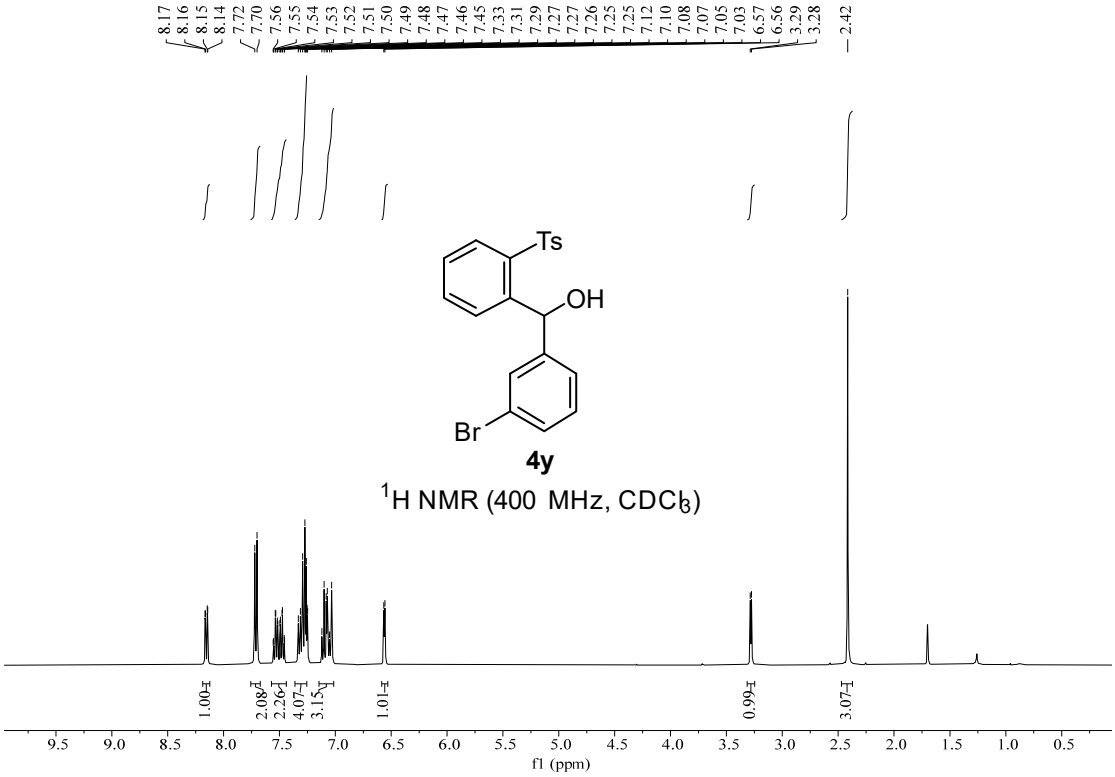


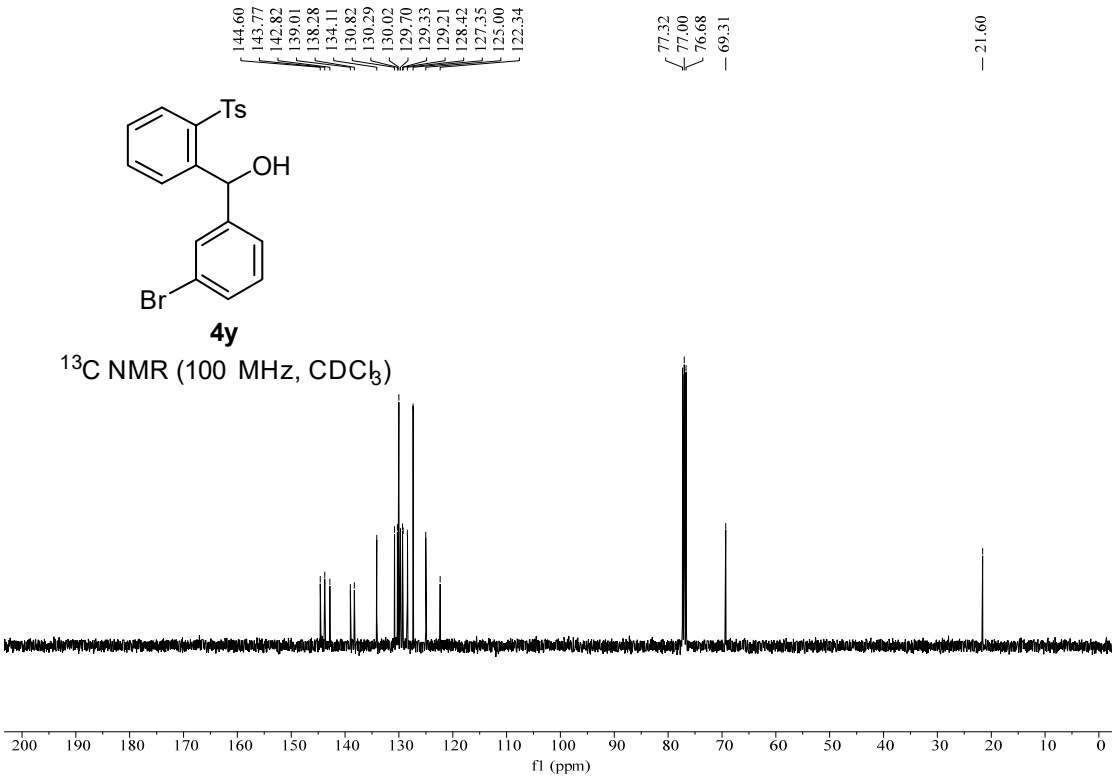
4w

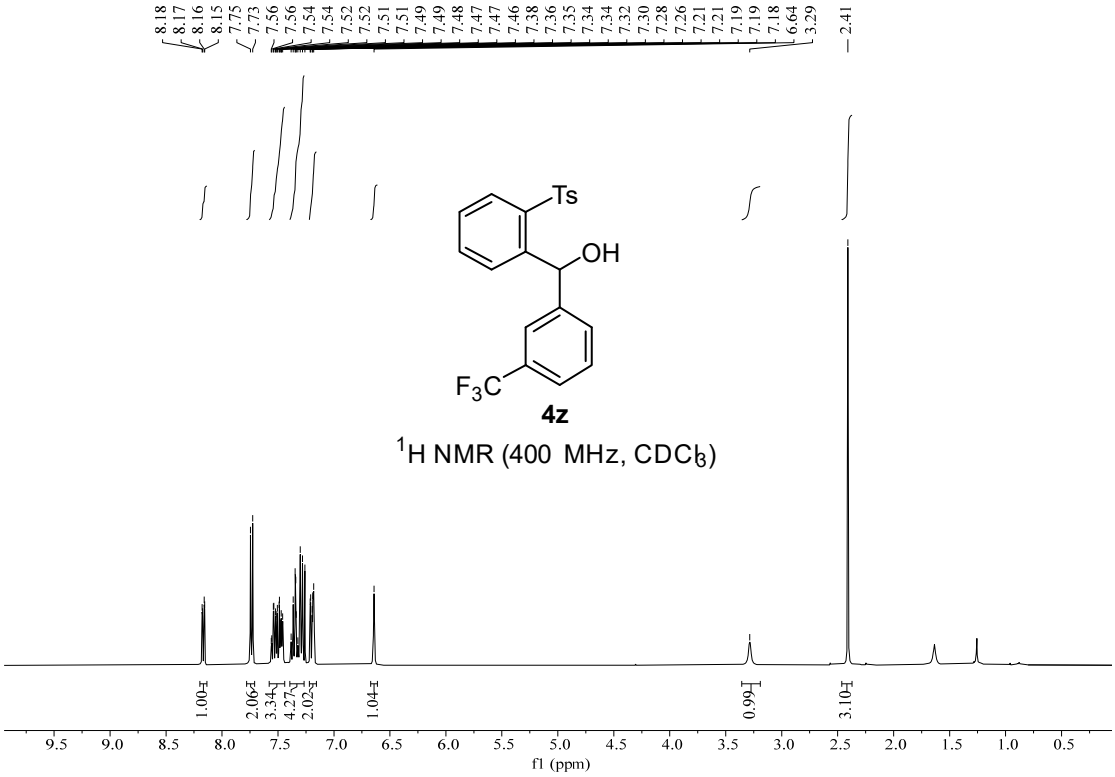
^{13}C NMR (100 MHz, $CDCl_3$)

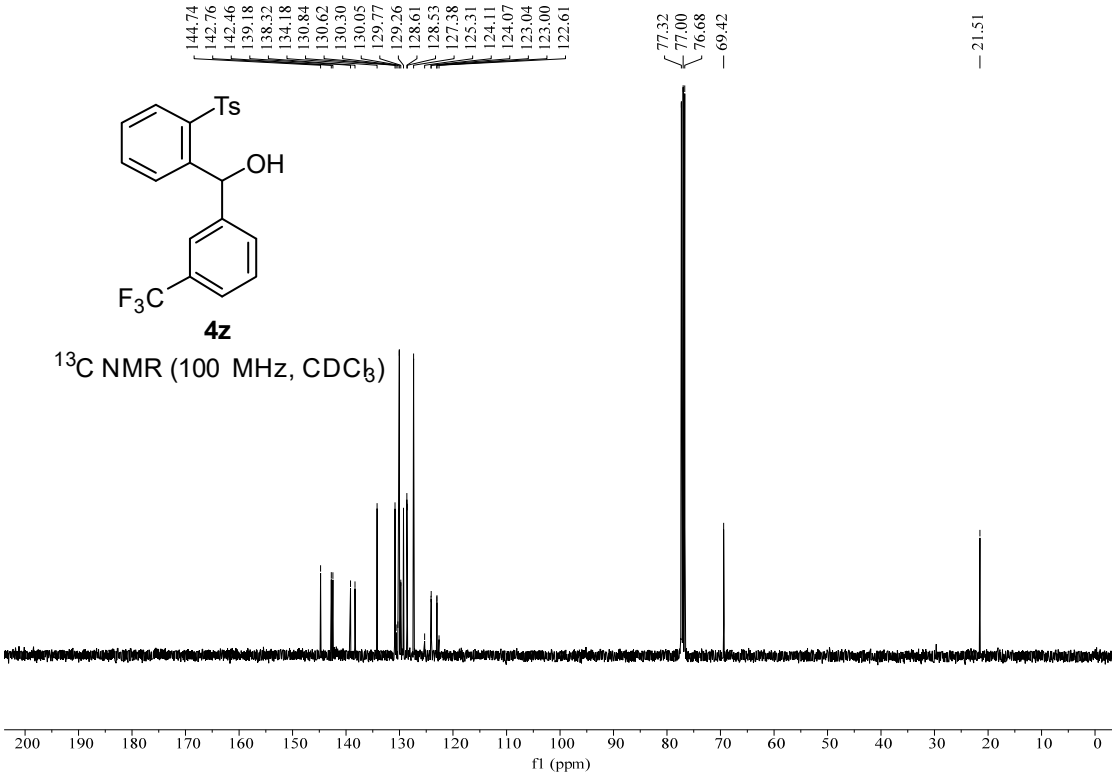

8.17
8.16
8.15
8.14
7.75
7.73
7.53
7.52
7.51
7.51
7.49
7.49
7.47
7.47
7.45
7.45
7.43
7.43
7.30
7.28
7.26
7.16
7.14
7.12
6.77
6.76
6.75
6.74
6.68
6.64
6.62
6.55
6.54
3.72
3.26
3.25
2.41
Ts
OH
MeO
4x
1H NMR (400 MHz, CDCl3)
1.00
2.06
2.24
2.72
1.11
1.05
1.01
1.05
1.05
3.07
1.04
3.06
f1 (ppm)

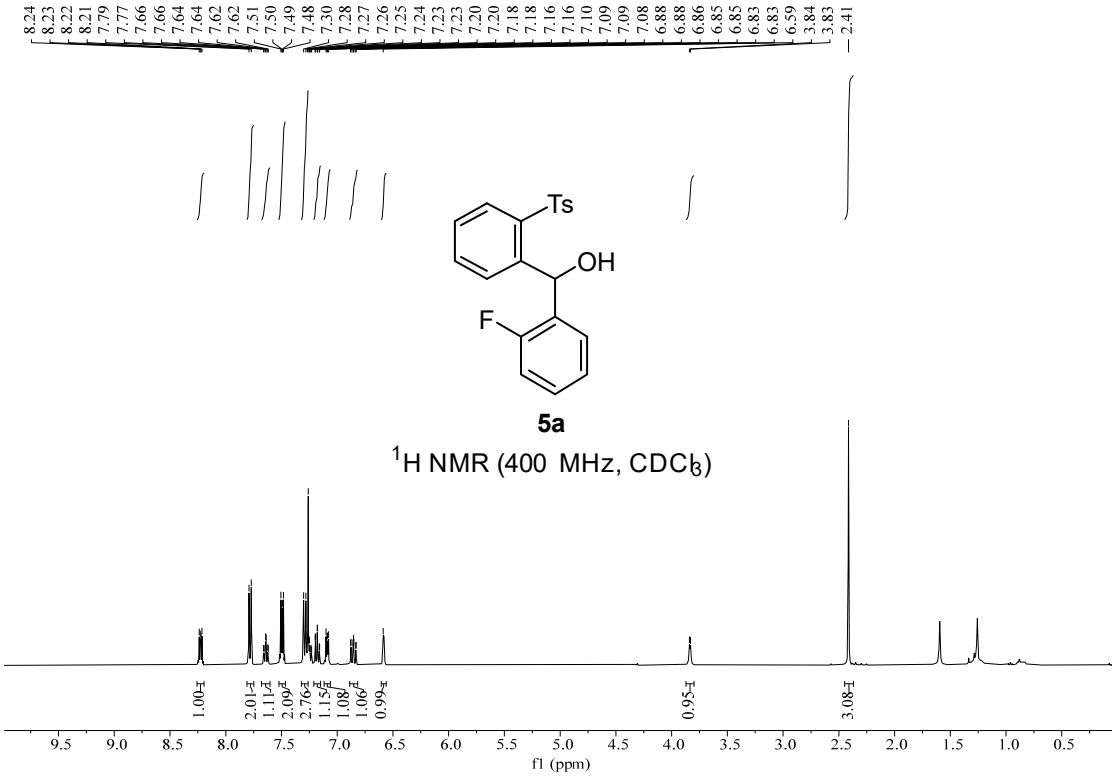

159.45
144.39
143.50
142.97
138.92
138.53
134.02
130.81
129.92
129.20
129.15
128.17
127.37
118.68
112.83
111.83
77.32
77.00
76.68
69.39
55.09
21.54
Ts
OH
MeO
4x
13C NMR (100 MHz, CDCl3)
f1 (ppm)


8.17
8.16
8.15
8.14
7.72
7.70
7.56
7.55
7.54
7.53
7.52
7.51
7.50
7.49
7.48
7.47
7.46
7.45
7.33
7.31
7.29
7.27
7.27
7.26
7.25
7.25
7.12
7.10
7.08
7.07
7.05
7.03
6.57
6.56
3.29
3.28
2.42
Ts
OH
Br
4y
1H NMR (400 MHz, CDCl3)
1.00
2.08
2.26
4.07
3.15
1.01
0.99
3.07
f1 (ppm)


144.60
143.77
142.82
139.01
138.28
134.11
130.82
130.29
130.02
129.70
129.33
129.21
128.42
127.35
125.00
122.34
77.32
77.00
76.68
69.31
21.60
Ts
OH
Br
4y
13C NMR (100 MHz, CDCl3)
f1 (ppm)

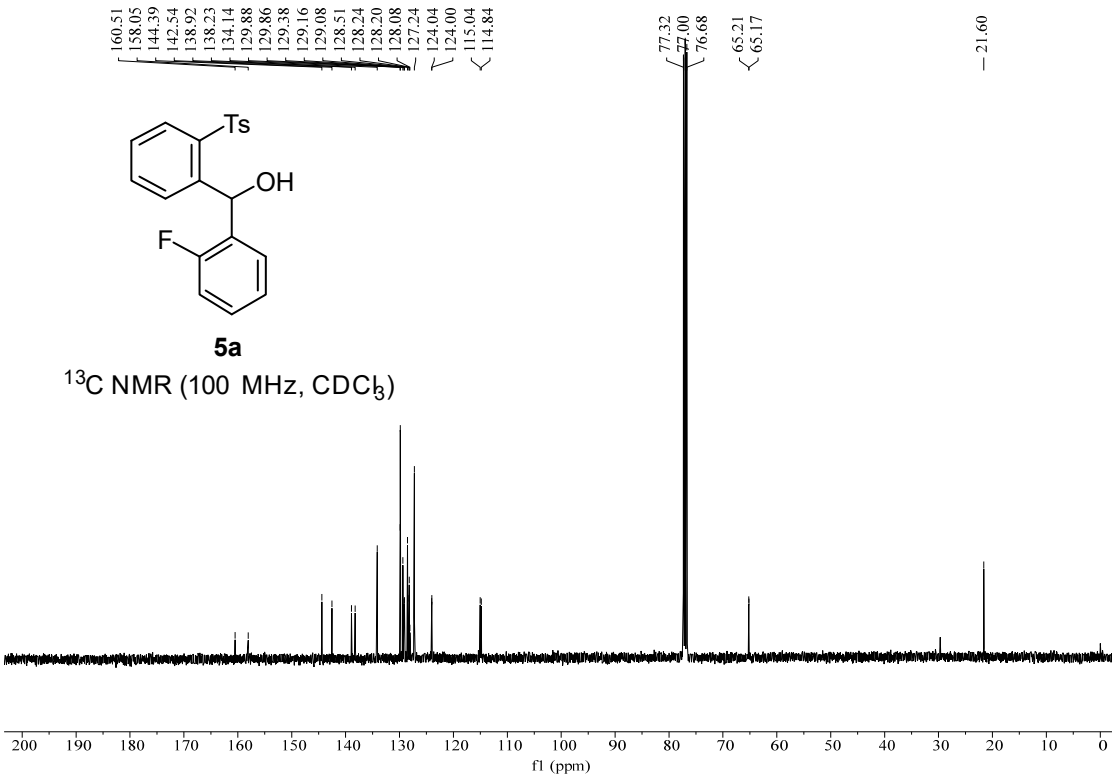

8.18
8.17
8.16
8.15
7.75
7.73
7.56
7.56
7.54
7.54
7.52
7.52
7.51
7.51
7.49
7.49
7.48
7.47
7.47
7.46
7.38
7.36
7.35
7.34
7.34
7.32
7.30
7.28
7.26
7.21
7.21
7.19
7.19
7.18
6.64
3.29
2.41
Ts
OH
F3C
4z
1H NMR (400 MHz, CDCl3)
1.00
2.06
3.34
4.27
2.02
1.04
0.99
3.10
f1 (ppm)


144.74
142.76
142.46
139.18
138.32
134.18
130.84
130.62
130.30
130.05
129.77
129.26
128.61
128.53
127.38
125.31
124.11
124.07
123.04
123.00
122.61
77.32
77.00
76.68
69.42
21.51
Ts
OH
F3C
4z
13C NMR (100 MHz, CDCl3)
f1 (ppm)



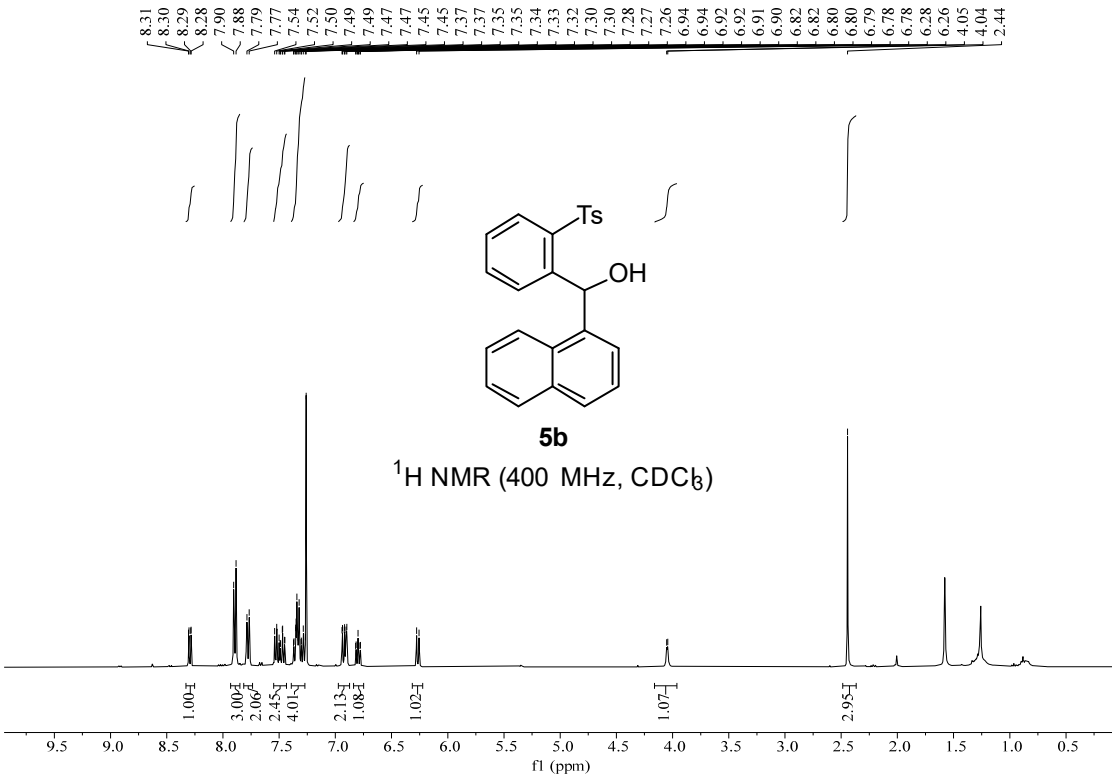
5a

^{1}H NMR (400 MHz, $CDCl_3$)



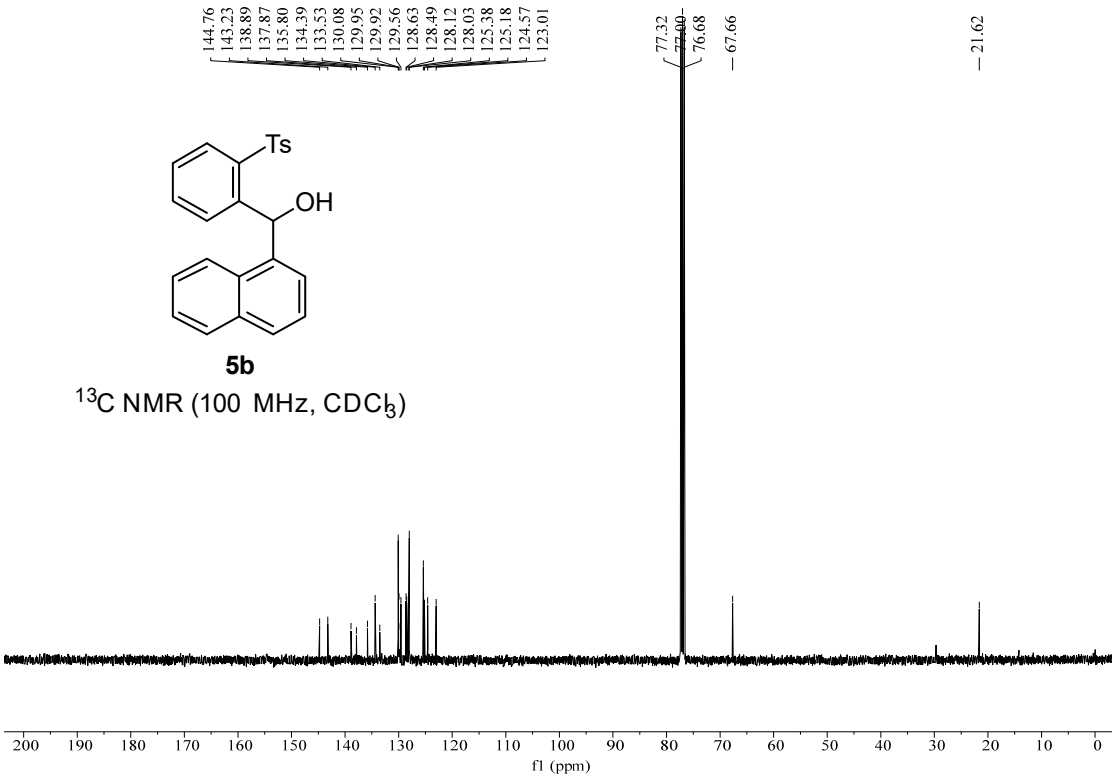
5a

^{13}C NMR (100 MHz, $CDCl_3$)



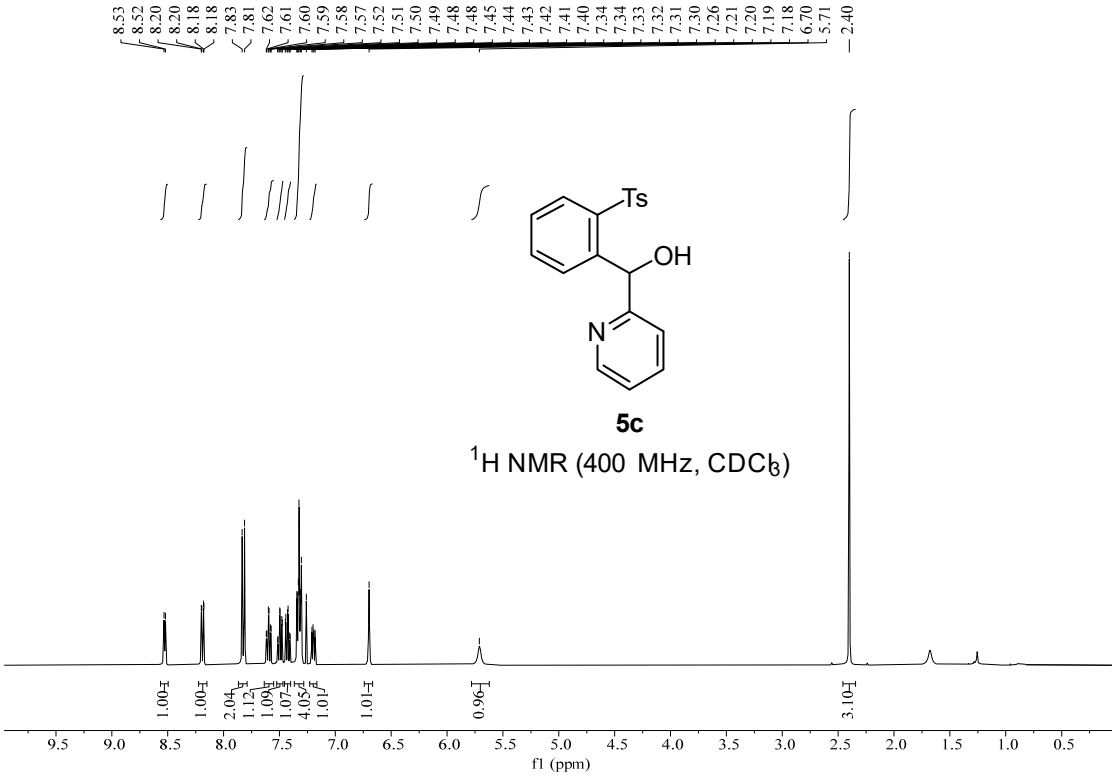
5b

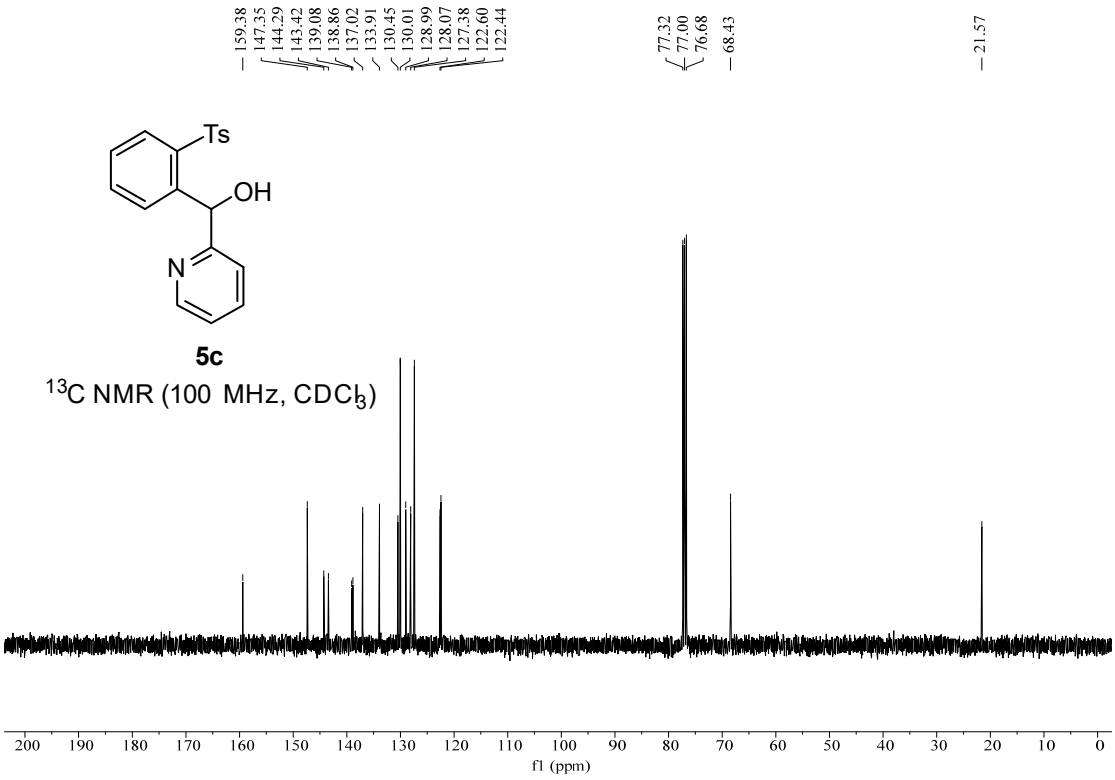
^{1}H NMR (400 MHz, $CDCl_3$)

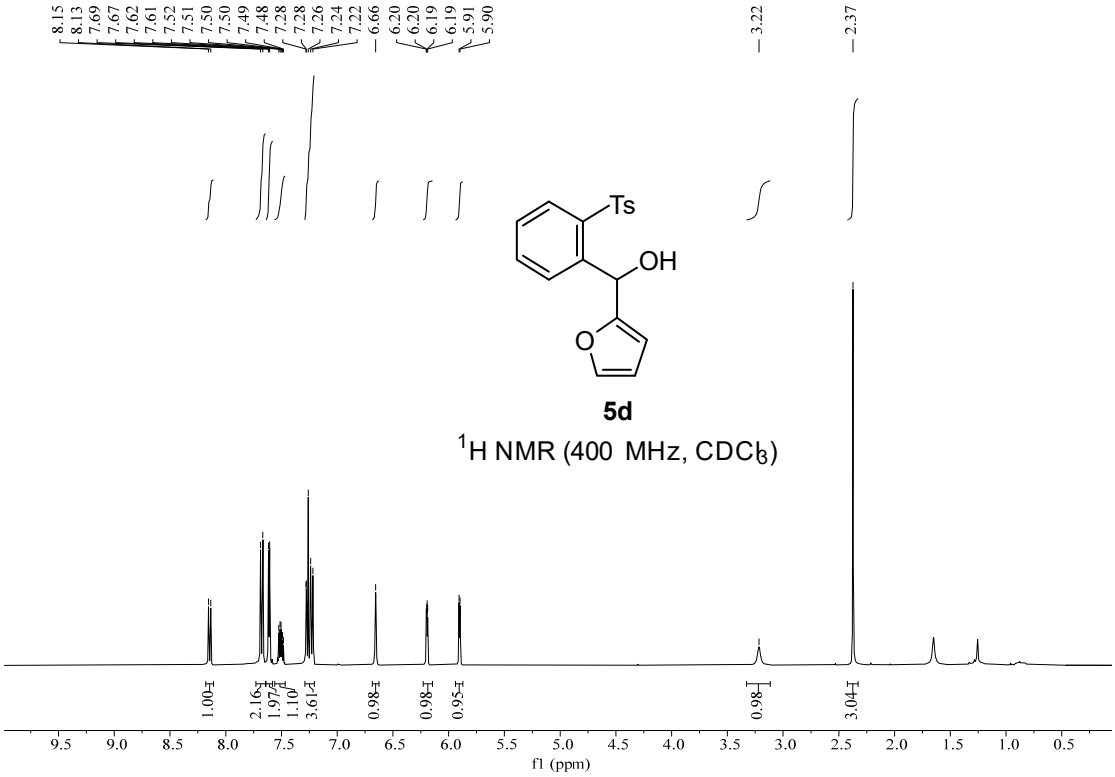


5b

^{13}C NMR (100 MHz, $CDCl_3$)

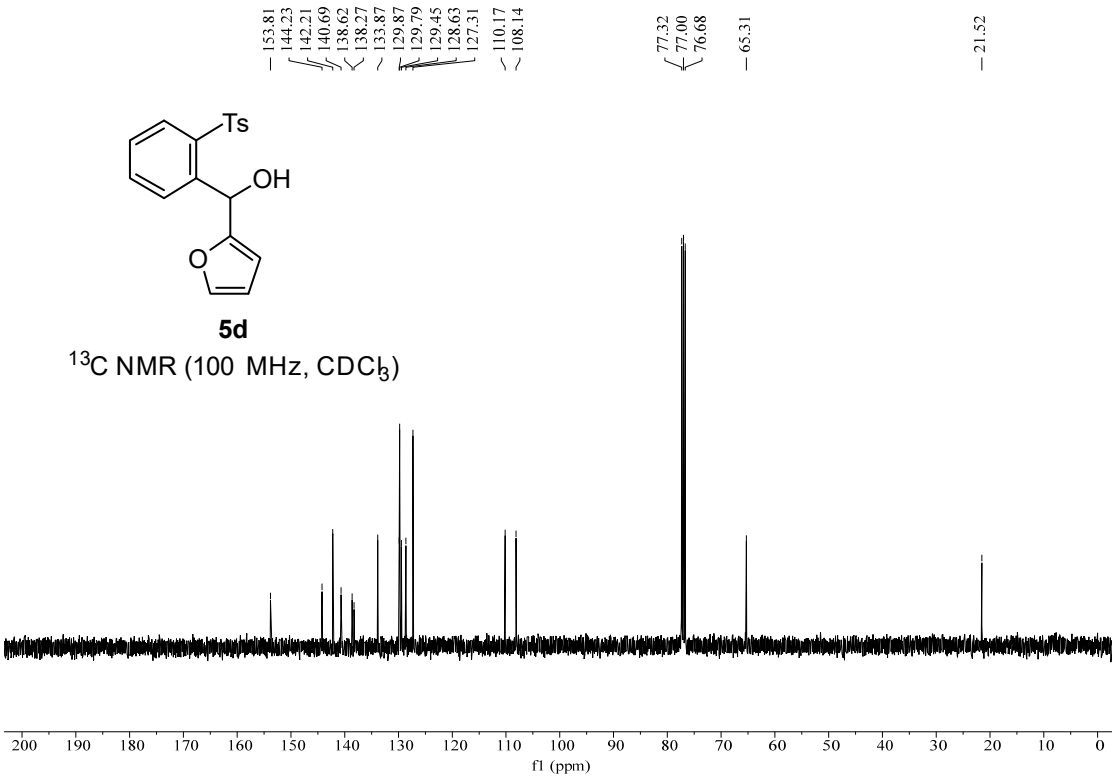

Ts
OH
N
5c
1H NMR (400 MHz, CDCl3)
f1 (ppm)


Ts
OH
N
5c
13C NMR (100 MHz, CDCl3)
f1 (ppm)



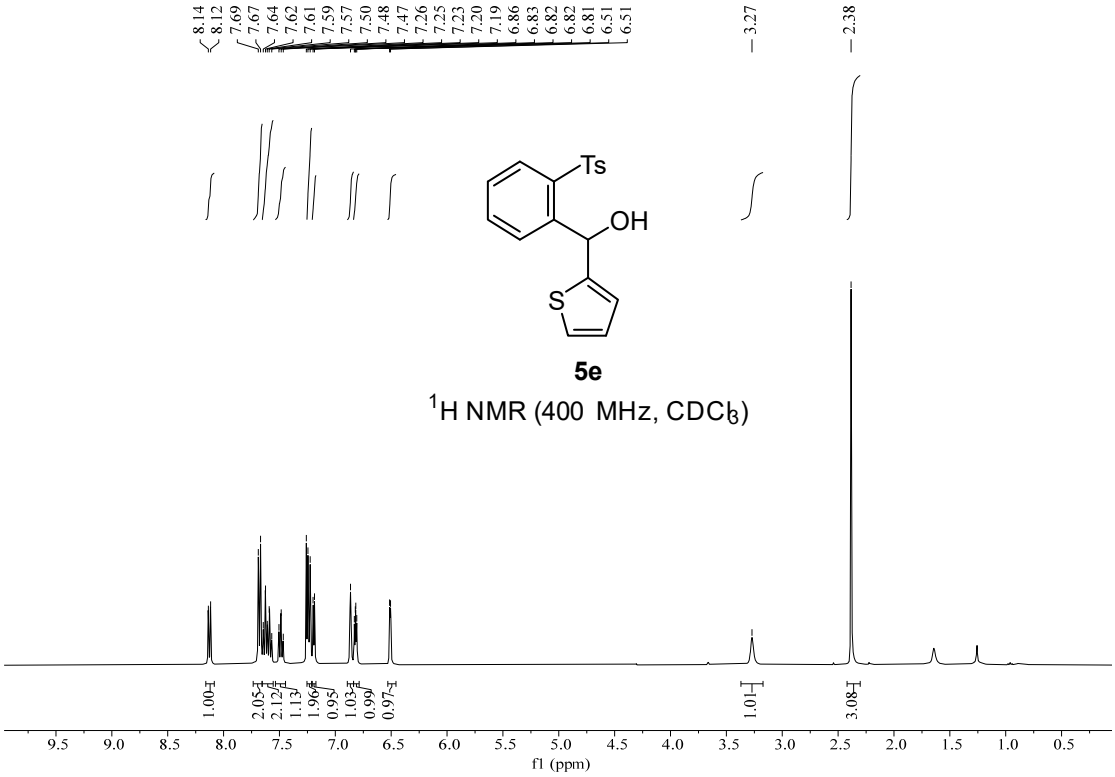
5d

^{1}H NMR (400 MHz, $CDCl_3$)



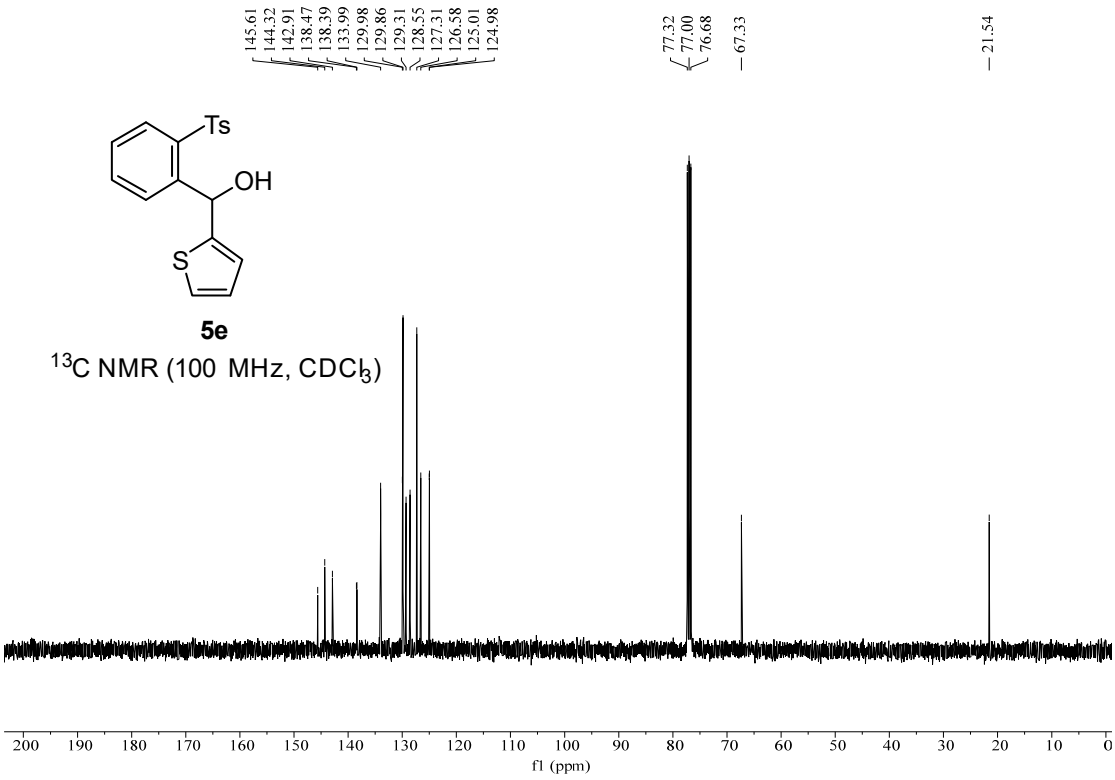
5d

^{13}C NMR (100 MHz, $CDCl_3$)



5e

^{1}H NMR (400 MHz, $CDCl_3$)



5e

^{13}C NMR (100 MHz, $CDCl_3$)